Designation: D1510 – 24

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.

[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.

Current edition approved Jan. 1, 2024. Published February 2024. Originally approved in 1957. Last previous edition approved in 2023 as D1510 – 23a. DOI: 10.1520/D1510-24.

[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 *Shelf Life*—The iodine solution is stable for 1 year when properly stored between 15 to 30°C in airtight glass containers under light protection, for example, amber glass bottles. The thiosulfate solution is stable for 1 year when stored at 15 to 25°C in an airtight container.

6.3 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.

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

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

6.6 *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.)

6.7 *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.

NOTE 1—Instead of using a 0.0394 N sodium thiosulfate solution the use of a 0.05 N sodium thiosulfate solution is also allowed, provided that the actual concentration is used in the calculations. In this case the blank result in 10.1.4 or 10.2.10 will change accordingly.

6.8 *Sulfuric Acid,* 10 %.

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

6.10 *Deionized Water.*

7. Standardization of Solutions

7.1 *Sodium Thiosulfate,* 0.0394 *N* (±0.00015):

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 2—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.

[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.

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 3—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:

$$N = 20\,(0.0394)/T \quad (1)$$

where:

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

7.1.6 If N is not equal to 0.0394 (±0.00015), 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 (±0.00015)—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:

$$N = (0.0394)\,T/20 \quad (2)$$

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 (±0.00015), 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:

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

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:*