Designation: C816 – 15

An American National Standard

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D3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke (Withdrawn 2012)[3]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

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[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.

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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] 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**

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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.

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

8.3 *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.

8.4 *Standard Sulfur*—Prepare sulfur standards from coke samples that have been analyzed by the Eschka Method in accordance with Test Method D3177.

9. Procedure

9.1 Adjust the furnace temperature to 1400 °C to 1425 °C.

9.2 Turn on the oxygen and set the flow to 1 L/min.

9.3 Fill the buret with the iodate solution.

9.4 Rinse the titration vessel with HCl solution and drain.

9.5 Fill the SO_2 titration vessel to the operating level with HCl solution.

9.6 Add 2 mL of the starch-KI solution to the titration vessel.

9.7 Adjust the color of the solution in the titration vessel to a medium blue by additions of small amounts of KIO_3 solution. This color will be the color of the end point.

NOTE 2—If using a commercial sulfur titrator, follow the manufacturer's instructions for its operation.

9.8 Remove the stopper from the mouth of the combustion tube. Insert a boat containing a 0.2 g to 1.0 g sample into the combustion tube and push into the hot zone.

NOTE 3—Furnace tubes used for high sulfur samples (above 1000 ppm S) should not be used for low sulfur samples. Separate furnace tubes should be used for low and high sulfur samples.

9.9 Replace the stopper sending the flow of oxygen through the combustion tube.

9.10 Titrate the solution with the KIO_3 solution to maintain the blue color developed in 9.7.

9.11 When the combustion of the sample is complete, record the volume of KIO_3 solution used for the titration.

9.12 Make a blank run on an empty prefired boat, igniting for the same length of time as the sample.

9.13 Run standard sulfur samples to obtain a calibration factor. Place a weighed amount of the standard in a prefired boat, ignite, and titrate following steps 9.9 to 9.12.

10. Calculation

10.1 Calculate the sulfur factor as follows:

$$F = S/(R - B)$$

where:

F = sulfur factor,
S = amount of sulfur in the standard, µg,
R = amount of titrant for the standard, mL, (Note 4), and,
B = amount of titrant for the blank, mL, (Note 4).

NOTE 4—Or substitute the apparent percentage of sulfur for "direct reading" burets.

10.2 Calculate the amount of sulfur in the test sample as follows:

$$Sulfur, ppm = [(T - B) \times F]/G$$

where:

T = amount of titrant for the sample, L, (Note 4),
B = amount of titrant for the blank, mL, (Note 4),
F = sulfur factor, and
G = amount of sample, g.

11. Precision and Bias

11.1 The reproducibility among four laboratories was 14 % at the 9 ppm level and 4.2 % at the 86 ppm level.

12. Keywords

12.1 combustion; graphite; sulfur; titration

SUMMARY OF CHANGES

Subcommittee D02.F0 has identified the location of selected changes to this standard since the last issue (C816 – 85 (2010)$^{\varepsilon 1}$) that may impact the use of this standard. (Approved Dec. 1, 2015.)

(1) Revised the title.
(2) Added new Terminology Section 3.
(3) Revised subsection 4.1.

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