INTERNATIONAL

Designation: D1510 − 21

Standard Test Method for Carbon Black—Iodine Adsorption Number[1]

This standard is issued under the fixed designation D1510; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method covers the determination of the iodine adsorption number of carbon black.

1.1.1 Method A is the original test method for this determination and Method B is an alternate test method using automated sample processing and analysis.

1.2 The iodine adsorption number of carbon black has been shown to decrease with sample aging. Iodine Number reference materials have been produced that exhibit stable iodine number upon aging. One or more of these reference materials are recommended for daily monitoring (x-charts) to ensure that the results are within the control limits of the individual reference material. Use all Iodine Number reference materials from a set for standardization of iodine testing (see Section 8) when target values cannot be obtained.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

E969 Specification for Glass Volumetric (Transfer) Pipets

2.2 *European Standards:*[3]

ISO/EN/DIN 8655-3 Piston-operated volumetric apparatus - Part 3: Piston burettes

3. Summary of Test Methods

3.1 In Test Method A, a weighed sample of carbon black is treated with a portion of standard iodine solution and the mixture shaken and centrifuged. The excess iodine is then titrated with standard sodium thiosulfate solution, and the adsorbed iodine is expressed as a fraction of the total mass of black.

3.2 In Test Method B, a weighed sample of carbon black is treated with a portion of standard iodine solution using an automated sample processor where the mixture is stirred, settled and aliquoted for automatic titration. The excess iodine is titrated with standard sodium thiosulfate solution, and the adsorbed iodine is expressed as a fraction of the total mass of black.

4. Significance and Use

4.1 The iodine adsorption number is useful in characterizing carbon blacks. It is related to the surface area of carbon blacks and is generally in agreement with nitrogen surface area. The presence of volatiles, surface porosity, or extractables will influence the iodine adsorption number. Aging of carbon black can also influence the iodine number.

5. Apparatus

5.1 *Vials,* glass, optically clear type, with polyethylene stoppers, 45 cm^3.

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[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 Carbon Black Surface Area and Related Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, CP 56, CH-1211 Geneva 20, Switzerland, http://www.iso.org.

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

5.3 *Buret,* either of the following may be used:

5.3.1 *Digital Buret,* 25-cm^3 capacity, with 0.01-cm^3 increment counter and zero reset control, or

5.3.2 *Buret,* glass 25-cm^3, Class A, side-arm filling, graduated in 0.05 cm^3 and with automatic zero.

5.4 *Repetitive Dispenser,* 25-cm^3 capacity, ±0.1% reproducibility and calibrated to within ±0.03-cm^3 accuracy.

5.5 *Balance,* analytical, with 0.1-mg sensitivity.

5.6 *Centrifuge,* with minimum speed of 105 rad/s (1000 r/min).

5.7 *Volumetric Flask,* 2000-cm^3 with standard taper stopper.

5.8 *Funnel,* large diameter, with standard taper joint to fit the 2000-cm^3 flask.

5.9 *Glass Bottle,* amber, 2000-cm^3, with standard taper stopper.

5.10 *Glass Jug,* approximate capacity 20-dm^3.

5.11 *Stirrer,* approximately 300 by 300 mm for mixing.

5.12 *Stirrer,* approximately 100 by 100 mm for titrating.

5.13 *Desiccator.*

5.14 *Miscellaneous Class A Glassware,* and equipment necessary to carry out the test as written.

5.15 *Mechanical Shaker,* with at least 1 in. stroke length and a minimum of 240 strokes/min.

5.16 *Automatic Titrator.*

5.17 *Redox Electrode,* combined platinum ring electrode with an Ag/AgCl/KCl reference electrode and a ceramic frit.

5.18 *Volumetric Flask,* 500 cm^3 with standard taper stopper.

5.19 *Flask,* 250 cm^3 with ground glass stopper.

5.20 *Automatic Sample Processor and Titration Apparatus,* equipped with disposable filter.[4]

6. Reagents and Solutions

6.1 *Purity of Reagents*—Unless otherwise stated, all chemicals shall be of reagent grade.

6.2 The preparation of the solutions listed below is described in Annex A1. Pre-mixed 0.04728 *N* iodine solution and 0.0394 *N* sodium thiosulfate may be purchased from commercial sources. It is recommended that the normality of pre-mixed solutions be verified before use.

6.3 *Iodine Solution,* $c(I_2)$ = 0.02364 mol/dm^3 (0.04728 *N*), containing 57.0 g potassium iodide Kl per dm^3.

[4] The sole source of supply of the apparatus known to the committee at this time is Brinkmann Instruments, Inc., One Cantiague Rd., PO Box 1019, Westbury, NY 11590-0207. The sole source of supply of the filter (disposable filter part #17594 K 5 µm Minisart with luer lock outlet) known to the committee at this time is Sartorius Stedim North America Inc., 131 Heartland Blvd., Edgewood, NY 11717. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

6.4 *Potassium Iodate Solution,* $c(KIO_3)$ = 0.00657 mol/dm^3 (0.0394 *N*) containing 45.0 g potassium iodide per dm^3.

6.5 *Potassium Dichromate Solution,* $c(K_2Cr_2O_7)$ = 0.006567 mol/cm^3 (0.0394 *N*), containing 1.932 g potassium dichromate (certified/traceable primary standard) per dm^3. (**Warning—**Potassium dichromate is carcinogenic.)

6.6 *Sodium Thiosulfate Solution,* $c(Na_2S_2O_3)$ = 0.0394 mol/dm^3 (0.0394 *N*), containing 5 cm^3 n-amyl alcohol per dm^3.

6.7 *Sulfuric Acid,* 10 %.

6.8 *Soluble Starch Solution,* 1 %, containing 0.02 g salicylic acid per dm^3.

6.9 *Deionized Water.*

7. Standardization of Solutions

7.1 *Sodium Thiosulfate,* 0.0394 *N* (±0.00008):

7.1.1 Use potassium dichromate solution as follows:

7.1.1.1 Measure approximately 20 cm^3 of 10 % potassium iodide (see A1.4) solution into a small graduated cylinder and transfer to a 250 cm^3 iodine flask with a ground glass stopper.

7.1.1.2 Measure approximately 20 cm^3 of 10 % sulfuric acid solution (see A1.5) into a small graduated cylinder and add to the KI solution in the iodine flask. The mixture should remain colorless.

NOTE 1—If a yellow color should develop, discard this KI solution.

7.1.1.3 Using a 20 cm^3 pipet, transfer 20 cm^3 of standard 0.0394 N potassium dichromate solution (see A1.8) into the 250 cm^3 iodine flask, replace stopper, swirl, and place in the dark for 15 min.

7.1.1.4 Titrate the contents of the iodine flask against the new sodium thiosulfate solution following 7.1.3 or 7.1.4.

7.1.2 Use potassium iodate/iodide solution as follows:

7.1.2.1 Pipet exactly 20 cm^3 of 0.0394 *N* potassium iodate/iodide solution into a 250-cm^3 iodine flask.

7.1.2.2 Measure approximately 5 cm^3 of 10 % sulfuric acid into a small graduated cylinder and add to the iodate/iodide solution.

7.1.2.3 Cap immediately and mix thoroughly.

7.1.2.4 Titrate the contents of the iodine flask against the new sodium thiosulfate solution following 7.1.3 or 7.1.4.

7.1.3 *Digital Buret:*

7.1.3.1 Switch the digital buret to fill mode, fill the reservoir with unstandardized sodium thiosulfate solution, and flush the inlet and delivery tubes.

7.1.3.2 Change to the titrate mode and zero the counter.

7.1.3.3 Add sodium thiosulfate until the contents of the iodine flask are a pale yellowish (potassium iodate) or pale yellowish-green (potassium dichromate). Wash the buret tip and the walls of the flask with water.

7.1.3.4 Add 5 drops of starch solution to the flask.

7.1.3.5 Continue adding sodium thiosulfate dropwise until the blue or blue-violet color almost disappears.

7.1.3.6 Wash the tip and walls of the flask with water, then advance the counter in 0.01-cm^3 increments. Continue this sequence until the endpoint is reached, indicated by a colorless (potassium iodate) or sea-green (potassium dichromate) solution.

7.1.3.7 Record the titration value and repeat from 7.1.1 or 7.1.2 for a duplicate determination.

7.1.3.8 Calculate the normality of the sodium thiosulfate solution as in 7.1.5 and proceed as in 7.1.6. If the titration is made to standardize the iodine solution as described in 7.2 calculate the normality of the iodine solution as in 7.2.1.2 and proceed as in 7.2.1.3.

7.1.4 *Glass Buret:*

7.1.4.1 Using a conventional glass buret, fill the buret with unstandardized sodium-thiosulfate solution and flush 2 to 3 cm^3 through the tip.

7.1.4.2 Adjust to the mark and titrate to a pale yellowish (potassium iodate) or pale yellowish-green (potassium dichromate).

7.1.4.3 Wash the buret tip and the walls of the flask with water.

7.1.4.4 Add 5 drops of starch solution to the iodine flask.

7.1.4.5 Continue adding sodium thiosulfate dropwise until the endpoint is reached, indicated by a colorless (potassium iodate) or sea-green (potassium dichromate) solution.

7.1.4.6 Record the titration value to the nearest 0.025 cm^3 and repeat from 7.1.1 or 7.1.2 for a duplicate determination.

NOTE 2—To achieve maximum performance from a glass buret, it is necessary to use a small magnifier and to read to the nearest 0.025 cm^3.

7.1.4.7 Calculate the normality of the sodium thiosulfate solution as in 7.1.5 and proceed as in 7.1.6. If the titration is made to standardize the iodine solution as described in 7.2 calculate the normality of the iodine solution as in 7.2.1.2 and proceed as in 7.2.1.3.

7.1.5 Calculate the normality of the sodium thiosulfate solutions as follows:

$$N = 20\,(0.0394)/T \tag{1}$$

where:

N = normality, and
T = titration volume, cm^3.

7.1.6 If N is not equal to 0.0394, adjust the solution in the following manner: if the solution is too strong, add water (2.5 cm^3 water per dm^3 sodium thiosulfate solution for each 0.0001 N over 0.0394); if the solution is too weak, add solid sodium thiosulfate (0.025 g solid sodium thiosulfate per dm^3 sodium thiosulfate solution for each 0.0001 N under 0.0394).

7.2 *Iodine Solution* 0.04728 N (±0.00003)—This solution may be standardized against the secondary standard sodium-thiosulfate solution (see A1.3) standardized as in 7.1.

7.2.1 Use sodium thiosulfate solution as follows:

7.2.1.1 Pipet exactly 20 cm^3 of iodine solution into a 250-cm^3 iodine flask and cap. Continue as in 7.1.3 or 7.1.4.

7.2.1.2 Calculate the normality of the iodine solution as follows:

$$N = (0.0394)\ T/20 \tag{2}$$

where:

N = normality, and
T = cm^3 of 0.0394 N sodium thiosulfate solution.

7.2.1.3 If N is not equal to 0.04728 N, adjust solution in the following manner: if the solution is too concentrated, add water (2.1 cm^3 water per dm^3 iodine solution for each 0.0001 N over 0.04728); if the solution is too diluted, add iodine (12.7 mg iodine per dm^3 iodine solution for each 0.0001 N under 0.04728). (This iodine may be more conveniently dispensed from a concentrated solution.)

8. Normalization Using Iodine Number Reference Materials

8.1 The SRB HT reference materials (previously known as SRB HT Iodine Standards) are no longer commercially available but may still be in use in some laboratories. A new lot was prepared by the same process as the SBR HT reference materials and was designated as Iodine Number Reference (INR) to be consistent with D24's naming protocol for reference materials. The SRB HT and INR reference materials are each a set of three materials with different reference values. The three materials from either SRB HT or INR reference materials should be used together for normalization. Do not normalize using some materials from both sets.

8.2 When a laboratory cannot obtain target values for all three SRB HT or INR reference materials within established control limits, the user should review recommendations found in Guide D4821. If any one of the three SRB HT or INR reference materials is still outside acceptable control limits, the method described in 8.3 – 8.6 should be used to normalize all test results.

8.3 Test the three SRB HT or INR reference materials four times each.

8.4 Perform a regression analysis using the target value of the SRB HT or INR reference materials (y value) and the individual measured value (x value).

8.5 Normalize the values of all subsequent test results using this regression equation:

$$\text{Normalized value} = (\text{measured value} \times \text{slope}) + y - \text{intercept} \tag{3}$$

8.6 Alternatively, a table of numbers may be generated based on the regression equation to find the correspondence between a measured value and a normalized value.

8.7 Reevaluate the need for normalization whenever replacement apparatus or new lots of iodine or sodium thiosulfate solutions, or both, are put into use.

9. Sampling

9.1 Samples shall be taken in accordance with Practices D1799 and D1900.

10. Blank Iodine Determination

10.1 *Method A—Blank Iodine Determination:*

10.1.1 Make a blank iodine determination by pipeting 20 cm^3 or dispensing 25 cm^3 of 0.04728 N iodine solution into a 125-cm^3 Erlenmeyer flask and titrating with 0.0394 N sodium thiosulfate as in 11.10.1, 11.10.2, or 11.10.3.

10.1.2 A 25-cm^3 blank must be multiplied by 0.8 for use in the formula of 13.1.

10.1.3 Make a duplicate blank determination and use the average of the two in the calculations.

NOTE 3—A duplicate blank determination need be run only once each day, unless new solutions are introduced during the day.

10.1.4 If both solutions are within acceptable limits, the blank will measure 24.00 ± 0.09 cm^3. If not, the normalities of one or both solutions should be rechecked. If, after the recheck of solutions, normalities are still outside the acceptable limits refer to 7.2.1.3 to adjust iodine solution. See Table 1 for blank tolerance components.

10.1.5 The blank tolerance for a 20 cm^3 volume of iodine solution is defined as the sum of *(1)* titration volume deviation for acceptable variation in both iodine and sodium thiosulfate solution concentrations, and *(2)* dispenser tolerance for Class A 20 mL pipet.

10.1.6 The solution deviation is based on the maximum variation in solution concentrations defined in 7.1 and 7.2. Tolerances for Class A volumetric pipets are from Specification E969.

10.2 *Method B—Blank Iodine Determination:*

10.2.1 Make a blank iodine determination by placing a magnetic stir bar into an empty beaker and place the beaker into the automated sample processor.

10.2.2 Initiate the automatic sample processor and titration apparatus.

10.2.3 Dispense an appropriate volume of 0.04728 *N* iodine solution into the beaker. Treat the blank in the same manner as the sample, refer to Section 12.

NOTE 4—For different size beakers, ensure stir bar covers the bottom surface of beaker for good mixing.

10.2.4 Measures should be taken to ensure adequate purging of the entire system prior to delivering the final aliquot for titration (see Note 5).

NOTE 5—An example of adequate purging of the system is achieved by double rinsing with the current blank solution followed with a distilled water rinse. This can be done in the following manner: *(1)* fill the dosing device, which is equipped with a disposable filter, with an aliquot of the blank solution from the beaker, dispense the entire volume into titration vessel, and pump out into the waste container; *(2)* repeat previous step one more time and fill the dosing device with the final aliquot of blank solution (this aliquot should have an excess amount that will be used to flush the air bubbles, possibly formed during the two previous steps—the volume of aliquot used for titration can vary depending on user's preference (7 to 20 cm^3 has been found satisfactory)); *(3)* dispense a small portion of the blank solution into the reaction vessel, ensure that appropriate amount of the solution is left for titration in the dosing device; and *(4)* clean the reaction (titration) vessel by rinsing with distilled water and pumping out waste repetitively.

10.2.5 Dispense a final aliquot of the blank solution into the reaction vessel for titration and wash the walls of the vessel, stirrer, and redox-electrode with distilled water to ensure that any splashed iodine is washed into the mixture.

10.2.6 Automatically titrate the iodine solution with 0.0394 *N* sodium thiosulfate.

10.2.7 Make duplicate blank determinations. The average of two determinations is to be used in calculations.

TABLE 1 Blank Tolerance Components

Blank Volume cm^3	A. Solution Deviations cm^3	B. Dispenser Tolerance cm^3	Blank Tolerance cm^3
20.00	±0.06	±0.03	±0.09

10.2.8 Blank measurements may be made daily, especially where small solution lots are prepared within a lab. Alternatively, blanks may be measured once per solution lot or other prescribed frequency, for large solution lots which are purchased, and where adequate measures are used to monitor testing such as the daily use of x-charting HT or INR standards.

NOTE 6—For daily blanks, a duplicate blank determination need be run only once each day, unless new solutions are introduced during the day.

NOTE 7—When the particulate filter is changed adequate measures should be taken to saturate the filter with iodine solution. An example of an adequate measure found to be satisfactory includes running a minimum of five blanks. The fourth and fifth blank are then averaged for the final blank value and use the average of the two in the calculations. If the filter has not been changed use the average of the first and second blanks for calculations.

10.2.9 Blank tolerances are found in Table 2 for different volumes of iodine solution. A blank tolerance is defined as the sum of *(1)* titration volume deviation for acceptable variation in both iodine and sodium thiosulfate solution concentrations, and *(2)* dispenser tolerance for a piston-operated volumetric apparatus.

10.2.10 A blank tolerance can be calculated from the linear equation as follows:

$$Y = 0.0056x + 0.0059 \quad (4)$$

where:

Y = tolerance ±, and
x = aliquot volume, mL.

10.2.11 Blank tolerances for Method B are also found in Fig. 1. The function for solution deviation only and solution deviation plus dispenser tolerance are included for reference.

10.2.12 The solution deviation is based on the maximum variation in solution concentrations defined in 7.1 and 7.2. Tolerances for piston-operated volumetric apparatus are from ISO/EN/DIN 8655-3.

11. Sample Preparation and Iodine Number Determination—Method A

11.1 Dry an adequate sample of carbon black for 1 h, in a gravity-convection oven set at 125°C, in an open container of suitable dimensions, so that the depth of the black is no more than 10 mm. Cool to room temperature in a desiccator before use.

11.2 Weigh a mass of the dried sample into a glass vial as shown by the following table. All masses must be to the nearest 0.001 g in case of iodine numbers from 0 to 520.9 and to the nearest 0.0001 g in case of iodine numbers from 521.0 and above.

TABLE 2 Blank Tolerances

Blank Volume cm^3	A. Solution Deviations cm^3	B. Dispenser Tolerance cm^3	Blank Tolerance cm^3
20.00	±0.064	±0.054	±0.118
10.00	±0.032	±0.027	±0.059
6.00	±0.019	±0.024	±0.043
1.00	±0.003	±0.007	±0.010

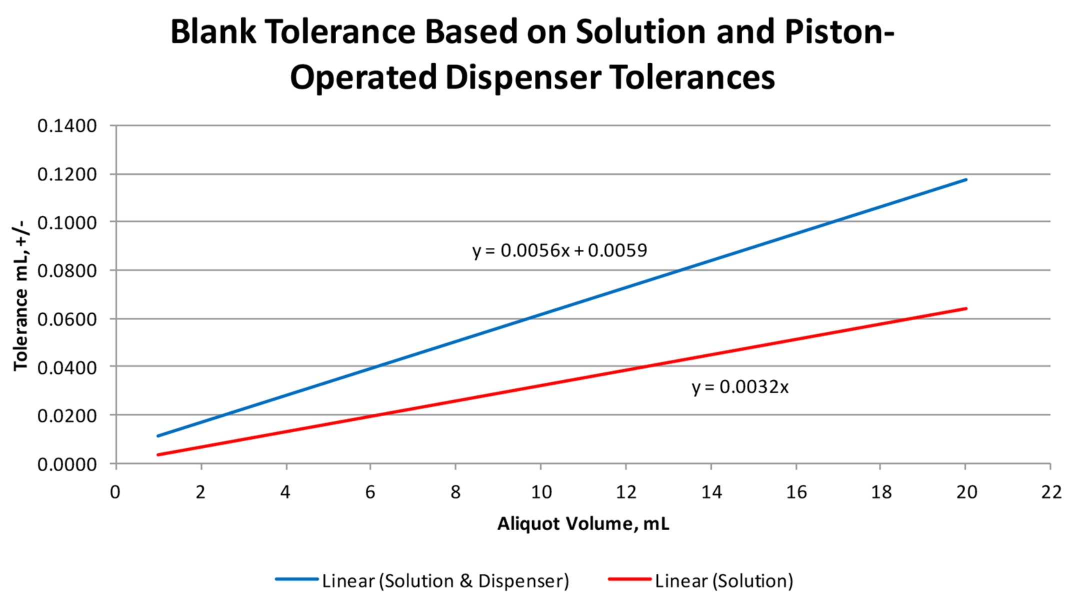

FIG. 1 Blank Tolerances for Method B as a Function of Aliquot Volume

Iodine Number	Sample Mass (g)	Ratio I_2: Sample Mass
0–130.9	0.500	50:1
131.0–280.9	0.250	100:1
281.0–520.9	0.125	200:1
521.0 and above	0.0625	400:1

11.3 Use the sample mass determined by the expected iodine number. If the result falls either above or below the range shown for that sample size, retest using the sample mass specified in 11.2 for the range into which it has fallen.

NOTE 8—Unagitated, unpelleted carbon black may be densified, if desired, before drying, prior to weighing.

11.4 The sample mass table given in 11.2 pertains to the 25 cm^3 iodine solution as given in 11.5. Different volumes of iodine solution and of sample masses are permissible only if the iodine solution to sample mass ratio is kept the same as that given by the table in 11.2. The sample mass must be kept to 1.000 g maximum. Should the sample mass and corresponding volume of iodine solution be increased, then a glass vial with a volume that is at least two times the amount of iodine solution used for the test should be used in order to preserve the efficiency of the shaking.

11.5 Pipet (or dispense from a calibrated repetitive dispenser) 25 cm^3 of 0.04728 *N* I_2 solution into the glass vial containing the sample and cap immediately.

11.6 Secure the vial in the mechanical shaker and shake for 1 min at a minimum of 240 strokes/min.

11.7 Centrifuge immediately for 1 min for pelleted black and 3 min for loose black.

NOTE 9—Make sure that the carbon black is separated from the iodine solution in such a way that enough carbon black free iodine solution is available for the titration. In case that carbon black particles are still visible in the iodine solution to be titrated, repeat the sample preparation and increase the centrifugation speed.

11.8 Decant immediately. If more than one sample is being analyzed, the solution should be decanted into small flasks or clean, dry vials and capped immediately.

11.9 Pipet 20 cm^3 of solution into a 250-cm^3 Erlenmeyer flask and titrate with standardized 0.0394 *N* sodium thiosulfate solution using either the digital or glass buret as described in 11.10.

11.10 *Titration of Iodine Solution:*

11.10.1 *Using a Digital Buret:*

11.10.1.1 Switch to the fill mode, fill the buret reservoir with solution, and flush the inlet and delivery tubes.

11.10.1.2 Change to the titrate mode, zero the counter, and clean the tip with tissue.

11.10.1.3 Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

11.10.1.4 Add 5 drops of starch solution.

11.10.1.5 Continue adding sodium thiosulfate dropwise until the blue or blue-violet color almost disappears.

11.10.1.6 Wash the tip and walls of the flask with water and then advance the counter in 0.01-cm^3 increments. Continue this sequence until the endpoint is reached as indicated by a colorless solution.

11.10.1.7 Record the buret reading to the nearest 0.01 cm^3.

11.10.2 *Using a Conventional Glass Buret:*

11.10.2.1 Remove any adherent drop on the tip of the buret by gently toughing the drop with the wall of a clean flask. The flask may be used several times by toughing a clean part of the wall to remove further drops prior to titration. Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

11.10.2.2 Add 5 drops of starch solution.

11.10.2.3 Continue adding sodium thiosulfate dropwise until the endpoint is reached as indicated by a colorless solution.

11.10.2.4 Record the titration volume to the nearest 0.025 cm^3.

11.10.3 *Using an Auto-titrator:*

11.10.3.1 Two redox equivalence point titration methods should be programmed into the autotitrator:

(1) A method to store two blank determinations as an average blank value, and

(2) A method to analyze samples for iodine number.

NOTE 10—Follow the recommendations of the manufacturer when setting the parameters. For good repeatability of the test, special care should be taken when defining the criteria for the detection of the equivalence point.

11.10.3.2 Pipet 20 cm^3 of test solution into an appropriate sample container, place the container on the auto-titrator, and wash the walls of the container, stirrer, and redox electrode with distilled water.

11.10.3.3 Run titration method using standardized 0.0394 *N* sodium thiosulfate solution.

11.10.3.4 Method should report equivalence point volume to at least 0.01 cm^3.

12. Sample Preparation and Iodine Number Determination—Method B

12.1 Dry an adequate sample of carbon black for a minimum of 1 h, in a gravity-convection oven set at 125°C, in an open container of suitable dimensions, so that the depth of the black is no more than 10 mm. Cool to room temperature in a desiccator before use.

12.2 Weigh a mass of the dried sample into an appropriate beaker as shown by the following table. All masses must be to the nearest 0.001 g, and the sample mass must be kept to 1.000 g maximum. This sample mass table pertains to 50 cm^3 of iodine solution.

Iodine Number	Sample Mass (g)	Ratio I_2: Sample Mass
0–130.9	1.000	50:1
131.0–280.9	0.500	100:1
281.0–520.9	0.250	200:1
521.0 and above	0.125	400:1

12.3 Different volumes of iodine solution and sample masses are permissible as long as the appropriate ratio of iodine to sample mass is maintained as indicated in 12.2. A sample mass table for 25 cm^3 of iodine solution is shown below. The sample mass must be kept to 0.5 g maximum.

Iodine Number	Sample Mass (g)	Ratio I_2: Sample Mass
0–130.9	0.500	50:1
131.0–280.9	0.250	100:1
281.0–520.9	0.125	200:1
521.0 and above	0.0625	400:1

12.4 Use the sample mass determined by the expected iodine number. If the result falls either above or below the range shown for that sample size, retest using the sample mass specified in 12.2 or 12.3 for the range into which it has fallen.

NOTE 11—Unagitated, unpelleted carbon black may be densified, if desired, before drying, prior to weighing.

12.5 Two redox equivalence point titration methods should be programmed into the automatic sample processor and titration apparatus: *(1)* a method to store two blank determinations as an average blank value as described in 10.2; *(2)* a method to analyze samples for iodine number using calculations found in Section 13.

NOTE 12—Users may choose to titrate different volumes of blank and sample aliquots for testing; also it is possible that equipment functionality may differ. Follow the recommendations of the manufacturer when setting parameters for rinsing times, fill rates, start/stop volumes for titration, etc. For good repeatability of the test, special care should be taken when defining the criteria for the detection of the equivalence point. End-point criterion set to 25 and EP recognition set to "greatest" have been found sufficient.

12.6 Carefully place a magnetic stir bar in the beaker with the dried sample and place the beaker into the automated sample processor. Take adequate precautions to prevent any loss of sample from the beaker.

12.7 Initiate the automatic sample processor and titration apparatus.

12.8 Dispense 50 cm^3 or appropriate volume of 0.04728 *N* I_2 solution into the beaker containing the sample and stir bar using a calibrated repetitive dispenser (dosing device).

12.9 Stir the sample for 3.0 min then turn off the stir motor.

12.10 Allow the slurry to settle for a minimum of 30 s. Longer settling time may be needed for non-pelleted carbon black. Settling times may vary due to additional time caused by the sample processor waiting for previous titrations to complete in the reaction vessel.

12.11 Take adequate steps to completely purge the dosing system and reaction vessel. An example cleaning procedure is found in Note 5.

12.12 Dispense a final aliquot of iodine solution into the reaction vessel for titration using a calibrated repetitive dispenser (dosing system) which includes a disposable 5 µm filter to remove particulates of carbon black. Wash the walls of the reaction vessel, stirrer, and redoxelectrode with distilled water.

12.13 Automatically titrate the iodine solution using 0.0394 *N* sodium thiosulfate.

NOTE 13—A disposable filter's useful life has been reported at approximately 50 samples, but may vary with sample type and physical form. Whenever the filter is changed always insure adequate measures are taken to saturate the filter as described in Note 7.

12.14 Report the equivalence point volume to at least 0.01 cm^3 and calculate iodine number to 0.1 mg/g.

12.15 Since Method B may give slightly different results than Method A, the SRB HT or INR reference materials should be analyzed with each lot of both iodine and sodium thiosulfate solutions. If the measured results of the three SRB HT or INR reference materials are not within stated control limits, a normalization using either the three SRB HT or the three INR reference materials (as described in Section 8) should be applied to all test results.

13. Calculation

13.1 Calculate the iodine adsorption number to the nearest 0.1 g/kg as follows:

$$I = [(B - S)/B] \times (V/W) \times N \times 126.91 \quad (5)$$

where:

I = iodine adsorption number, grams of iodine/kilograms of carbon black expressed as g/kg,
B = cm^3 of sodium thiosulfate required for the blank,
S = cm^3 of sodium thiosulfate required for the sample,
V = calibrated volume of the 25-cm^3 iodine pipet or dispenser,

W = grams of carbon black sample,
N = normality of the iodine solution, meq/cm^3, and
126.91 = equivalent mass of iodine mg/meq.

Using the units shown above results in units of milligrams of iodine/grams of carbon black, which is equivalent to grams of iodine/kilograms of carbon black.

14. Report

14.1 Report the following information:

14.1.1 Proper identification of the sample,

14.1.2 Sample mass, and

14.1.3 Result obtained from an individual determination, reported to the nearest 0.1 g/kg.

15. Precision and Bias

15.1 These precision statements have been prepared in accordance with Practice D4483-99. Refer to this practice for terminology and other statistical details.

15.2 An Interlaboratory precision program (ITP) information was conducted as detailed in Table 3. 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*.

15.3 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 in 15.2. 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 3.

15.3.1 The Iodine Adsorption Number of Carbon Black may decrease over time. Therefore, the SRB material shall not be used for test method validation or to monitor testing. It is strictly recommended to use INR reference material for Iodine Adsorption Number monitoring.

15.4 A type 1 interlaboratory precision program was conducted. 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 material on each of two days (total of four tests). The number of participating laboratories is listed in Table 3.

15.5 The results of the precision calculations for this test are given in Table 3. The materials are arranged in ascending "mean level" order.

15.6 *Repeatability*—The pooled relative repeatability, *(r)*, of this test method has been established as 1.5 %. Any other value in Table 3 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 3 must be suspected of being from different populations and some appropriate action taken.

NOTE 14—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, and so forth, which generated the two test results.

15.7 *Reproducibility*—The pooled relative reproducibility, *(R)*, of this test has been established as 6.1 %. Any other value in Table 3 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.

15.8 *Bias*—Bias is the difference between a test value and a reference value. However, for this test method, bias has not been determined.

TABLE 3 Precision Parameters for D1510, Method A & B (Type 1 Precision)[A]

Units			g/kg						
Material	Period	Number of Laboratories (M/H/L)[B]	Mean Level	Sr	r	(r)	SR	R	(R)
SRB-9A	Mar 2013	76(1/1/2)	78.1	0.43	1.23	**1.6**	1.00	2.84	**3.6**
SRB-9B	Mar 2016	85(1/2/1)	82.0	0.37	1.05	**1.3**	0.96	2.71	**3.3**
SRB-9D	Mar 2018	73(0/1/1)	19.5	0.30	0.84	**4.3**	0.89	2.51	**12.9**
SRB-9E	Aug 2016	79(2/3/1)	36.2	0.29	0.81	**2.2**	0.68	1.93	**5.3**
SRB-9F	Mar 2015	73(1/2/2)	36.1	0.37	1.05	**2.9**	0.64	1.80	**5.0**
SRB-9G	Aug 2017	71(1/3/0)	7.7	0.25	0.69	**9.0**	0.83	2.34	**30.2**
SRB-9B2	Mar 2019	75(0/2/1)	80.7	0.35	0.98	**1.2**	1.19	3.37	**4.2**
SRB-9C	Aug 2019	77(1/2/0)	140.3	0.50	1.41	**1.0**	3.65	10.33	**7.4**
SRB-9A2	Aug 2018	75(1/2/1)	83.5	0.33	0.93	**1.1**	0.90	2.55	**3.1**
SRB-9H	Mar/Apr 2020	73(2/2/0)	128.0	0.40	1.12	**0.9**	1.51	4.28	**3.3**
Average			69.2						
Pooled Values				0.36	1.03	**1.5**	1.49	4.21	**6.1**

[A] Preferred precision shown in bold text.
[B] M = number of outliers for Mean; H = number of outliers for High variation; L = number of outliers for Low variation.