Designation: D 6647 – 01

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

This standard is issued under the fixed designation D 6647; 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:*

D 2652 Terminology Relating to Activated Carbon[2]

D 1193 Specification for Reagent Water[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[2]

E 287 Specification for Laboratory Glass Graduated Burets[5]

E 288 Specification for Laboratory Glass Volumetric Flasks[5]

E 300 Practice for Sampling Industrial Chemicals[5]

2.2 *NIST Publication:*

Circular 602 Testing of Glass Volumetric Apparatus[6]

3. Terminology

3.1 *Definitions*—Terms relating to this standard are defined in Terminology D 2652.

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 E 300. 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:

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

[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] *Annual Book of ASTM Standards*, Vol 15.01.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 15.05.

[6] Available from National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD, 20899.