Designation: D1510 – 16a

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. New SRB HT Iodine Standards have been produced that exhibit stable iodine number upon aging. One or more of these SRB HT Iodine Standards are recommended for daily monitoring (x-charts) to ensure that the results are within the control limits of the individual standard. Use all SRB HT Iodine Standards 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 and health practices and determine the applicability of regulatory limitations prior to use.*

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.

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

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

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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.

5.3.1 *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.

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

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[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.

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 SRB HT Iodine Standards

8.1 When a laboratory cannot obtain target values for all three SRB HT Iodine Standards within established x-chart tolerances, the user should review recommendations found in Guide D4821. If any one of the three SRB HT Iodine Standards is still outside acceptable tolerances, the method described in 8.2 – 8.5 should be used to normalize all test results.

8.2 Test the three SRB HT Iodine Standards four times each.

8.3 Perform a regression analysis using the target value of the SRB HT Iodine Standards (y value) and the individual measured value (x value).

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

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

8.5 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.

8.6 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

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

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.

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:

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

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.

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

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

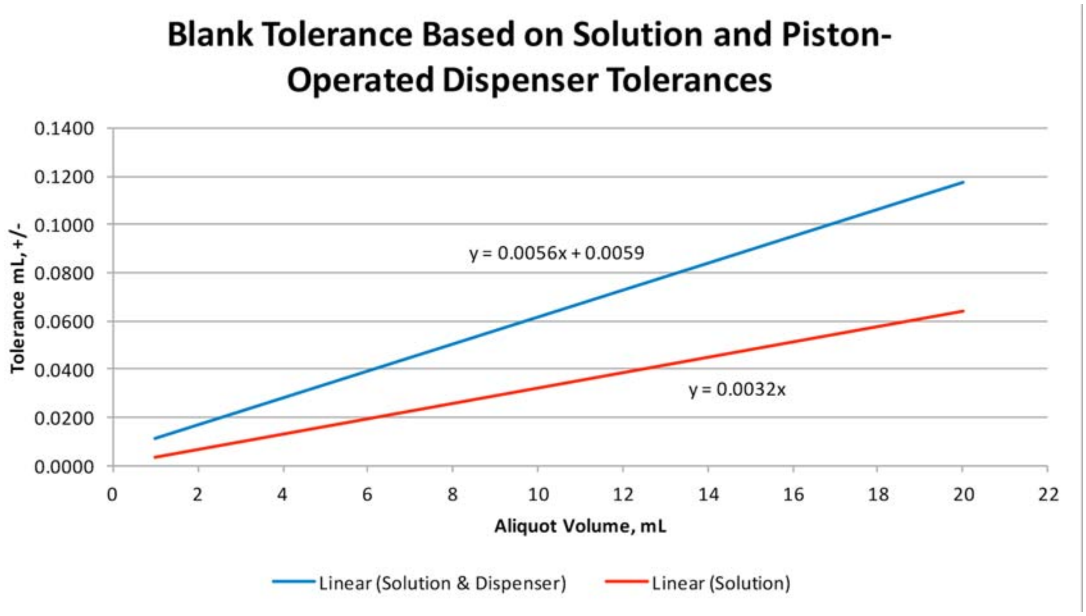

FIG. 1 Blank Tolerances for Method B as a Function of Aliquot Volume

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 carbon black is separated sufficiently from the iodine solution to obtain a sufficient quantity of carbon black free iodine solution to be titrated. Increase the centrifugation speed in order to obtain an adequate separation, if required.

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 standards should be analyzed with each lot of both iodine and sodium thiosulfate solutions. If the measured results of the three HT or INR Standards are not within stated xchart tolerances, a normalization using either HT or INR standards (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:

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

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. 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 4.

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 4. 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 1.6 %. 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.

TABLE 4 Precision Parameters for D1510, Iodine Number Methods A & B (Type 1 Precision)[A]

Units		g/kg					
Material	Mean Level	Sr	r	**(r)**	SR	R	**(R)**
SRB-8B2	146.3	0.57	1.61	**1.1**	1.70	4.80	**3.3**
SRB-8C	138.8	0.68	1.92	**1.4**	2.11	5.96	**4.3**
SRB-8B	135.6	0.68	1.91	**1.4**	1.93	5.47	**4.0**
SRB-8A	80.5	0.36	1.03	**1.3**	0.88	2.49	**3.1**
SRB-8A2	78.1	0.88	2.49	**3.2**	1.33	3.78	**4.8**
SRB-8F	35.9	0.32	0.89	**2.5**	0.57	1.61	**4.5**
SRB-8E	35.8	0.32	0.91	**2.5**	0.60	1.71	**4.8**
SRB-8D	21.7	0.28	0.80	**3.7**	0.55	1.55	**7.1**
Average	84.1						
Pooled Values		0.48	1.35	**1.6**	1.32	3.72	**4.4**

[A] The preferred precision values are shown in bold text.

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 4.4 %. 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 4 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

15.8 *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.

16. Keywords

16.1 carbon black; iodine adsorption number

TABLE 3 SRB8 ITP Information

SRB8 Material	Grade	Producer	Test Period	Number of Labs (M/H/L) D1510
SRB-8A	N326	Continental	March 2008	61 (0/2/1)
SRB-8A2	N326	Continental	March 2013	71 (1/1/2)
SRB-8B	N134	Cabot	June 2009	66 (1/2/2)
SRB-8B2	N134	Cabot	March/April 2014	39 (3/3/4)
SRB-8C	HS Tread	Columbian	September 2010	64 (1/2/5)
SRB-8D	LS Carcass	Cabot	March 2009	67 (1/2/2)
SRB-8E	N660	Orion	September 2008	57 (0/3/0)
SRB-8F	N683	Orion	March 2010	68 (1/1/0)
SRB-8F2	N683	Orion	March 2015	60 (1/2/2)
SRB-8G[A]	N990	Cancarb	Last half of 1996	N/A

[A] SRB-8G was produced and approved in the last half of 1996 as SRB-5G and has continued to be included in the current SRB sets since that time. At the time it was produced and approved it was D24's practice to only publish the within-laboratory standard deviation, Sr, and associated limits. The between-laboratory standard deviation, *SR*, was never published and since the data is no longer available it is not possible to calculate or publish the *SR* values and corresponding limits. The SRB G material was only tested for NSA, STSA, and OAN per the test method version available in 1996.

ANNEXES

(Mandatory Information)

A1. PREPARATION OF SOLUTIONS

NOTE A1.1—The designated iodine solution strength for running the iodine test is 0.04728 *N*, which is made from a stronger (10 X) solution.

NOTE A1.2—Standardization of the working 0.04728 *N* iodine solution will always be necessary and should be done with sodium-thiosulfate secondary standard (see 7.2).

A1.1 Iodine Solution, 0.4728 *N*

A1.1.1 Place approximately 1200 g of potassium iodide (KI) in a glass tray and dry it an oven at 125°C for 1 h. Place it in a desiccator and allow to cool down to room temperature.

A1.1.2 Weigh 1140 ± 0.2 g (±0.02 %) of dried KI into a 1000-cm^3 beaker.

A1.1.3 Transfer approximately 300 g of KI from the 1000 cm^3 beaker into a new 500-cm^3 beaker.

A1.1.4 Add enough water to cover the 300 g of KI (approximately 400 cm^3).

A1.1.5 Add enough water to cover the remaining KI in the 1000 cm^3 beaker (approximately 700 cm^3).

A1.1.6 Place a clean stir-bar into each beaker and stir them on stir plates until KI is fully dissolved (this could take considerable time). If desired, beakers can be placed in 40 to 45°C water baths, with stirring, to speed up the dissolution of the KI.

A1.1.7 Remove both beakers from the stirrers (or water bath, or both) and allow them to stand until the solutions reach ambient temperature (if the heated water bath was not used the solution will be cold and also will require additional standing to reach ambient temperature).

A1.1.8 Obtain the weight to 3 decimal places of an empty tall-form 1000 cm^3 weighing bottle fitted with ground glass stopper (W_1). Remove the glass stopper then tare the balance with the bottle on it.

A1.1.9 Using only a porcelain spoon, add 120.00 ± 0.07 g (±0.06 %) of iodine (I_2) into the weighing bottle on the balance. Insert the stopper into the bottle.

A1.1.10 Remove the bottle containing the iodine from the balance, and tare the balance. Weigh the bottle containing the iodine plus stopper to 3 decimal places (W_2). Calculate the weight of iodine as ($W_2 – W_1$).

A1.1.11 If the weight of iodine is outside the range of 120.00 ± 0.07 g, adjust the weight of iodine, then reweigh the bottle containing the iodine plus stopper to 3 decimal places (W_2) and again calculate the new iodine weight as ($W_2 – W_1$). Repeat this until the iodine weight falls in the correct range.

A1.1.12 Quantitatively transfer the KI solution from the 1000 cm^3 beaker into the weighing bottle containing I_2 crystals. Use 50 cm^3 of deionized water to quantitatively rinse residual KI solution into the weighing bottle containing the iodine crystals. Repeat this rinse three times.

A1.1.13 Add a clean stir-bar into the 1000 cm^3 weighing bottle, insert the stopper, and stir for approximately 30 min. It is good practice to cover the outside of the bottle with aluminum foil to prevent reaction of I_2 with light.

A1.1.14 Using a clean funnel quantitatively transfer the I_2 solution from the 1000 cm^3 weighing bottle into a clean 2000 cm^3 volumetric flask. Use the KI solution in the 500-cm^3 beaker to quantitatively rinse residual I_2 solution into the volumetric flask. Use the entire KI solution in the 500-cm^3 beaker. Then use about 50 cm^3 of fresh deionized water to quantitatively rinse residual KI solution from the 500-cm^3 beaker and the stir-bar into the 2000-cm^3 volumetric flask. Repeat this rinse with water three times.

A1.1.15 Adjust the volume in the 2000 cm^3 flask to the mark with deionized water, invert the flask once or twice, and let it sit for 1 to 2 min to allow solution to drain from the neck of the flask. An additional adjustment to the mark might be needed (approximately 1 to 2 cm^3 of deionized water).

A1.1.16 Cover the outside of the volumetric flask with aluminum foil to prevent ambient light from decomposing the iodine. Place a clean stir-bar into the flask and place the flask on the stirrer. Continue stirring at medium speed for at least 2 h.

A1.1.17 Let the flask stand overnight. Stir for 5 min.

A1.1.18 If it is desired to prepare 20 dm^3 of 0.04728 *N* working iodine solution, go to A1.2.

A1.1.19 If it is desired to prepare smaller than 20-dm^3 portions of 0.04728 *N* working iodine solution, then transfer the solution to the 2000 cm^3 amber bottle and store in a cool dark place. Use volumetric pipettes and volumetric flasks to prepare exactly 1:10 dilutions.

A1.2 Iodine Solution, 0.04728 *N*

A1.2.1 To make 20 dm^3 of 0.04728 *N* iodine solution use the 0.4728 *N* iodine solution in A1.1.18.

A1.2.2 Secure a clean graduated 20-dm^3 jug on a stir plate. Ensure the stir plate is capable of holding the full weight of the jug plus 20 dm^3 of liquid securely.

A1.2.3 Take a clean 1-dm^3 graduated glass cylinder and fill it to the 1-dm^3 mark with fresh deionized water. This cylinder will be used to add all necessary water to obtain the 1:10 dilution.

A1.2.4 Using a clean funnel quantitatively transfer the I_2 solution from the 2000-cm^3 volumetric flask into a clean graduated 20-dm^3 jug. Ensure that no solution is lost during this step.

A1.2.5 Add approximately 200 cm^3 of deionized water from the graduated cylinder into the volumetric flask. Carefully swirl to wash remaining I_2 solution off the walls of the flask. Carefully transfer this entire wash into the 20 dm^3 jug. Continue washing until no color remains in the flask or on the funnel, using water from the graduated cylinder. Add any remaining water that is left in the graduated cylinder to the 20-dm^3 jug.

A1.2.6 A total of 18 dm^3 of water needs to be added to the 20-dm^3 jug. Repeat A1.2.2 until all the water is added, including water used for rinsing the volumetric flask from the I_2 solution. To ensure the accurate volume of water is added to the jug, it is important to use the graduated cylinder and to keep track of the number of transfers. Use the graduated marks on the jug only as a reference point to ensure the correct number of transfers was made. Do not rely on the graduated marks alone to measure the volume, as these marks are not accurate enough to be used in this procedure.

A1.2.7 Add a large clean stir bar into the jug. Close the jug ensuring that the stopper or cover is resistant to iodine (for example, Teflon or polypropylene).

A1.2.8 Cover the outside of the volumetric flask with aluminum foil to prevent ambient light from decomposing the iodine. Stir at medium speed for at least 2 h. Sometimes it can be difficult to initiate the stirring. The stirring can be started manually by using a clean long Teflon or glass rod.

NOTE A1.3—Do not rinse residual I_2 solution off the rod into the jug!

A1.2.9 Let the jug stand overnight. Stir for 30 min. Store in a cool dark place.

A1.3 Sodium Thiosulfate, 0.0394 *N*

A1.3.1 Add about 4 dm^3 of water to a 20-dm^3 glass jug (calibrated for 16 dm^3) that has been placed on a magnetic stirrer.

NOTE A1.4—Calibration of the 20-dm^3 jug, either by weight or by measuring 2-dm^3 increments from a graduated cylinder, should be done as carefully as possible in order to minimize the number of standardization titrations.

A1.3.2 Weigh 156.5 g of sodium thiosulfate ($Na_2\ S_2O_3 \cdot 5H_2O$) and wash through the filling funnel into the jug.

A1.3.3 Add 80 cm^3 of n-amyl alcohol to the jug.

A1.3.4 Stir until the crystals are dissolved.

A1.3.5 Add water to the 16-dm^3 mark.

A1.3.6 Place the jug on the magnetic stirrer and stir for 1 to 2 h.

A1.3.7 Age for 1 to 2 days, then stir for 0.5 h before using.

A1.4 Potassium Iodide (KI), 10 % Solution

A1.4.1 Weigh approximately 10 g of potassium iodide into a small stoppered flask or bottle.

A1.4.2 Fill a graduated cylinder with 90 cm^3 of water and transfer to the small stoppered flask or bottle.

A1.4.3 Mix thoroughly until dissolved.

NOTE A1.5—It is possible for the KI solution to be oxidized and release iodine. Discard the KI solution if any yellow color is observed.

A1.5 Sulfuric Acid, 10 % Solution

A1.5.1 Fill a graduated cylinder with 90 cm^3 of deionized water and transfer to a 250-cm^3 Erlenmeyer flask.

A1.5.2 Measure about 7 cm^3 of concentrated sulfuric acid into a small graduated cylinder or beaker.

A1.5.3 Very carefully pour the acid into the 250-cm^3 flask of water and swirl gently to mix. Rinse the graduate with diluted acid from the 250-cm^3 flask.

A1.5.4 Allow the acid to cool to ambient temperature before using.

A1.6 Soluble Starch, 1 % Solution

A1.6.1 Into a 100-cm^3 beaker weigh about 1 g of soluble starch and 0.002 g of salicylic acid.

A1.6.2 Add enough water to make a thin paste while stirring with a stirring rod.

A1.6.3 Add about 100 cm^3 of water to a 250-cm^3 beaker and bring to a boil on a hotplate.

A1.6.4 Add the starch paste to the water, stir, and continue to boil for 2 to 3 min.

A1.6.5 Allow the starch solution to cool. Add 2 to 3 g KI and stir to dissolve.

A1.7 Potassium Iodate/Iodide Solution, 0.0394 *N* (Primary Standard)

A1.7.1 Dry an adequate quantity of analytical grade potassium iodate for 1 h in an oven set at a temperature of 125°C. Allow to cool to ambient temperature in a desiccator.

A1.7.2 Dissolve 45 g of potassium iodide in about 200 cm^3 of water contained in a 1000-cm^3 volumetric flask.

A1.7.3 Weigh 1.4054 g of the freshly dried potassium iodate and add to the iodide solution in the 1000-cm^3 flask.

A1.7.4 Make up to the mark and cap the flask.

A1.8 Potassium Dichromate Solution, 0.0394 *N* (Primary Standard)

NOTE A1.6—Potassium dichromate is available as a certified oxidimetric primary standard in a finely divided granular form. All precautions should be used to ensure the accuracy of the prepared solution.

A1.8.1 Dry an adequate quantity of potassium dichromate for 1 h in an oven set at 125°C. Allow to cool to ambient temperature in a desiccator.

A1.8.2 Weigh 0.9660 g of the dried potassium dichromate ($K_2Cr_2O_7$), then using a standard-taper joint funnel, add this to a 500-cm^3 volumetric flask.

A1.8.3 Add about 200 cm^3 of water to the volumetric flask.

A1.8.4 Stopper and shake the flask until all crystals are dissolved.

A1.8.5 Add water to the mark and shake well.

A1.8.6 Stopper the flask and age for 30 min prior to use.

A2. DETERMINATION OF POTASSIUM IODIDE CONTENT[5]

A2.1 This method describes the determination of potassium iodide content by evaporation of water and iodine. Since the concentration of potassium iodide (and iodine) is crucial for obtaining correct test results for iodine adsorption number, this method allows a verification of the correct concentration of KI, 57.0 ± 0.5 g/L (see 6.3).

A2.2 Apparatus

A2.2.1 *Beaker,* 50 cm^3 (50 mL).

A2.2.2 *Desiccator*

A2.2.3 *Repetitive Dispenser,* 25 cm^3 capacity, ±0.1 % reproducibility and calibrated within ±0.003 cm^3 accuracy.

A2.2.4 *Pipet,* 25 cm^3 (25 mL), class A (an alternative to A2.2.3).

A2.2.5 *Fume Hood*

A2.2.6 *Heating Plate*

A2.2.7 *Balance,* analytical, with 0.1 mg sensitivity.

A2.3 Procedure

A2.3.1 Place two clean 50 cm^3 (50 mL) beakers in an oven at 125°C for 1 h.

A2.3.2 Remove the beakers and place them into a desiccator to cool for 30 min.

A2.3.3 Weigh the beakers and record the mass (m_1).

A2.3.4 Dispense 25.0 cm^3 (25 mL) of iodine solution (0.04728 N) into each beaker.

A2.3.5 Set the beakers on a hot plate under a hood. Heat at a temperature below the boiling point in order to slowly remove the water and the iodine as a vapor. If the solution splashes or boils out, discard and start again.

NOTE A2.1—When the evaporation is near completion the solid remaining will change from a brown color (iodine) to white (potassium iodide).

A2.3.6 When the liquid is completely gone place the beakers in an oven at 125°C for 1 h.

A2.3.7 Remove the beakers and place them into a desiccator to cool for 30 min.

A2.3.8 Weigh the beakers with the remaining solid and record the mass (m_2).

A2.4 Calculate the concentration of potassium iodide as follows:

$$KI = (m_2 - m_1)/0.025 \qquad (A2.1)$$

where:

KI = potassium iodide content, g/dm^3 (g/L)
m_1 = mass of the beaker, g
m_2 = mass of the beaker with the residue, g
0.025 = volume of iodine/potassium iodide solution used for the test, dm^3 (L)

A2.5 Report the average of the single determinations to the nearest 0.1 g/dm^3 (g/L).

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D24-1041.