



Designation: D 3765 – 03

## 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 specific surface area of carbon black exclusive of area contained in micropores too small to admit hexadecyltrimethyl ammonium bromide (cetyltrimethyl ammonium bromide, commonly referred to as CTAB). However, it should be noted that the preferred method for measuring external surface area is STSA (Test Method D 6556), and the CTAB method is being maintained by ASTM International in order to allow time for laboratories to obtain the proper instrumentation.

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 colloidal 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  $83.0 \times 10^3 \text{ m}^2/\text{kg}$  ( $83.0 \text{ m}^2/\text{g}$ ).

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 and a separate magnetic stirrer may be used.)

5.3 *Magnetic Spinbars*, 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.

5.4 *Microfiltration Membrane Filter Disks*, mixed cellulose ester or cellulose nitrate, 47-mm diameter.

5.5 *Filter Holder*.

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>1</sup> 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.

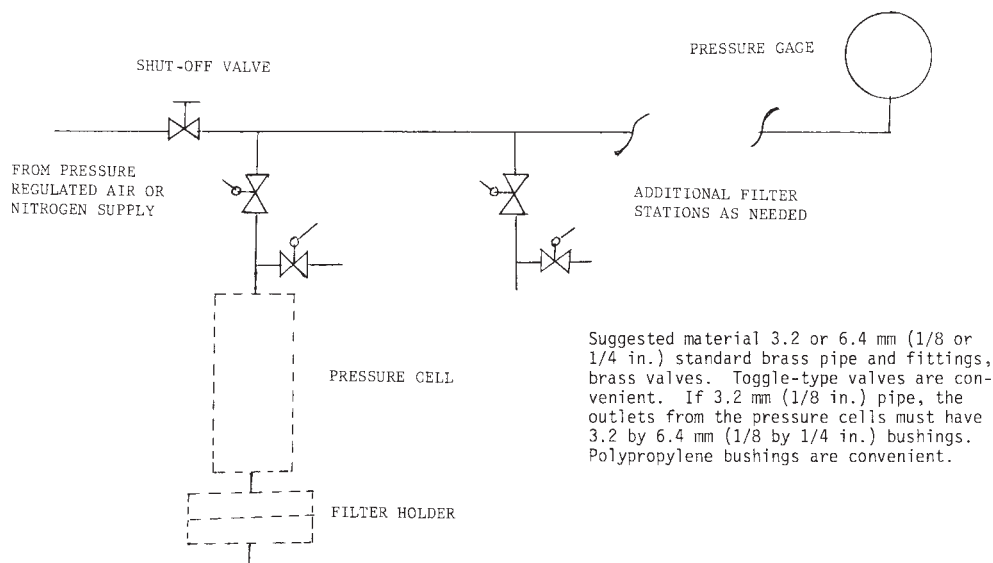
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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

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

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

<sup>5</sup> This equipment is no longer commercially available. As an alternative, the sample vial to be immersed in the ultrasonic bath may be fixed to an automatic shaker in order to assure proper agitation during the ultrasonic treatment.



**FIG. 1 Pressure Filtration Manifold**

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*, 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.

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*, equipped with a photoelectrical detector, preferably working at 660 nm.

5.22 *Titration Beakers*, suitable for the 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>6</sup> Other grades may be used, 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 (KH<sub>2</sub>PO<sub>4</sub>), 4.260 g of dibasic sodium phosphate (Na<sub>2</sub>HPO<sub>4</sub>) and 1.169 g of sodium chloride (NaCl) in 1 dm<sup>3</sup> of distilled water. Alternatively, this prepared solution may be purchased.

6.4 *CTAB Standard Solution (0.0100 M)*—Dissolve 3.64 g of reagent-grade CTAB<sup>7</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. (**Warning**—Storage of the solution below 22°C will result in slow crystallization of the CTAB.)

<sup>6</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*.

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

6.5 *Aerosol OT*<sup>4,8</sup> *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>9</sup> equals 83.0 × 10<sup>3</sup> m<sup>2</sup>/kg (83.0 m<sup>2</sup>/g) surface area.

6.8 *Distilled or Deionized Water*.

6.9 *Octylphenoxy Polyethoxyethanol (Triton X-100*<sup>10</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 ± 1°C.

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 10.3-10.8 and the titration in accordance with 10.9.1 or 10.9.2.

8.4 Plot the titration volumes,  $V (= y)$ , against the sample masses,  $W (= x)$  and determine the slope,  $b$ , (cubic centimetres per gram) and the volume axis intercept,  $V_o$  (cubic centimeters) by linear regression.

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<sup>3</sup> m<sup>2</sup>/kg (m<sup>2</sup>/g),
- $V_o$  = 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 × 10<sup>3</sup> m<sup>2</sup>/kg.

<sup>8</sup> Available from American Cyanamid Co., Process Chemicals Dept., Wayne, NM 07470. Also available as Catalog No. A-349 from Fisher Scientific Co.

<sup>9</sup> Available from Laboratory Standards and Technologies LLC, 227 Somerset, Borger, TX 79007, website: www.carbonstandard.com.

<sup>10</sup> Triton X-100 is a registered trademark of Rohm and Haas Co.

8.7 The five data points from the standardization should be

**TABLE 1 Surface Area Values for SRB-5 Series**

Material	STSA (m <sup>2</sup> /g)	CTAB (m <sup>2</sup> /g)
SRB A5 (N135)	124.6	128.2
SRB B5 (N330)	73.1	77.4
SRB C5 (N220)	111.7	117.3
SRB D5 (N762)	27.1	29.4
SRB E5 (N660)	34.3	37.5
SRB F5 (N683)	37.1	40.9

inserted into this formula to check the reliability of the standardization. CTAB SA of 83.0 ± 1 × 10<sup>3</sup> m<sup>2</sup>/kg (83.0 ± 1 m<sup>2</sup>/g) 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 10.3 and 10.4.

## 9. Calibration

9.1 The CTAB solution ages rather quickly and the final results are affected by this aging phenomenon. Therefore, it is recommended that a standard reference black (SRB) with a CTAB surface area similar to the sample being analyzed be run on a frequent basis to monitor the condition of the solutions. If necessary, prepare a new calibration curve in accordance with 8.1 to 8.7.

9.2 The CTAB and STSA values for the SRB Series 5 materials are given in Table 1.

9.3 During the certification of the SRB-6 series, no CTAB data was obtained because STSA has become the preferred method for measuring external surface area and it is the intention of Test Method D24 to discontinue the CTAB method. More recently it has been requested that SRB-6 data be made available for users of the CTAB test method in order to assist them in switching to the STSA methodology. Therefore, based on the regression analysis of SRB-5 data, estimated CTAB values were calculated for the SRB Series 6 materials using Eq 2.

$$CTAB = STSA * 1.02 + 2.64 \quad (2)$$

9.4 The predicted CTAB values for the SRB-6 series are given in Table 2.

## 10. Procedure

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

NOTE 3—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,  $W$ .

**TABLE 2 Estimated CTAB Surface Area for SRB-6 Series**

Material	STSA (m <sup>2</sup> /g)	CTAB (m <sup>2</sup> /g)
SRB A6 (N134)	135.7	140.7
SRB B6 (N220)	105.4	109.8
SRB C6 (N326)	79.2	83.2
SRB D6 (N762)	29.6	32.7
SRB E6 (N660)	35.1	38.3
SRB F6 (N683)	34.1	37.3

10.2 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, $10^{-3}$ m <sup>2</sup> /kg (m <sup>2</sup> /g)	Sample Mass, g
N100 Series	125 to 150	0.30
N200	100 to 130	0.35
N300	75 to 105	0.40
N351-N440	50 to 75	0.60
N500-N600	35 to 50	0.90
N700	25 to 30	1.35

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

10.3 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.

10.4 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 4—If a combination magnetically stirred ultrasonic dispersion bath is not available, there are two alternatives to assure a proper dispersion of the sample: (1) immerse the sample vial in the ultrasonic bath and attach the vial with a clamp to a mechanical shaker; or (2) immerse the sample in the ultrasonic bath and shake it manually for 1 min, place the vial on the magnetic stirrer, stir for 1 min and repeat this cycle two more times for a total time of 6 min.

NOTE 5—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.

10.5 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.

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

NOTE 6—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.

10.7 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 6). Agitate or stir the collected filtrate to ensure uniformity.

NOTE 7—In case of black filtrate due to a filter leak, discard the sample; do not refilter.

10.8 Proceed immediately to either 10.9.1 or 10.9.2.

10.9 *Titration:*

10.9.1 *Test Method A—Automatic Titration:*

10.9.1.1 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 8—If Atmast automatic titration equipment is employed, adjust titrant flow rate to 6 cm<sup>3</sup>/min, per manufacturer's recommendation.

10.9.1.2 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.).

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

NOTE 9—For convenience, 50 cm<sup>3</sup> of a diluted (1:10) solution of Triton X-100 can be added in one single step (combine 10.9.1.2 and 10.9.1.3).

10.9.1.4 Pipet a 10.00-cm<sup>3</sup> aliquot of the CTAB solution from 10.7 to be titrated into the beaker, taking care to avoid formation of excess foam.

10.9.1.5 Place the beaker into the automatic titrator sample well and adjust the stirrer to produce a vortex without foaming. Make sure there are no air bubbles in the light path of the photoelectrical detector.

10.9.1.6 Start the titration following the manufacturer's instructions. The titrator will stop automatically as the endpoint is reached. Depending on the equipment used, either the result of surface area or the added volume of the titrant will be displayed.

10.9.1.7 Remove the sample beaker and rinse the photoelectrical detector (model to be immersed into the fluid), stirrer and admission tube.

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.

10.9.2 *Test Method B—Manual Turbidity Titration:*

10.9.2.1 Same as 10.9.1.1.

10.9.2.2 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.

10.9.2.3 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.

10.9.2.4 Same as 10.9.1.3.

10.9.2.5 Same as 10.9.1.4.

10.9.2.6 Place a white filter paper on top of a magnetic stirrer and place beaker with red cross and contents from 10.9.2.3, 10.9.2.4 and 10.9.2.5 on the filter paper. Adjust the spin rate so that the vortex is not more than 5-mm deep.

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



10.9.2.8 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.

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

10.9.2.10 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.

## 11. Calculation

11.1 Calculate the CTAB surface area to the nearest  $0.1 \times 10^3 \text{ m}^2/\text{kg}$  ( $0.1 \text{ m}^2/\text{g}$ ) as follows:

$$S = \frac{(V_0 - V)}{W} \times \frac{SA_{ITRB}}{-b} \quad (3)$$

where:

- $S$  = CTAB surface area,  $10^3 \text{ m}^2/\text{kg}$  ( $\text{m}^2/\text{g}$ ),
- $V$  = 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.

## 12. Report

12.1 Report the following information:

12.1.1 Proper identification of the carbon black sample and

12.1.2 Result obtained from an individual determination, reported to the nearest  $0.1 \times 10^3 \text{ m}^2/\text{kg}$  ( $0.1 \text{ m}^2/\text{g}$ ).

## 13. Precision and Bias

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

13.2 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 4.

13.3 A type 1 interlaboratory precision program was conducted as detailed in Table 3. Both repeatability and reproducibility represent short term (daily) testing conditions. The

**TABLE 3 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	SRB D4 (N762)	28
September 1997	SRB A5 (N135)	25
March 1998	N550	21

**TABLE 4 Precision Parameters for D 3765 CTAB Surface Area, (Type 1 Precision)**

Units	$10^3 \text{ m}^2/\text{kg}$ ( $\text{m}^2/\text{g}$ )					
	Material	Mean Level	Sr	(r)	SR	(R)
SRB D4 (N762)		25.1	0.22	2.5	0.59	6.7
N650		38.4	0.48	3.5	0.74	5.5
N550		43.5	0.35	2.3	0.57	3.7
IRB#6 (N330)		77.4	0.50	1.8	1.10	4.0
N234		118.1	0.68	1.6	2.28	5.5
SRB A5 (N135)		128.2	0.51	1.1	1.59	3.5
Average		71.8				
Pooled Values			0.48	1.9	1.30	5.1

testing was performed using two operators in each laboratory performing the test once on each material on each of two days (total of four tests).

13.4 The results of the precision calculations for this test are given in Table 4. The materials are arranged in ascending “mean level” order.

13.5 *Repeatability*—The **pooled relative** repeatability, ( $r$ ), of this test has been established as 1.9 %. Any other value in Table 4 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 4 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.

13.6 *Reproducibility*—The **pooled relative** reproducibility, ( $R$ ), of this test has been established as 5.1 %. Any other value in Table 4 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 3 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

13.7 *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.

## 14. Keywords

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

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