Designation: C816 – 15

Standard Test Method for Sulfur Content in Graphite by Combustion-Iodometric Titration Method[1]

This standard is issued under the fixed designation C816; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope*

1.1 This test method covers the determination of sulfur in graphite in the concentration range from 1 µg⁄g to 1000 µg⁄g (ppm).

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke (Withdrawn 2012)[3]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

3. Terminology

3.1 *Definitions:*

3.1.1 *combustion, n*—chemical reaction by which graphite is combined in a controlled manner with pure oxygen in a high temperature furnace for analytical purposes.

3.1.2 *sulfur content, n*—percentage content by weight of elemental sulfur present in graphite.

3.1.3 *titration, n*—quantitative chemical analysis method used to determine the unknown concentration of a specified element by reacting a solution prepared from the sample to be analyzed with a known concentration and volume of specific reagent.

4. Summary of Test Method

4.1 The sample is combusted with pure oxygen in a high-temperature furnace and a major portion of the sulfur is converted to sulfur dioxide. The sulfur dioxide is passed through a potassium iodide-starch solution where it is titrated with potassium iodate solution. The potassium iodate solution is standardized against samples of known sulfur content.

5. Significance and Use

5.1 Sulfur, even in very low concentrations, is of concern in a nuclear reactor because of potential corrosion of metallic components. This test method has the sensitivity to analyze very low sulfur contents in graphite using very small samples.

5.2 This test method can be used to characterize graphite for design purposes.

6. Interferences

6.1 Any substance that releases volatile material, which tends to enhance or to bleach the starch-iodine complex, will interfere. Halogens and oxides of nitrogen interfere through darkening the color of the starch-iodine complex. Ultraviolet light will also darken the solution. A tube packed with either silver wool or antimony filings placed in the line between the furnace and titration assembly will remove halogens from the gas stream.

6.2 If the solution in the titration vessel becomes colorless during the titration, some SO_2 will be lost and a low result will be obtained for the sulfur content.

7. Apparatus

7.1 Apparatus for the determination of sulfur by direct combustion shall be in accordance with Practices E50.

8. Reagents and Materials

8.1 *Potassium Iodate Solution (0.2 mM)*—Dissolve 44.4 mg of potassium iodate (KIO_3) in water and dilute to 1 L.

[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.F0 on Manufactured Carbon and Graphite Products.

Current edition approved Dec. 1, 2015. Published January 2016. Originally approved in 1977. Last previous edition approved in 2010 as C816 – 85 (2010)ε1. DOI: 10.1520/C0816-15.

[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.

***A Summary of Changes section appears at the end of this standard**