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Designation: D 1510 – 02

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

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.

1. Scope

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

1.1.1 Procedure A is the original procedure for this determination.

1.1.2 Procedure B specifies an increased sample mass of carbon black and volume of iodine solution (the ratio is the same as in Procedure A) which results in a simplified formula for the calculation of the iodine number.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 The iodine adsorption number of carbon black has been shown to decrease with sample aging. Standard values for current SRB's may be obtained from the Subcommittee Chairman of D24.61.

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:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias[2]

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 cm^3.

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 1 in. stroke length and capable of 240 strokes/min.

6. Reagents

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

6.2 *Iodine*.

6.3 *Potassium Iodide*.

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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 Adsorptive Properties of Carbon Black.

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[2] *Annual Book of ASTM Standards*, Vol 09.01.

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6.4 *Potassium Dichromate*.
6.5 *Potassium Iodate*.
6.6 *Sodium Thiosulfate*.
6.7 *n-Amyl Alcohol*.
6.8 *Sulfuric Acid*.
6.9 *Soluble Starch*.
6.10 *Salicylic Acid*.
6.11 *Arsenious Oxide*, 1.9995 milliequivalent vials.[3]
6.12 *Water*.

7. Sampling

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

8. Solutions

8.1 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.

8.2 The solutions may be mixed in the laboratory. For instructions on mixing these solutions, refer to Annex A1.

9. Standardization of Solutions

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

9.1.1 Use potassium dichromate solution (as follows: see A1.1).

9.1.2 Measure approximately 20 cm^3 of 10 % potassium iodide solution into a small graduated cylinder and transfer to a 250-cm^3 iodine flask with a ground glass stopper.

9.1.3 Measure approximately 20 cm^3 of 10 % sulfuric acid solution (see A1.6) 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.

9.1.4 Using a 20-cm^3 pipet, transfer 20 cm^3 of standard 0.0394 *N* potassium dichromate solution into the 250-cm^3 iodine flask, replace stopper, swirl, and place in the dark for 15 min.

9.1.5 Titrate the contents of the iodine flask against the new sodium thiosulfate solution following 9.1.6 or 9.1.7.

9.1.6 *Digital Buret*:

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

9.1.6.2 Change to the titrate mode and zero the counter.

9.1.6.3 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.

9.1.6.4 Add 5 drops of starch solution to the flask.

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

9.1.6.6 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 of the pale sea-green color of chromic sulfate is reached.

9.1.6.7 Record the titration value and repeat from 9.1.4 for a duplicate determination.

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

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

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

9.1.6.9 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 cm^3/dm^3 solution for each 0.0001 *N* over 0.0394); if the solution is too weak, add solid sodium thiosulfate (0.025 g/dm^3 for each 0.0001 *N* under 0.0394).

9.1.7 *Glass Buret*:

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

9.1.7.2 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.

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

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

9.1.7.5 Continue adding sodium thiosulfate dropwise until the blue color is just discharged. There will be a residual sea-green color of chromic sulfate.

9.1.7.6 Record the titration value to the nearest 0.025 cm^3 and repeat from 9.1.7.1 for a duplicate determination.

9.1.7.7 Calculate the normality of the sodium thiosulfate solution as in 9.1.6.8.

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

9.2.1 Use potassium iodate/iodide solution as follows:

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

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

9.2.4 Cap immediately and mix thoroughly. Continue as in 9.1.6-9.1.6.8 or as in 9.1.7-9.1.7.7. (However, note that the endpoint is colorless rather than sea-green in color.)

9.3 *Iodine Solution* 0.0473 *N* (±0.00003)—This solution may be standardized against standard arsenious oxide (As_2O_3) (see 9.3.1) or against the secondary standard sodium-thiosulfate solution (see A1.3) standardized as in 9.1 or 9.2.

9.3.1 Use arsenious oxide solution as follows:

9.3.1.1 Remove the cap and plug from the arsenious-oxide vial and pour the contents into a titrating vessel. Rinse the vial thoroughly and combine the rinse water with contents in the titration vessel.

9.3.1.2 Add approximately 5 g of sodium bicarbonate to the titration vessel.

9.3.1.3 Place the titration vessel on an ultrasonic stirrer for 5 min or until all solids have dissolved.

9.3.1.4 Add 5 cm^3 of starch solution.

9.3.1.5 Titrate with iodine solution to a pale blue endpoint

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[3] Available from Chemical Services Laboratories, P.O. Box 281, Largo, FL 33540.

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that remains blue for 30 s. Record the volume of iodine solution to the nearest 0.01 cm^3.

9.3.1.6 Calculate the normality of the iodine solution as follows:

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

where:

N = normality,
S = milliequivalents As_2O_3 on the vial label, and
T = cm^3 of iodine solution

9.3.2 Use sodium thiosulfate solution as follows:

9.3.2.1 Pipet exactly 20 cm^3 of iodine solution into a 250-cm^3 iodine flask and cap. Continue as in 9.1.6-9.1.6.7 or as in 9.1.7-9.1.7.6.

9.3.2.2 Calculate the normality of the iodine solution as follows:

$$N = \frac{(0.0394)T}{20} \tag{3}$$

where:

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

9.3.2.3 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 cm^3/dm^3 for each 0.0001 N over 0.0473); if the solution is too diluted, add iodine (12.7 mg/dm^3 for each 0.0001 N under 0.0473). (This iodine may be more conveniently dispensed from a concentrated solution.)

10. Procedure A

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
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 cm^3 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 proper capacity should be used in order to preserve the efficiency of the shaking.

10.3 Pipet (or dispense from a calibrated repetitive dispenser) 25 cm^3 of 0.0473 N I_2 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 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 cm^3 of solution into a 250-cm^3 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-cm^3 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 cm^3.

10.7.1.8 Make a blank iodine determination by pipeting 20 cm^3 or dispensing 25 cm^3 of 0.0473 N iodine solution into a 125-cm^3 Erlenmeyer flask and titrating with 0.0394 N sodium thiosulfate as in 10.7.1 or 10.7.2.

(1) A 25-cm^3 blank must be multiplied by 0.8 for use in the formula of 11.1.

10.7.1.9 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.

10.7.1.10 If both solutions are within acceptable limits, the blank will measure 24.00 ± 0.05 cm^3. 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 cm^3.

10.7.2.5 Make blank determinations as in 10.7.1.8, 10.7.1.9, and 10.7.1.10.

11. Calculation—Procedure A

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

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

where:

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