



Designation: D4607 – 14 (Reapproved 2021)

Standard Test Method for Determination of Iodine Number of Activated Carbon¹

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1. Scope

1.1 This test method covers the determination of the relative activation level of unused or reactivated carbons by adsorption of iodine from aqueous solution. The amount of iodine absorbed (in milligrams) by 1 g of carbon using test conditions listed herein is called the iodine number.

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

1.3 *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.* Specific hazard statements are given in Section 7.

1.4 *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

C819 Test Method for Specific Surface Area of Carbon or Graphite (Withdrawn 1987)³

D1193 Specification for Reagent Water

D2652 Terminology Relating to Activated Carbon

D2867 Test Methods for Moisture in Activated Carbon

D3860 Practice for Determination of Adsorptive Capacity of Activated Carbon by Aqueous Phase Isotherm Technique

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E287 Specification for Laboratory Glass Graduated Burets

E288 Specification for Laboratory Glass Volumetric Flasks

E300 Practice for Sampling Industrial Chemicals

2.2 *NIST Publication:*

Circular 602 Testing of Glass Volumetric Apparatus⁴

3. Summary of Test Method

3.1 This test method is based upon a three-point adsorption isotherm (see Practice **D3860**). A standard iodine solution is treated with three different weights of activated carbon under specified conditions. The carbon-treated solutions are filtered to separate the carbon from the treated iodine solution (filtrate). Iodine remaining in the filtrate is measured by titration. The amount of iodine removed per gram of carbon is determined for each carbon dosage and the resulting data used to plot an adsorption isotherm. The amount of iodine adsorbed (in milligrams) per gram of carbon at a residual iodine concentration of 0.02 *N* is reported as the iodine number.

3.2 Iodine concentration in the standard solution affects the capacity of an activated carbon for iodine adsorption. Therefore, the normality of the standard iodine solution must be maintained at a constant value (0.100 ± 0.001 *N*) for all iodine number measurements.

3.3 The apparatus required consists of various laboratory glassware used to prepare solutions and contact carbon with the standard iodine solution. Filtration and titration equipment are also required.

4. Significance and Use

4.1 The iodine number is a relative indicator of porosity in an activated carbon. It does not necessarily provide a measure of the carbon's ability to absorb other species. Iodine number may be used as an approximation of surface area for some types of activated carbons (see Test Method **C819**). However, it must be realized that any relationship between surface area and iodine number cannot be generalized. It varies with

¹ This test method is under the jurisdiction of ASTM Committee **D28** on Activated Carbon and is the direct responsibility of Subcommittee **D28.02** on Liquid Phase Evaluation.

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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 The last approved version of this historical standard is referenced on www.astm.org.

⁴ Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, <http://www.nist.gov>.

changes in carbon raw material, processing conditions, and pore volume distribution (see Terminology **D2652**).

4.2 The presence of adsorbed volatiles, sulfur, and water extractables may affect the measured iodine number of an activated carbon.

5. Apparatus

NOTE 1—All volumetric measuring equipment should meet or exceed the requirements of NIST Circular 602. Volumetric glassware meeting these specifications is generally designated as “Class A.” See also Specifications **E287** and **E288**.

5.1 *Analytical Balance*, accuracy ± 0.0001 g.

5.2 *Buret*, 10-mL capacity or 5-mL precision buret.

5.3 *Flasks*, Erlenmeyer 250-mL capacity with ground glass stoppers.

5.4 *Flask*, Erlenmeyer wide-mouthed, 250-mL capacity.

5.5 *Beakers*, assorted sizes.

5.6 *Bottles*, amber, for storage of iodine and thiosulfate solutions.

5.7 *Funnels*, 100-mm top inside diameter.

5.8 *Filter Paper*, 18.5-cm prefolded paper, Whatman No. 2V or equivalent.

5.9 *Pipets*, volumetric type, 5.0, 10.0, 25.0, 50.0, and 100.0-mL capacity.

5.10 *Volumetric Flasks*, 1 L.

5.11 *Graduated Cylinders*, 100 mL and 500 mL.

6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁵ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—References to water shall be understood to mean reagent water conforming to Specification **D1193** for Type II reagent water.

6.3 *Hydrochloric Acid*, concentrated.

6.4 *Sodium Thiosulfate*, ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$).

6.5 *Iodine*, United States Pharmacopeia, resublimed crystals.

6.6 *Potassium Iodide*.

6.7 *Potassium Iodate*, primary standard.

6.8 *Starch*, soluble potato or arrowroot.

6.9 *Sodium Carbonate*.

7. Hazards

7.1 Several potential hazards are associated with conducting this test procedure. It is not the purpose of this standard to address all potential health and safety hazards encountered with its use. The user is responsible for establishing appropriate health and safety practices before use of this test procedure. Determine the applicability of federal and state regulations before attempting to use this test method.

7.2 Personnel conducting the iodine number procedure should be aware of potential safety and health hazards associated with the chemicals used in this procedure. The “Material Safety Data Sheet” (MSDS) for each reagent listed in Section 6 should be read and understood. Special precautions to be taken during use of each reagent are included on the “Material Safety Data Sheet” (MSDS). First aid procedures for contact with a chemical are also listed on its “MSDS.” A “Material Safety Data Sheet” for each reagent may be obtained from the manufacturer. Other safety and health hazard information on reagents used in this procedure is available.^{6,7,8}

7.3 Careful handling and good laboratory technique should always be used when working with chemicals. Avoid contact with hydrochloric acid or acid vapor. Care should also be taken to prevent burns during heating of various solutions during this test procedure.

7.4 The user of this test method should comply with federal, state, and local regulations for safe disposal of all samples and reagents used.

8. Preparation of Solutions

8.1 *Hydrochloric Acid Solution* (5 % by weight)—Add 70 mL of concentrated hydrochloric acid to 550 mL of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

8.2 *Sodium Thiosulfate* (0.100 N)—Dissolve 24.820 g of sodium thiosulfate in approximately 75 ± 25 mL of freshly boiled distilled water. Add 0.10 ± 0.01 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Quantitatively transfer the mixture to a 1-L volumetric flask and dilute to the mark. Allow the solution to stand at least four days before standardizing. The solution should be stored in an amber bottle.

8.3 *Standard Iodine Solution* (0.100 ± 0.001 N)—Weigh 12.700 g of iodine and 19.100 g of potassium iodide (KI) into a beaker. Mix the dry iodine and potassium iodide. Add 2 to 5 mL of water to the beaker and stir well. Continue adding small increments of water (approximately 5 mL each) while stirring until the total volume is 50 to 60 mL. Allow the

⁵ Reagent Chemicals, American Chemical Society Specifications, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁶ The “Chemical Safety Data Sheet” for the subject chemical is available from the Manufacturing Chemists Association, Washington, DC.

⁷ Sax, N. I., *Dangerous Properties of Industrial Materials*, 4th edition, 1975, Van Nostrand Reinhold Company, New York, NY.

⁸ *NIOSH/OSHA Pocket Guide to Chemical Hazards*, 1978, U.S. Department of Labor, Occupational Safety and Health Administration, Washington, DC. Available from U.S. Government Printing Office, Washington, DC.

solution to stand a minimum of 4 h to ensure that all crystals are thoroughly dissolved. Occasional stirring during this 4-h period will aid in the dissolution. Quantitatively transfer to a 1-L volumetric flask and fill to the mark with distilled water. It is important that the standard iodine solution has an iodide-to-iodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

8.4 Potassium Iodate Solution (0.1000 *N*)—Dry 4 or more grams of primary standard grade potassium iodate (KIO₃) at 110 ± 5 °C for 2 h and cool to room temperature in a desiccator. Dissolve 3.5667 ± 0.1 mg of the dry potassium iodate in about 100 mL of distilled water. Quantitatively transfer to a 1-L volumetric flask and fill to the mark with distilled water. Mix thoroughly and store in a glass-stoppered bottle.

8.5 Starch Solution—Mix 1.0 ± 0.5 g of starch with 5 to 10 mL of cold water to make a paste. Add an additional 25 ± 5 mL of water while stirring to the starch paste. Pour the mixture, while stirring, into 1 L of boiling water and boil for 4 to 5 min. This solution should be made fresh daily.

9. Standardization of Solutions

9.1 Standardization of 0.100 *N* Sodium Thiosulfate—Pipet 25.0 mL of potassium iodate (KIO₃) solution from 8.4 into a 250-mL titration (or wide-mouthed Erlenmeyer) flask. Add 2.00 ± 0.01 g of potassium iodide (KI) to the flask and shake the flask to dissolve the potassium iodide crystals. Pipet 5.0 mL of concentrated hydrochloric acid into the flask. Titrate the free iodine with sodium thiosulfate solution until a light yellow color is observed in the flask. Add a few drops of starch indicator (8.5) and continue the titration dropwise until one drop produces a colorless solution. Determine sodium thiosulfate normality as follows:

$$N_1 = (P \cdot R) / S \quad (1)$$

where:

N_1 = sodium thiosulfate, *N*,
 P = potassium iodate, mL,
 R = potassium iodate, *N*, and
 S = sodium thiosulfate, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 *N*.

9.2 Standardization of 0.100 ± 0.001 *N* Iodine Solution—Pipet 25.0 mL of iodine solution (8.3) into a 250-mL wide-mouthed Erlenmeyer flask. Titrate with standardized sodium thiosulfate (9.1) until the iodine solution is a light yellow color. Add a few drops of starch indicator and continue titration dropwise until one drop produces a colorless solution. Determine the iodine solution normality as follows:

$$N_2 = (S \cdot N_1) / I \quad (2)$$

where:

N_2 = iodine, *N*,
 S = sodium thiosulfate, mL,
 N_1 = sodium thiosulfate, *N*, and

I = iodine, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 *N*. The iodine solution concentration must be 0.100 ± 0.001 *N*. If this requirement is not met, repeat 8.3 and 9.2.

10. Procedure

10.1 The procedure applies to either powdered or granular activated carbon. When granular carbon is to be tested, grind a representative sample (see Practice E300) of carbon until 60 wt % (or more) will pass through a 325-mesh screen and 95 wt % or more will pass through a 100-mesh screen (U.S. sieve series, see Specification E11). Carbon received in the powdered form may need additional grinding to meet the particle size requirement given above.

10.2 Dry the ground carbon from 10.1 in accordance with Test Method D2867. Cool the dry carbon to room temperature in a desiccator.

10.3 Determination of iodine number requires an estimation of three carbon dosages. Paragraph 11.4 describes how to estimate the carbon dosages to be used. After estimating carbon dosages, weigh three appropriate amounts of dry carbon to the nearest milligram. Transfer each weighed sample of carbon to a clean, dry 250-mL Erlenmeyer flask equipped with a ground glass stopper.

10.4 Pipet 10.0 mL of 5 wt % hydrochloric acid solution into each flask containing carbon. Stopper each flask and swirl gently until the carbon is completely wetted. Loosen the stoppers to vent the flasks, place on a hot plate in a fume hood, and bring the contents to a boil. Allow to boil gently for 30 ± 2 s to remove any sulfur which may interfere with the test results. Remove the flasks from the hot plate and cool to room temperature.

10.5 Pipet 100.0 mL of 0.100 *N* iodine solution into each flask. Standardize the iodine solution just prior to use. Stagger the addition of iodine to the three flasks so that no delays are encountered in handling. Immediately stopper the flasks, and shake the contents vigorously for 30 ± 1 s. Quickly filter each mixture by gravity through one sheet of folded filter paper (Whatman No. 2V or equivalent) into a beaker. Filtration equipment must be prepared in advance so no delay is encountered in filtering the samples.

10.6 For each filtrate, use the first 20 to 30 mL to rinse a pipet. Discard the rinse portions. Use clean beakers to collect the remaining filtrates. Mix each filtrate by swirling the beaker and pipet 50.0 mL of each filtrate into a clean 250-mL Erlenmeyer flask. Titrate each filtrate with standardized 0.100 *N* sodium thiosulfate solution until the solution is a pale yellow. Add 2 mL of the starch indicator solution and continue the titration with sodium thiosulfate until one drop produces a colorless solution. Record the volume of sodium thiosulfate used.

11. Calculation

11.1 The capacity of a carbon for any adsorbate is dependent upon the concentration of the adsorbate in solution. The