



Designation: **D6647 – 01 (Reapproved 2011) D6647 – 18**

Standard Test Method for Determination of Acid Soluble Iron Viavia Atomic Absorption¹

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~~ safety, health, and ~~health~~ environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 8.

1.3 *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

[D1193 Specification for Reagent Water](#)

[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](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

2.2 *NIST Publication:*³

[Circular 602 Testing of Glass Volumetric Apparatus](#)

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 [Practice 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

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](#).

hydrochloric acid. The solution is boiled for 5 ~~minutes~~min 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:

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%~~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~~, 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~~mL, Pyrex or equivalent.

6.3 ~~Cylinder~~—ASTM, graduated, 100 ~~ml~~mL.

6.4 ~~Flask~~—Filtering, with side tube, 250 ~~ml~~mL.

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

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

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

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

6.9 ~~325-Mesh~~325-Mesh Screen (U.S. Sieve series).

6.10 ~~Analytical Balance~~, accuracy ~~plus or minus 0.0001~~±0.0001 g.

6.11 ~~Hot Plate~~Hotplate.

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”A.” See also ~~specifications~~Specifications [E287](#) and [E288](#).

7. Reagents

7.1 ~~Purity of Reagents~~—~~Reagent grade~~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.