

ASTM Designation: D4607-94

Standard Test Method for
Determination of Iodine Number of Activated Carbon

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 1g of carbon using test conditions listed herein is called the iodine number.

1.2 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. Specific hazard statements are given in Section 7.

2. Referenced Documents

2.1 ASTM Standards:

C819 Test Method for Specific Surface Area of Carbon or Graphite

D1193 Specification for Reagent Water

D2652 Terminology Relating to Activated Carbon

D2867 Test Method for Moisture in Activated Carbon

D3860 Practices for Determination of Adsorptive Capacity of Carbon by Isotherm Technique

E11 Specification for Wire-Cloth Sieves for Testing Purposes

E177 Practice for Use for 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 Apparatus

3. Summary of Test Method

3.1 This test method is based upon a three-point adsorption isotherm (see Practices 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 weight and the resulting data used to plot an isotherm. The amount of iodine adsorbed (in milligrams) per gram of carbon at a residual iodine concentration of 0.02N 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±0.001N) 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 changes in carbon raw material, processing conditions, and pore volume distribution (see Definitions 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-All volumetric measuring equipment should meet or exceed the requirements of NIST Circular 602. Volumetric glassware meeting those specifications is generally designated as "Glass 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, 1L

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. 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.

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 70ml of concentrated hydrochloric acid to 550ml of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

8.2 Sodium Thiosulfate (0.100N)--- Dissolve 24.820g sodium thiosulfate in approximately 75+-2.5mL of freshly boiled distilled water. Add 0.10+-0.01g of sodium carbonate to minimize bacterial decomposition of the thiofulfate solution. Quantitatively transfer the mixture to a 1-L volumetric flask and dilute to the mark. Allow the solution to stand at least 4 days before standardizing. The solution should be stored in an amber bottle.

8.3 Standard Iodine Solution (0.100+-0.001N)--- Weigh 12.700g of iodine and 19.100g 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 solution to stand a minimum of 4h 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-iodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

8.4 Pottassium Iodate Solution (0.1000N)--- Dry 4 or more grams of primary standard grade pottassium iodate (KIO_3) at 110+-5C for 2h and cool to room temperature in a desiccator. Dissolve 3.5667+_0.1mg of the dry pottassium iodate in about 100-mL of disstilled 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+-0.5g 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 whire 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 0.100N Sodium Thiosulfate --- Pipet 25.0 mL of potassium iodate (KIO_3) solution from 8.4 into a 250 mL titration (or wide-mouthed Erlenmeyer) flask. Add 2.00+-0.01g 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:

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

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 0.001N.

9.2 Standardization of 0.100+-0.001N Iodine Solution---- Pipet 25.0 mL of iodine solution (8.3) into a 250-mL widemouthed 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:

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

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 0.003N. The iodine solution concentration must be 0.100+-0.001N. 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 95wt% 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. Section 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 +/- 2s 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 0.100N iodine solution into each flask. Standardize into 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 +/- 1s. 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 0.100N 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 concentrations of the standard iodine solution and filtrates must be specified or known. This is necessary to determine an appropriate carbon weight to produce final concentrations agreeing with the definition of iodine number. The amount of carbon sample to be used in the determination is governed by the activity of the carbon. If filtrate normalities (C) are not within the range of 0.08N to 0.04N, repeat the procedure using different carbon weights.

11.2 Two calculations are required for each carbon dosage, as X/M and C.

11.2.1 To calculate the value of X/M, first derive the following values:

$$A = (N_2)(12693.0)$$

Where:

N_2 = iodine, N (from 9.2)

$$B = (N_1)(126.93)$$

Where:

N_1 = sodium thiosulfate, N (from 9.1)

$$DF = (I + H) / F$$

Where:

DF = dilution factor

I = iodine, mL (from 9.2)

H = 5% hydrochloric acid, mL, and

F = filtrate, mL

For example, if 10 mL of HCl and 50 mL of filtrate are used:

$$DF = (100 + 10) / 50 = 2.2$$

11.2.1.1 Calculate the value of X/M as follows:

$$X/M = [A-(DF)(B)(S)]/M$$

Where:

X/M = iodine absorbed per gram of carbon, mg/g

S = sodium thiosulfate, mL and

M = carbon used, g

11.2.2 Calculate the value of C as follows:

$$C = (N_1 * S) / F$$

Where:

C = residual filtrate, N

N₁ = sodium thiosulfate, N, and

F = filtrate, mL

11.3 Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of the three carbon dosages (see Fig.1). Calculate the least squares fit for the three points and plot. The iodine number is the X/M value at a residual iodine concentration (C) of 0.02N. The regression coefficient for the least squares fit should be greater than 0.995.

11.4 Carbon dosage may be estimated as follows:

$$M = [A-(DF)(C)(126.93)(50)]/E$$

Where:

M = carbon, g

A = (N₂)(12693.0),

DF = dilution factor (see 11.2.1),

C = residual iodine, and

E = estimated iodine number of the carbon

Three carbon dosages are calculated using three values of C (usually 0.01, 0.02 and 0.03).

12. Report

12.1 The reports should include the following:

12.1.1 Complete identification of the sample, including source, manufacturer's lot number, and carbon type.

12.1.2 The iodine number adsorption isotherm, the iodine value with a 95% confidence limit for the determination.

13. Precision and Bins

13.1 Interlaboratory precision for this test method was determined by a round robin in which six laboratories participated. Activated carbon made from five different raw materials was used in the round robin.

13.2 The following criteria should be used in determining the acceptability of the results:

13.2.1 Repeatability (Within Laboratories)---- Precision of this test method in the determination of iodine number of activated carbons ranging from 600 to 1450 iodine number is +5.6% of the average value measured in milligrams iodine absorbed per gram of carbon. This range corresponds to 2S, or the 95% confidence limits, as defined in Practice E177. If two results determined in the

same laboratory differ by more than 5.6%, they should be considered suspect.

13.2.2 Reproducibility (Between Laboratories)--- The between laboratory precision of this test method in the determination of iodine number of activated carbons ranging from 600 to 1450 iodine number, is $\pm 10.2\%$ of the average value, as measured in milligrams of iodine absorbed per gram of carbon. This range corresponds to $2S$ or the 95% confidence limits, as defined in Practice E177. Results obtained by two different laboratories which differ by more than 10.2% should be considered suspect.