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Standard Test Method for
Determination of Acid Soluble Iron Via Atomic Absorption[1]

This standard is issued under the fixed designation D6647; 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 acid soluble iron content of granular and powdered activated carbons, using an atomic absorption spectroscopy method by direct aspiration. Hydrochloric acid is used to extract the iron. This test method is not directly usable to determine the total iron concentration of a sample.

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.* For a specific hazard statement, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2652 Terminology Relating to Activated Carbon
D1193 Specification for Reagent Water
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 Apparatus[3]

3. Terminology

3.1 *Definitions*— Terms relating to this standard are defined in Terminology D2652.

3.1.1 *atomic absorption*—in flame atomic absorption spectrometry, a sample is aspirated into a flame and atomized. A light beam is directed through the flame, into a monochromator, and onto a detector that measures the amount of light absorbed by the atomized element in the flame. Because each metal has its own characteristic absorption wavelength, a source lamp of that element is used. The amount of energy at the characteristic wavelength absorbed in the flame is proportional to the concentration of the element in the sample over a limited concentration range.

4. Summary of Test Method

4.1 A representative sample of the material to be analyzed is collected according to E300. A known weight of the sample is ground until 95 % or more of the sample passes 325 mesh. The ground sample is oven dried, and then mixed with a dilute hydrochloric acid. The solution is boiled for 5 minutes to convert the iron into a soluble chloride, and then cooled and filtered. The filter cake is washed with water. Then the filtrate is transferred quantitatively to a beaker. The concentration of iron in the filtrate is measured by atomic absorption against a set of standards. The acid soluble iron concentration is then calculated against the weight of the original sample.

5. Significance and Use

5.1 In certain applications, such as acid purification, acidic food or chemical purification or decolorization, or other applications wherein iron may be leached out of the carbon, the use of acid-washed carbons will reduce or eliminate color pickup in the effluent or in the product. The acid soluble iron content is usually specified by the carbon supplier to prevent unacceptable levels of iron leach occurring.

5.2 Detection limits, sensitivity, and optimum ranges will vary with the models of atomic absorption spectrophotometers. General instrumental parameters are as follows:

5.2.1 Iron hollow cathode lamp.

5.2.2 Wavelength: 248.3 nm.

5.2.3 Fuel: acetylene (high purity).

5.2.4 Oxidant: air (from compressed air line, laboratory compressor, or a cylinder of compressed air—all need to be clean and dry).

5.2.5 Type of flame: oxidizing.

5.2.6 The following lines may also be used:

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

248.8 nm Relative Sensitivity 2
271.9 nm Relative Sensitivity 4
302.1 nm Relative Sensitivity 5
252.7 nm Relative Sensitivity 6
372.0 nm Relative Sensitivity 10

5.3 The method, as written, is intended for carbons having an acid soluble iron content of 0.0030 to 0.050 % For carbons having higher iron contents, larger dilutions or smaller aliquots may be used.

5.4 The user should validate that there are no interferences from other metals in the sample matrix.

5.5 To prevent erroneous results, the user should ensure that no iron instruments contact any of the sample or the solutions used in this procedure. Only glass, ceramics, or plastic should be allowed to contact the sample or solutions.

6. Apparatus

6.1 *Atomic absorption spectrometer ,* consisting of a light source emitting the line spectrum of an element (see 5.2.1 above), a device for vaporizing the sample (usually a flame), a means for isolating an absorption line (monochromator or filter and adjustable slit—see 5.2.2 and 5.2.6 above), and a photoelectric detector with its associated electronic amplifying and measuring equipment.

6.2 *Beakers*—Griffen, low form, 400 ml., Pyrex or equivalent.

6.3 *Cylinder*—ASTM, graduated, 100 ml.

6.4 *Flask*—Filtering, with side tube, 250 ml.

6.5 *Buchner Funnel*—Size D, 71 mm. ID.

6.6 *Filter Paper*—Whatman No. 3, 7.0 cm. or equivalent.

6.7 *Flasks*—Volumetric, 50, 100. 250, 500, and 1000 ml.

6.8 *Pipettes*—Volumetric, 1,2,5,10,25, and 100 ml.

6.9 *325 Mesh Screen* (U.S. Sieve series).

6.10 *Analytical Balance,* accuracy plus or minus 0.0001 g.

6.11 *Hot Plate.*

NOTE 1—All volumetric measuring equipment should meet or exceed the requirements of NIST Circular 602. Volumetric glassware meeting those specifications is normally designated as "Class A". See also specifications E287 and E288.

7. Reagents

7.1 *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.

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

7.3 *Standard Iron Solution I*—Completely dissolve 0.1000 grams of iron wire, C.P., in 20 ml. of hydrochloric acid, concentrated ACS hydrochloric acid grade, and 50 ml. of water. Allow to stand overnight. Quantitatively transfer the solution into a one liter volumetric flask and dilute to the mark with water. Mix thoroughly. This solution contains 0.1 mg. of iron per ml.

7.4 *Standard Iron Solution II*—Pipette 100.0 ml. of standard iron solution I into a one liter volumetric flask. Dilute to the mark with water. Mix thoroughly. This solution contains 0.01 mg. of iron/ml.

NOTE 2—Commercially available iron solutions obtained from chemical supply houses have been found to be acceptable, provided the accuracy of the solution meets specified requirements.

8. Hazards

8.1 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 associated with its use. The user is responsible for establishing appropriate health and safety practices and to determine the applicability of any federal and state regulations before attempting to use this test method.

8.1.1 Since this procedure involves the use of hydrochloric acid, all necessary personal protective equipment for handling acids should be used, including rubber gloves, lab apron, and goggles. Careful handling and good laboratory technique should always be used when working with chemicals. Avoid contact with hydrochloric acid or acid vapor. Care should be taken to prevent burns during handling of various solutions during this test procedure.

8.1.2 The atomic absorption spectrometer should be vented appropriately as recommended by the instrument manufacturer to dissipate fumes and vapors generated by the combustion the flame. This precaution protects laboratory personnel from toxic vapors, protects the instrument from corrosive vapors, and prevents flame stability from being affected by room drafts.

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

9. Sampling

9.1 Collect a representative sample of the carbon to be tested using Standard Practice E300. Approximately 10 grams of if the representative sample will be needed for this test.

10. Procedure

10.1 *Calibration Standards*

10.1.1 For those instruments which do not read out directly in concentration, a calibration curve is prepared to cover the appropriate concentration range. Usually, this means the preparation of standards which produce an absorbance of 0.0 to 0.7. Calibration standards are prepared by diluting the stock metal solutions at the time of analysis. For best results, calibration standards should be prepared fresh each time a batch of samples is analyzed.

10.1.2 Prepare a blank and several calibration standards as follows: For the blank, pipette 25.0 ml. of concentrated hydrochloric acid into a 250 ml. volumetric flask, and dilute to the mark with water. Follow sections 10.12-10.16 to determine the absorbance relative to 0 % acid soluble iron. For the standards, pipette 1.0, 3.0, 5.0, and 10.0 ml. of Standard Iron Solution II, from paragraph 7 above, into separate 50 ml. volumetric flasks. Repeat sections 10.12-10.16 to determine the absorbance relative to each level of acid soluble iron measured. Plot the absorbance versus milligrams of iron per ml. on linear graph paper. This will give you four points on the curve, representing 0.0002 mg/ml., 0.0006 mg/ml., 0.001 mg/ml., and 0.002 mg/ml. Retain the calibration curve for use in the final report.

10.2 This procedure is applicable to either powdered or granular activated carbon. When granular carbon is to be tested, grind approximately 10 grams of the sample until 95 % or more of the sample will pass through a 325 mesh screen (US Sieve Series, see Specification E11). Carbon received in the powder form may need additional grinding to meet the particle size requirement given above.

10.3 Oven dry the sample at 150°C for three hours and cool to room temperature in a desiccator.

10.4 Weigh 5.0 grams of the dried sample to the nearest 0.1 mg. and record the weight. This weight represents the sample weight (SW) used in Section 11.

10.5 Quantitatively transfer the sample to a clean 400 ml. beaker.

10.6 Carefully add 100.0 ml. of water and 25.0 ml. of concentrated hydrochloric acid to the beaker and swirl the contents until the carbon is wetted.

10.7 Place the beaker on the hotplate and allow to boil for 5 minutes. A watch glass placed on top of the beaker will reduce water loss during the 5 minute boil.

10.8 Remove the beaker from the hotplate and allow to cool to room temperature.

10.9 Filter the sample by vacuum through a Buchner funnel, using Whatman No. 3 filter paper, or equivalent. The filter should be prepared in advance by wetting the paper with water, and having the vacuum on before the sample is added to the filter.

10.10 Wash the carbon retained on the filter paper with several 5 ml. portions of water.

10.11 Quantitatively transfer the filtrate into a 250 ml. volumetric flask, and dilute to the mark with water. Mix thoroughly. If the carbon is suspected of having an acid soluble iron content above 0.03 %, the filtrate may be diluted to 500 ml. This volume represents the initial volume (IV) used in the calculation below, and should be recorded.

10.12 Pipette a 25.0 ml. aliquot of the solution into a 50.0 ml. volumetric flask. Dilute to the mark with water and mix thoroughly. This aliquot is the aliquot value (AT) used in the calculation below, and the volume here represents the final volume value (FV) used in the calculation below. These values should be recorded.

10.13 Differences between the various makes and models of satisfactory atomic absorption spectrophotometers prevent the formulation of detailed instructions applicable to every instrument. The analyst should follow the manufacturer's operating instructions for a particular instrument. In general, after choosing the proper lamp for the analysis per step 5.2 above, allow the lamp to warm up for a minimum of 15 minutes, unless operated in a double beam mode. During this period, align the instrument, position the monochromator at the correct wavelength per step 5.2 above, select the proper monochromator slit width, and adjust the current according to the manufacturer's recommendation.

10.14 Light the flame and regulate the flow of fuel and oxidant. Adjust the burner and nebulizer flow rate for maximum percent absorption and stability. Balance the photometer.

10.15 Aspirate the sample and determine the absorbance. Compare to the calibration curve to determine the concentration of iron in mg./ml. of the diluted sample.

10.16 Calculate the concentration of the acid soluble iron in % for the sample by the procedure in step 11 below.

11. Calculation

11.1

$$\%\ \text{Acid Soluble Iron} = \frac{C \times IV \times FV \times 100}{SW \times AT \times 1000} \quad (1)$$

where:

C = concentration of iron in mg/ml. (section 10.15),
IV = initial volume in ml. (section 10.11),
FV = final volume in ml. (section 10.12),
SW = sample weight in grams (section 10.4),
AT = aliquot taken in ml. (section 10.12),
1000 = conversion from g to mg, and
100 = conversion decimal to percent.

12. Report

12.1 In the report of this test, include the values for initial volume, final volume, aliquot taken, the conditions used for the spectrophotometer, and the calculated result for % acid soluble iron.

13. Precision and Bias

13.1 This procedure has not been subjected to round-robin testing as yet.

14. Keywords

14.1 acid soluble iron; activated carbon; atomic absorption; direct aspiration

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