



## Standard Test Method for Carbon Black—CTAB (Cetyltrimethylammonium Bromide) Surface Area<sup>1</sup>

This standard is issued under the fixed designation D 3765; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the measurement of the specific surface area of carbon black exclusive of area contained in micropores too small to admit hexadecyltrimethylammonium bromide (cetyltrimethylammonium bromide, commonly referred to as CTAB) molecules. This test method is suitable for characterizing rubber-grade carbon blacks of all types.

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 1193 Specification for Reagent Water<sup>2</sup>

D 1799 Practice for Carbon Black—Sampling Packaged Shipments<sup>3</sup>

D 1900 Practice for Carbon Black—Sampling Bulk Shipments<sup>3</sup>

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries<sup>3</sup>

### 3. Summary of Test Method

3.1 The isotherm for adsorption of an aqueous solution of CTAB on carbon black has a long horizontal plateau corresponding to a monolayer coverage of the substrate surface from which the adsorbate is not sterically excluded. The CTAB adsorption by carbon black is independent of residual tars and functional groups containing hydrogen and oxygen, etc. Rapid equilibration is achieved by using mechanical stirring and

ultrasonic vibration. Titration with dioctyl sodium sulfosuccinate (Aerosol OT<sup>4</sup>) solution to a turbidity maximum end point is used to determine the unadsorbed CTAB after removal of the colloiddally dispersed carbon black by ultrafiltration. All results are scaled by using the Industry Tint Reference Black (ITRB) as a primary standard and taking its accessible surface area to be exactly  $83.0 \times 10^3 \text{ m}^2/\text{kg}$ .

3.2 Titration of the unadsorbed CTAB with Aerosol OT solution is accomplished by the following:

3.2.1 *Part A*—Automatic Titration.

3.2.2 *Part B*—Manual Turbidity Titration.

### 4. Significance and Use

4.1 The CTAB molecule is relatively large; so it is not adsorbed in micropores or on surface roughness. Thus, the CTAB surface area reflects only the surface of the carbon black that is available for interaction with rubber molecules.

### 5. Apparatus

5.1 *Analytical Balance*, 0.1-mg sensitivity.

5.2 *Ultrasonic Cleaning Bath*,<sup>5</sup> modified to incorporate integral magnetic stirring and vial holder to properly position two sample vials. (If desired, a separate ultrasonic cleaning bath<sup>6</sup> and a separate magnetic stirrer<sup>7</sup> may be used.)

5.3 *Magnetic Spinbars*,<sup>8</sup> chemically resistant covered (polychlorotrifluoroethylene or TFE-fluorocarbon), 6.4 or 4.8-mm ( $1/4$  or  $3/16$ -in.) diameter, and length nearly equal to the diameter of 50 or 100-cm<sup>3</sup> Berzelius beakers, glass vials, or other glass vessels.

<sup>4</sup> Aerosol OT is a registered trademark of the American Cyanamid Co., Process Chemicals Dept., Wayne, NM 07470.

<sup>5</sup> Available from L.A. King Manufacturing Corp., LAKO Division, P.O. Box 2415, Tulsa, OK 74101.

<sup>6</sup> Millipore Corp., Bedford, MA 01730, Catalog No. XX 66 00900 is satisfactory.

<sup>7</sup> Sargent magnetic stirrer S-76490 is satisfactory.

<sup>8</sup> Examples of suitable spinbars are Catalog No. 9235-U7, A. H. Thomas Co., Philadelphia, PA 19105, or Catalog No. S-76497-30, Sargent Welch Scientific Co., Skokie, IL 60076.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-24 on Carbon Black and is the direct responsibility of Subcommittee D24.21 on Adsorptive Properties of Carbon Black.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 09.01.

5.4 *Microfiltration Membrane Filter Disks*,<sup>9</sup> 47-mm diameter.

5.5 *Filter Holder*.<sup>10</sup>

5.6 *Sampling Cylinder*, 30-cm<sup>3</sup> stainless steel, double end with 6.4-mm (1/4-in.) NPT female openings, suitable for 0.7-MPa (100-psi) pressure.<sup>11</sup>

5.7 *Pressure Manifold*, connected to air or nitrogen supply regulated at 0.4 to 0.7 MPa (60 to 100 psi). Fig. 1 shows a schematic diagram of a typical assembly.

5.8 *Glass Funnel*, small.

5.9 *Glass Vials*, with plastic screw caps, 28 mm in outside diameter, about 40-cm<sup>3</sup> capacity (11-dram size).

5.10 *Buret*, 50-cm<sup>3</sup>, 0.1-cm<sup>3</sup> divisions, Class A, preferably of automatic refilling and zeroing type (Note 1) with reagent reservoir.

NOTE 1—Automatic burets are usually not certified to Class A tolerance. Such burets should be checked for accuracy, and, if in error by more than 0.05 cm<sup>3</sup> at any point, a calibration curve should be prepared and used to correct observed buret readings. Burets with TFE-fluorocarbon manostat valves offer some advantage in ease of stopcock manipulation in delivering small increments of titrant.

5.11 *Dispenser-Type Pipet*,<sup>12</sup> 50-cm<sup>3</sup>, attached to a suitable reservoir for CTAB solution.

5.12 *Pipet*, 10-cm<sup>3</sup>, Class A.

5.13 *Erlenmeyer Flasks*, or beakers, 50 or 100-cm<sup>3</sup>.

5.14 *Dropping Bottle*, 60-cm<sup>3</sup> amber.

5.15 *Jar*, wide-mouth, plastic screw cap, 118 or 236-cm<sup>3</sup> (4 or 8-oz) size.

<sup>9</sup> This test is based on the use of three brands of microfiltration membrane: (1) Gelman HT-200, Gelman No. 66199, Baxter Scientific Cat. No. F2988-2, Baxter Scientific Products, 1430 Waukegan Rd., McGraw Park, IL 60085; (2) Microfiltration Systems, Cat. No. A-010A047A, 6800 Sierra Court, Dublin, CA 94566; and (3) Millipore Cat. No. SAIJ076H7 Filters, Millipore Corp., Bedford, MA 01730.

<sup>10</sup> This test is based on the use of Swinnex-47 filter holders obtained from Millipore Corp. as Catalog No. SX00 047 00.

<sup>11</sup> Hoke, Inc., Kresskill, NJ 07626, No. 4DY30 is suitable.

<sup>12</sup> This test is based on the use of the Universal Repipet, a registered trademark of Labindustries, 1802 Second St., Berkeley, CA 94710.

5.16 *Gravity Convection Drying Oven*, capable of maintaining 125 ± 5°C.

5.17 *Microscope Spotlight*.

5.18 *TFE-fluorocarbon Pipe Thread Sealing Tape*.

5.19 *Magnetic Stirrer*.

5.20 *Containers*, suitable for preparation and storage of reagent solutions.

5.21 *Automatic Titration Equipment*,<sup>13</sup> with instruction manual.

5.22 *Beakers*, 100-cm<sup>3</sup> Berzelius, tall form, (one furnished with automatic titration equipment).

5.23 *Thermometer*, to measure temperature in a range from 20 to 40°C.

5.24 *Polyethylene Tubing* (do not use vinyl tubing).

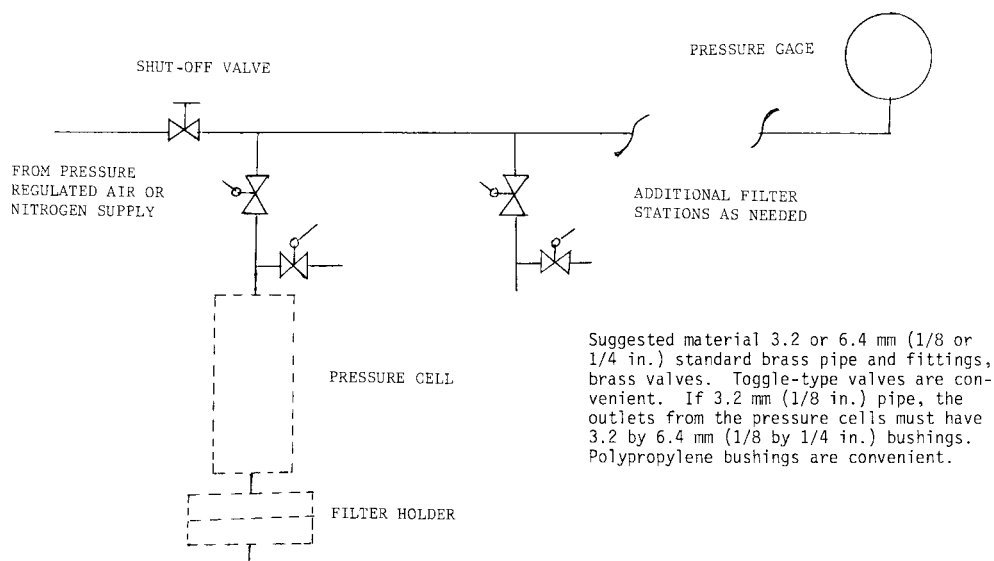
NOTE 2—All apparatus must be kept chemically clean. Contamination of equipment, water supply, or chemicals by ionic surfactants must be particularly avoided. The filtration equipment, especially the region below the perforated plate in the filter holder, must not be permitted to accumulate carbon black or cleanser residues.

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>14</sup> Other grades may be used,

<sup>13</sup> This test is based on the use of three sources of automatic titration equipment: (1) Mettler Memotitrator DL40RC, Mettler Instrument Co., Box 71, Highstown, NJ 08520, (2) Brinkman Dosimat 665 Buret, Brinkman Instruments, Inc., Cantiague Rd., Westbury, NY, and (3) ATMAST, available from L. A. King Manufacturing Corp., LAKO Division, P.O. Box 2415, Tulsa, OK 74101. The Probe Colorimeter, also available from Brinkman Instruments, has been found to be an acceptable automatic means of detecting the titration end point. This piece of equipment may be integrated with the Brinkman Dosimat 665 Buret.

<sup>14</sup> "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."



**FIG. 1 Pressure Filtration Manifold**

provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 in Specification D 1193.

6.3 *Buffer Solution of pH 7 (0.05M)*—Prepare buffer solution by dissolving 2.722 g of monobasic potassium phosphate ( $\text{KH}_2\text{PO}_4$ ), 4.260 g of dibasic sodium phosphate ( $\text{Na}_2\text{HPO}_4$ ) and 1.169 g of sodium chloride ( $\text{NaCl}$ ) in 1 dm<sup>3</sup> of distilled water.<sup>15</sup> Alternatively, this prepared solution may be purchased.<sup>16</sup>

6.4 *CTAB Standard Solution (0.0100 M)*—Dissolve 3.64 g of reagent-grade CTAB<sup>17</sup> per 900 cm<sup>3</sup> of distilled or deionized water. Add 100 cm<sup>3</sup> of the 7.0 pH buffer solution to this solution. Warm the solution to a temperature of 27 to 37°C to facilitate the solution. Cool to a temperature between 22 and 25°C before using.

NOTE 3—**Caution:** Storage of the solution below 22°C will result in slow crystallization of the CTAB.

6.5 *Aerosol OT<sup>4</sup> 18 Solution 100 % Grade (approximately 0.0022 M)*—Dissolve 1.000 g/dm<sup>3</sup> of Aerosol OT solution (100 % solids) in distilled or deionized water containing 2.5 cm<sup>3</sup>/dm<sup>3</sup> of 37 % formaldehyde, by swirling vigorously with a magnetic stirrer for 48 h. Allow to stand an additional twelve days before standardization and use. (Aerosol OT solution may be subject to slow biodegradation in the absence of formaldehyde.) The solution should be capped tightly and stored in a cool place. (Once opened, 100 % Aerosol OT should be stored in a desiccator.)

6.6 *Formaldehyde Solution*, approximately 37 %.

6.7 *ITRB*,<sup>19</sup> equals  $83.0 \times 10^3 \text{ m}^2/\text{kg}$  surface area.

6.8 *Bromophenol Blue*, reagent grade.

6.9 *Ethanol*, reagent grade.

6.10 *Distilled or Deionized Water*.

6.11 *Octylphenoxy Polyethoxyethanol (Triton X-100<sup>20</sup>) Solution (0.15 %)*— Dissolve 1.5 g/dm<sup>3</sup> of Triton X-100 (100 % liquid) in distilled or deionized water by swirling vigorously with a magnetic stirrer until a homogeneous solution is obtained.

## 7. Sampling

7.1 Samples shall be taken in accordance with Practice D 1799 or D 1900.

## 8. Standardization of Reagents

8.1 Dry an adequate portion of ITRB for 1 h at  $125 \pm 1^\circ\text{C}$ .

<sup>15</sup> Bates, R. G., Hamer, W. J., Manor, G. G., and Acree, S. F., *Journal of Research*, National Institute of Standards and Technology, No. 29, 1942, p. 183.

<sup>16</sup> Available from Fisher Scientific Co., Catalog No. SB-108, 711 Forbes Ave., Pittsburgh, PA 15105.

<sup>17</sup> Available from J. T. Baker Chemical Co., 222 Red School Lane, Phillipsburgh, NJ 08865, Catalog No. 7N121.

<sup>18</sup> Available from American Cyanamid Co., Process Chemicals Dept., Wayne, NM 07470. Also available as Catalog No. A-349 from Fisher Scientific Co.

<sup>19</sup> Available from Ashland Chemical Co., Carbon Black Division, P.O. Box 2219, Columbus, OH 43216.

<sup>20</sup> Triton X-100 is a registered trademark of Rohm and Haas Co.

8.2 Weigh, to 0.1 mg, five samples of the dried ITRB covering the range from 0.2 to 0.6 g in intervals of about 0.1 g.

8.3 With each sample, carry out the equilibration and filtration in accordance with 9.3-9.8 and the titration in accordance with 9.9.1 or 9.9.2.

8.4 Plot the titration volumes,  $V$ , against the sample masses,  $W$ . Fit the best possible straight line through the points (preferably by least squares regression) and determine the slope,  $b$ , (cubic centimetres per gram) and the volume axis intercept,  $V_o$ .

8.5 The factors  $V_o$  and  $b$  are the standardization factors used in the calculation of CTAB surface area. Standardization is necessary each time the CTAB or Aerosol OT solution is prepared.

8.6 From these numbers, the CTAB surface area ( $SA$ ) of an unknown black tested with these reagents can be obtained from the following equation:

$$SA = \frac{SA_{ITRB}}{-b} \times \frac{(V_o - V)}{W} \quad (1)$$

where:

$SA$  = CTAB surface area,  $10^3 \text{ m}^2/\text{kg}$ ,  
 $V_o$  = intercept from the standardization plot,  
 $b$  = slope from the standardization plot,  
 $V$  = volume of Aerosol OT solution,  
 $W$  = mass of unknown black, and  
 $SA_{ITRB}$  =  $83.0 \times 10^3 \text{ m}^2/\text{kg}$ .

8.7 The five data points from the standardization should be inserted into this formula to check the reliability of the standardization. CTAB  $SA$  of  $83.0 \pm 1 \times 10^3 \text{ m}^2/\text{kg}$  for ITRB should be obtained. If all five data points or daily calibration checks do not agree with assigned values, repeat the standardization, paying special emphasis to equipment and equilibration in 9.3 and 9.4.

## 9. Procedure

9.1 Dry an adequate sample of carbon black at 125°C for 1 h and cool in a desiccator.

NOTE 4—If the heating loss of the carbon black is known, the drying step may be omitted and the observed sample mass corrected for heating loss to obtain the mass of dry sample,  $W$ .

9.2 Weigh, to 0.1 mg, a suitable sample of the dried carbon black into a glass vial and record the mass as  $W$ . Appropriate sample masses may be selected as follows:

Grade of Black	CTAB Range, m <sup>2</sup> /kg	Sample Mass, g
N100 Series	125 to $150 \times 10^3$	0.30
N200	100 to $130 \times 10^3$	0.35
N300	75 to $105 \times 10^3$	0.40
N351-N440	50 to $75 \times 10^3$	0.60
N500-N600	35 to $50 \times 10^3$	0.90
N700	25 to $30 \times 10^3$	1.35

An appropriate sample mass may also be taken as  $V_o/S_{\text{est}}$  in grams, where  $S_{\text{est}}$  is some estimate of the expected CTAB surface area.

9.3 Insert a magnetic spinbar into the vial. Add 30.0 cm<sup>3</sup> of CTAB solution from the dispensing pipet or buret taking care to prevent foaming of the solution. Cap the vial.

9.4 Insert the vial into the ultrasonic bath that contains water at a temperature between 22 and 25°C to a depth of at least 50 mm (2 in.). Adjust the magnetic stirrer to give vigorous stirring

by the spinbar to produce a deep vortex without foaming. Turn on the ultrasonic power for 3 min to equilibrate the mixture. Ensure that the water temperature in the bath remains between 22 and 25°C throughout the equilibration.

**NOTE 5**—If a combination magnetically stirred ultrasonic dispersion bath is not available, dispersing may be done by alternately agitating in an ultrasonic cleaning bath and stirring with a magnetic stirrer. The sample is agitated in an ultrasonic bath for 1 min and then stirred for 1 min. This cycle is repeated two more times for a total time of 6 min.

**NOTE 6**—The ultrasonic energy tends to warm the bath, especially if several sets of samples are equilibrated in quick succession. Various means are employed to overcome this; for example, replacement of water when it becomes too warm, dropping in small pieces of ice, or installation of a cooling coil. However, the bath temperature must not be allowed to fall below 22°C.

9.5 Attach the top (threaded) part of a filter holder to a mating 30-cm<sup>3</sup> stainless steel pressure cell, using TFE-fluorocarbon thread tape and hand tighten sufficiently to avoid leakage.

9.6 Install the filter disk in the filter holder, shiny side toward the inlet, according to the instructions furnished with the filter holder.

**NOTE 7**—Curled filters can make leak-free installation difficult. They can be flattened sufficiently to alleviate this difficulty by drawing the convex side over an edge, such as that of a plastic rule, before blotting. Proper seating of the filter may be aided by applying suction to the bottom part of the filter holder during assembly. Care must be taken not to damage the filter by creasing or folding. Proper filter seating can be checked by pressure testing the assembly before the sample is added. Absence of gas flow, detectable by placing the finger over the outlet, indicates proper seating.

9.7 Wipe clinging water from the outside of the sample vial. Pour the sample through a small funnel into the pressure cell. Connect the cell to the pressure manifold and filter under 0.4 to 0.7 MPa (60 to 100 psi) of air or nitrogen pressure. Discard the first 3 to 5 cm<sup>3</sup> of filtrate and then collect the rest of the filtrate in a clean vial or other small container (Note 7). Agitate or stir the collected filtrate to ensure uniformity.

**NOTE 8**—In case of black filtrate due to a filter leak, discard the sample; do not refilter.

9.8 Proceed immediately to either 9.9.1 or 9.9.2.

9.9 *Titration:*

9.9.1 *Test Method A—Automatic Titration:*

9.9.1.1 Prepare the automatic titration apparatus according to the instructions furnished with it. Ascertain that the titrant reservoir contains sufficient Aerosol OT solution and that the fluid lines and the pump head are free of air bubbles and have been flushed sufficiently with titrant. Turn the power on and loosen the titrant reservoir cap to admit air as liquid flows out. Adjust the titrant flow rate to 10 cm<sup>3</sup>/min.

**NOTE 9**—If Atmast automatic titration equipment is employed, adjust titrant flow rate to 6 cm<sup>3</sup>/min, per manufacturer's recommendation.

9.9.1.2 Add 45 cm<sup>3</sup> of distilled or deionized water to a 100-cm<sup>3</sup> tall-form Berzelius beaker containing a TFE-fluorocarbon-covered magnetic spinbar 41 mm long by 10 mm in diameter (1.625 by 0.375 in.).

9.9.1.3 Add 5 cm<sup>3</sup> of 0.15 % Triton X-100 solution.

9.9.1.4 Pipet a 10.00-cm<sup>3</sup> aliquot of the CTAB solution from 9.7 to be titrated into the beaker, taking care to avoid

formation of excess foam.

9.9.1.5 Place the beaker into the automatic titrator sample well and adjust the magnetic stirrer speed control so that the vortex generated by the stirring action is just at the top of the light beam that passes through the beaker.

9.9.1.6 Lower the titrant delivery assembly so that the delivery needle is just below the surface of the liquid and open the titrant stopcock.

9.9.1.7 Reset the clock.

9.9.1.8 Set the pump control switch to the “titrate” position and press the “start” button.

9.9.1.9 Wait for the pump and clock to cut off at the turbidity maximum.

9.9.1.10 Record the clock (volume) reading to the nearest 0.01 cm<sup>3</sup>.

9.9.1.11 Raise the delivery tube clear of the beaker.

9.9.1.12 Move the pump control to “flush” and allow a few drops of titrant to clear the needle. In coincidence with the end of a squirt, move the pump control to “off.” After the pump stops, close the stopcock and move the needle out of the way of the beaker.

9.9.1.13 Remove the sample beaker from the well.

9.9.1.14 Wipe the needle with clean tissue (do not use solvent). The unit is now ready for another sample.

**NOTE 10**—Results may not be valid if the volume of Aerosol OT solution used is not within the same range used for standardization. It is then advisable to repeat the test adjusting the sample mass accordingly.

9.9.2 *Test Method B—Manual Turbidity Titration:*

9.9.2.1 Same as 9.9.1.1.

9.9.2.2 Prepare a standard 100-cm<sup>3</sup> beaker with a red cross painted on the exterior bottom surface.

**NOTE 11**—The red cross painted on the bottom of beaker should be bright red. Finger nail polish is satisfactory.

9.9.2.3 Add 45 cm<sup>3</sup> of distilled or deionized water to the 100-cm<sup>3</sup> beaker containing a TFE-fluorocarbon coated magnetic spinbar 41 mm long by 10 mm diameter.

9.9.2.4 Same as 9.9.1.3.

9.9.2.5 Same as 9.9.1.4.

9.9.2.6 Place a white filter paper on top of a magnetic stirrer and place beaker with red cross and contents from 9.9.2.3, 9.9.2.4 and 9.9.2.5 on the filter paper. Adjust the spin rate so that the vortex is not more than 5-mm deep.

9.9.2.7 Lower the titrant delivery tip just below the surface of the liquid.

9.9.2.8 Activate the titrator delivery system and titrate Aerosol OT at a constant rate (10-cm<sup>3</sup>/min.) until sufficient turbidity is developed so that the red cross on the bottom of the beaker completely disappears. At this point, the titrator is switched off manually.

9.9.2.9 Record the volume (cm<sup>3</sup>) Aerosol OT titrated.

9.9.2.10 Raise the delivery tube clear of the beaker. Clean buret tip and beaker with distilled water. The titration equipment is now ready for the next sample titration.

## 10. Calculation

10.1 Calculate the CTAB surface area to the nearest  $0.1 \times 10^3$  m<sup>2</sup>/kg as follows:

$$S = \frac{(V_0 - V)}{W} \times \frac{SA_{ITRB}}{-b} \quad (2)$$

where:

- $S$  = CTAB surface area,  $10^3 \text{ m}^2/\text{kg}$ ,
- $V$  = Aerosol OT solution required for the sample titration,  $\text{cm}^3$ ,
- $W$  = sample mass, g,
- $b$  and  $V_0$  = the standardization constants for the CTAB and Aerosol OT solutions, and
- $SA_{ITRB}$  =  $83.0 \times 10^3 \text{ m}^2/\text{kg}$  for ITRB.

## 11. Report

11.1 Report the following information:

- 11.1.1 Proper identification of the carbon black sample and
- 11.1.2 Result obtained from an individual determination, reported to the nearest  $0.1 \times 10^3 \text{ m}^2/\text{kg}$ .

## 12. Precision and Bias

12.1 These precision statements have been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

12.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from Table 1.

12.3 A type 1 inter-laboratory precision program was conducted as detailed in Table 2. Both repeatability and reproducibility represent short term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each of two days (total of four tests). A test result is the value obtained from a single determination. Acceptable difference values were not measured. The between operator component of variation is included in the calculated values for  $r$  and  $R$ .

**TABLE 1 Precision Parameters for D 3765 CTAB Surface Area, (Type 1 Precision)**

Units	$10^{-5} \text{ m}^2/\text{kg} (\text{cm}^3/100 \text{ g})$				
Material	Mean Level	Sr	(r)	SR	(R)
N762	64.75	0.36	1.6	0.93	4.1
N650	120.89	0.58	1.3	2.03	4.8
N550	121.01	0.57	1.3	1.92	4.5
IRB#6 (N330)	99.63	0.36	1.0	0.63	1.8
N234	123.94	0.49	1.1	1.07	2.4
SRB A5 (N135)	136.69	0.44	0.9	0.94	1.9
Average	111.15				
Pooled Values		0.47	1.2	1.36	3.5

**TABLE 2 Interlaboratory Precision Program**

Nominal Test Period	Material	Number of Laboratories
September 1995	N234	17
March 1996	N650	35
October 1996	IRB#6 (N330)	25
March 1997	N762	28
September 1997	SRB A5 (N135)	25
March 1998	N550	21

12.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending “mean level” order.

12.5 *Repeatability*—The pooled relative repeatability, ( $r$ ), of this test has been established as 1.9 %. Any other value in Table 1 may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 12—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

12.6 *Reproducibility*—The pooled relative reproducibility, ( $R$ ), of this test has been established as 5.1 %. Any other value in Table 1 may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from Table 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

12.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

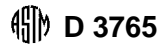
## 13. Keywords

13.1 carbon black; cetyltrimethylammonium bromide; CTAB; surface area by CTAB method; turbidity titration

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