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Standard Test Method for Carbon Black—Iodine Adsorption Number¹

This standard is issued under the fixed designation D 1510; 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 Department of Defense.

1. Scope

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

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. These SRB HT Iodine Standards are recommended for daily monitoring (x-charts) of testing and for standardization of iodine testing (see Section 8) when target values cannot be obtained.

1.3

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

D 1799 Practice for Carbon Black Sampling Packaged Shipments

D 1900 Practice for Carbon Black Sampling Bulk Shipments

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D 4821 Guide for Carbon Black Validation of Test Method Precision and Bias

3. Summary of Test Method

3.1 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.

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 cm³.

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-cm³ capacity, with 0.01-cm³ increment counter and zero reset control, or

5.3.2 *Buret*, glass 25-cm³, Class A, side-arm filling, graduated in 0.05 cm³ and with automatic zero.

5.4 *Repetitive Dispenser*, 25-cm³ capacity, ±0.1% reproducibility and calibrated to within ±0.03-cm³ accuracy.

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

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

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.

- 5.7 *Volumetric Flask*, 2000-cm³ with standard taper stopper.
- 5.8 *Funnel*, large diameter, with standard taper joint to fit the 2000-cm³ flask.
- 5.9 *Glass Bottle*, amber, 2000-cm³, with standard taper stopper.
- 5.10 *Glass Jug*, approximate capacity 20-dm³.
- 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 cm³ with standard taper stopper.
- 5.19 *Flask*, 250 cm³ with ground glass stopper.

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 0.0473 *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*, $c(I_2) = 0.02364 \text{ mol/dm}^3$ (0.0473 *N*), containing 57.0 g potassium iodide KI per dm³.
 - 6.4 *Potassium Iodate Solution*, $c(KIO_3) = 0.00657 \text{ mol/dm}^3$ (0.0394 *N*) containing 45.0 g potassium iodide per dm³.
 - 6.5 *Potassium Dichromate Solution*, $c(K_2Cr_2O_7) = 0.006567$ (0.0394 *N*), containing 1.932 g potassium dichromate (certified/traceable primary standard) per dm³. (**Warning**—Potassium dichromate is carcinogenic.)
 - 6.6 *Sodium Thiosulfate Solution*, $c(Na_2S_2O_3) = 0.0394 \text{ mol/dm}^3$ (0.0394 *N*), containing 5 cm³ n-amyl alcohol per dm³.
 - 6.7 *Sulfuric Acid*, 10 %.
 - 6.8 *Soluble Starch Solution*, 1 %, containing 0.02 g salicylic acid per dm³.
 - 6.9 *Water*.

7. Standardization of Solutions

- 7.1 *Sodium Thiosulfate*, 0.0394 *N* (± 0.00008):
 - 7.1.1 Use potassium dichromate solution as follows:
 - 7.1.2 Measure approximately 20 cm³ of 10 % potassium iodide (see A1.4) solution into a small graduated cylinder and transfer to a 250 cm³ iodine flask with a ground glass stopper.
 - 7.1.3 Measure approximately 20 cm³ 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.
 - 7.1.4 Using a 20 cm³ pipet, transfer 20 cm³ of standard 0.0394 *N* potassium dichromate solution (see A1.8) into the 250 cm³ iodine flask, replace stopper, swirl, and place in the dark for 15 min.
 - 7.1.5 Titrate the contents of the iodine flask against the new sodium thiosulfate solution following 7.2.6 and 7.2.7.
- 7.2 *Sodium Thiosulfate*, 0.0394 *N* (± 0.00008):
 - 7.2.1 Use potassium iodate/iodide solution as follows:
 - 7.2.2 Pipet exactly 20 cm³ of 0.0394 *N* potassium iodate/iodide solution into a 250-cm³ iodine flask.
 - 7.2.3 Measure approximately 5 cm³ of 10 % sulfuric acid into a small graduated cylinder and add to the iodate/iodide solution.
 - 7.2.4 Cap immediately and mix thoroughly.
 - 7.2.5 Titrate the contents of the iodine flask against the new sodium thiosulfate solution following 7.2.6 and 7.2.7.
 - 7.2.6 *Digital Buret*:
 - 7.2.6.1 Switch the digital buret to fill mode, fill the reservoir with unstandardized sodium thiosulfate solution, and flush the inlet and delivery tubes.
 - 7.2.6.2 Change to the titrate mode and zero the counter.
 - 7.2.6.3 Add sodium thiosulfate until the contents of the iodine flask is a pale yellowish-green. Wash the buret tip and the walls of the flask with water.
 - 7.2.6.4 Add 5 drops of starch solution to the flask.
 - 7.2.6.5 Continue adding sodium thiosulfate dropwise until the blue or blue-violet color almost disappears.
 - 7.2.6.6 Wash the tip and walls of the flask with water, then advance the counter in 0.01-cm³ increments. Continue this sequence until the endpoint is reached, indicated by a colorless (potassium iodate) or sea-green (potassium dichromate) solution.
 - 7.2.6.7 Record the titration value and repeat from 7.2.2 for a duplicate determination.
 - 7.2.6.8 Calculate the normality of the sodium thiosulfate solutions as follows:

$$N = \frac{20(0.0394)}{T} \quad (1)$$

where:

N = normality, and

T = titration volume, cm^3 .

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

7.2.7 Glass Buret:

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

7.2.7.2 Adjust to the mark and titrate to a pale yellowish-green endpoint.

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 .

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

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

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

7.2.7.6 Record the titration value to the nearest 0.025 cm^3 and repeat from 7.2.7.1 for a duplicate determination.

7.2.7.7 Calculate the normality of the sodium thiosulfate solution as in 7.2.6.8.

7.3 Iodine Solution 0.0473 N (± 0.00003)—This solution may be standardized against the secondary standard sodium-thiosulfate solution (see A1.3) standardized as in 7.2.

7.3.1 Use sodium thiosulfate solution as follows:

7.3.1.1 Pipet exactly 20 cm^3 of iodine solution into a 250-cm^3 iodine flask and cap. Continue as in 7.2.6 or 7.2.7.

7.3.1.2 Calculate the normality of the iodine solution as follows:

$$N = \frac{(0.0394)T}{20} \quad (2)$$

where:

N = normality, and

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

7.3.1.3 If N is not equal to 0.0473 N , adjust solution in the following manner: if the solution is too concentrated, add water ($2.1 \text{ cm}^3/\text{dm}^3$ for each 0.0001 N over 0.0473); if the solution is too diluted, add iodine ($12.7 \text{ mg}/\text{dm}^3$ for each 0.0001 N under 0.0473). (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 D 4821. 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 standardize all test results.

8.2 Test the three ASTM HT Iodine Standards in duplicate to establish the average measured value. Additional values are added periodically, typically on a daily or weekly basis or when solutions are changed. A rolling average can be calculated from the latest four results.

8.3 Perform a regression analysis using the standard value of the standard (y value) and the rolling average of the measured value (x value).

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

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

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.

9. Sampling

9.1 Samples shall be taken in accordance with Practices D 1799 and D 1900.

10. Test Procedure

10.1 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.

10.2 Weigh a mass of the dried sample into a glass vial as shown by the following table. All masses must be to the nearest 0.0001 g .

Iodine Number	Sample Mass (g)	Ratio I_2 : Sample Mass
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0–130.9	0.5000	50:1
131.0–280.9	0.2500	100:1
281.0–520.9	0.1250	200:1
521.0 and above	0.0625	400:1

10.2.1 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 10.2 for the range into which it has fallen.

NOTE 3—Unagitated, unpelleted carbon black may be densified, if desired, before drying, prior to weighing.

10.2.2 The sample mass table given in 10.2 pertains to the 25 cm³ iodine solution as given in 10.3. 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 10.2. The sample mass must be kept to 1.0000 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.

10.3 Pipet (or dispense from a calibrated repetitive dispenser) 25 cm³ of 0.0473 N I₂ solution into the glass vial containing the sample and cap immediately.

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

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

10.6 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.

10.7 Pipet 20 cm³ of solution into a 250-cm³ Erlenmeyer flask and titrate with standard 0.0394 N sodium thiosulfate solution using either the digital or glass buret as follows:

10.7.1 *Using a Digital Buret:*

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

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

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

10.7.1.4 Add 5 drops of starch solution.

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

10.7.1.6 Wash the tip and walls of the flask with water and then advance the counter in 0.01-cm³ increments. Continue this sequence until the endpoint is reached as indicated by a colorless solution.

10.7.1.7 Record the buret reading to the nearest 0.01 cm³.

10.7.1.8 Make a blank iodine determination by pipeting 20 cm³ or dispensing 25 cm³ of 0.0473 N iodine solution into a 125-cm³ Erlenmeyer flask and titrating with 0.0394 N sodium thiosulfate as in 10.7.1 or 10.7.2.

10.7.1.9 A 25-cm³ blank must be multiplied by 0.8 for use in the formula of 11.1.

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

NOTE 4—A duplicate blank determination need be run only once each day, unless new solutions are introduced during the day.

10.7.1.11 If both solutions are within acceptable limits, the blank will measure 24.00 ± 0.05 cm³. If not, the normalities of one or both solutions should be rechecked.

10.7.2 *Using a Conventional Glass Buret:*

10.7.2.1 Clean the tip with a tissue. Add sodium thiosulfate until the solution is pale yellow. Wash the buret tip and walls of the flask with water.

10.7.2.2 Add 5 drops of starch solution.

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

10.7.2.4 Record the titration volume to the nearest 0.25 cm³.

10.7.2.5 Make blank determinations as in 10.7.1.8, 10.7.1.10, and 10.7.1.11.

10.7.3 *Using an Auto-titrator:*

10.7.3.1 Two redox equivalence point titration methods should be programmed into the auto-titrator: 1) a method to store two blank determinations as an average blank value; 2) a method to analyze samples for iodine number.

NOTE 5—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.

10.7.3.2 Make duplicate blank determinations as in 10.7.1.8, 10.7.1.9, and 10.7.1.10 to update stored values.

10.7.3.3 Pipet 20 cm³ 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.

10.7.3.4 Run titration method using standard 0.0394 N sodium thiosulfate solution.

10.7.3.5 Method should report equivalence point volume to at least 0.01 cm³ and calculate iodine number to 0.1 mg/g.

11. Calculation

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

$$I = [(B - S)/B] \times (V/W) \times N \times 126.91 \quad (4)$$