Designation: C 816 – 85 (Reapproved 1998)ε1

An American National Standard

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

This standard is issued under the fixed designation C 816; 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 Department of Defense.

ε[1] NOTE—Section 11 was added editorially in April 1998.

1. Scope

1.1 This test method covers the determination of sulfur in graphite in the concentration range from 1 to 200 µg/g in a 1-g sample or 5 to 1000 µg/g in a 0.2-g 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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 3177 Test Method for Total Sulfur in the Analysis Sample of Coal and Coke[2]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]

3. Summary of Test Method

3.1 The sample is burned in oxygen 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.

4. Significance and Use

4.1 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.

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

5. Interferences

5.1 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.

5.2 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.

6. Apparatus

6.1 Apparatus for the determination of sulfur by direct combustion shall be in accordance with No. 13 in Fig. 13 of Practices E 50[4].

7. Reagents and Materials

7.1 *Potassium Iodate Solution (0.0444 g/L)*—Dissolve 0.0444 g of potassium iodate (KIO_3) in water and dilute to 1 L.

NOTE 1—The sulfur equivalent for the KIO_3 solution is based on the following reactions:

$$KIO_3 + 5KI + 6HCl = 3I_2 + 6KCl + 3H_2O$$
$$SO_2 + I_2 + 2H_2O = H_2SO_4 + 2HI$$

On the basis of 100 % conversion of sulfur to SO_2, 1 mL of this solution is equivalent to 20 µg of sulfur.

7.2 *Hydrochloric Acid*—Dilute 15 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 1 L with water.

7.3 *Starch-Potassium Iodide Solution*— Add 2 g of arrowroot starch to 50 mL of water. Separately boil 150 mL of water and slowly add the starch solution, stirring constantly. Cool, add 6 g of potassium iodide (KI), and pour the resulting solution into a flask. Store in a refrigerator.

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

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[2] *Annual Book of ASTM Standards,* Vol 05.06.

[3] *Annual Book of ASTM Standards,* Vol 03.05.

[4] Equipment found suitable for this method is available from Laboratory Equipment Co., St. Joseph, MI.