



Standard Test Method for Sulfur in Graphite by Combustion-Iodometric Titration Method¹

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

1. Scope

1.1 This test method covers the determination of sulfur in graphite in the concentration range from 1 to 200 $\mu\text{g/g}$ in a 1-g sample or 5 to 1000 $\mu\text{g/g}$ in a 0.2-g sample.

1.2 The values stated in SI units are to be regarded as the 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

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

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

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.

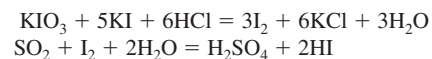
6. Apparatus

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**.³

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:



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 arrow-root starch to 50 mL of water. Separately boil 150 mL of water

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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 sole source of supply of the apparatus known to the committee at this time is available from Laboratory Equipment Co., St. Joseph, MI. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee ¹, which you may attend.

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.

7.4 *Standard Sulfur*—Prepare sulfur standards from coke samples that have been analyzed by the Eschka Method in accordance with Test Method **D 3177**.

8. Procedure

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

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

8.3 Fill the buret with the iodate solution.

8.4 Rinse the titration vessel with HCl solution and drain.

8.5 Fill the SO₂ titration vessel to the operating level with HCl solution.

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

8.7 Adjust the color of the solution in the titration vessel to a medium blue by additions of small amounts of KIO₃ 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.

8.8 Remove the stopper from the mouth of the combustion tube. Insert a boat containing a 0.2 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.

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

8.10 Titrate the solution with the KIO₃ solution to maintain the blue color developed in **8.7**.

8.11 When the combustion of the sample is complete, record the volume of KIO₃ solution used for the titration.

8.12 Make a blank run on an empty pre-fired boat, igniting for the same length of time as the sample.

8.13 Run standard sulfur samples to obtain a calibration factor. Place a weighed amount of the standard in a pre-fired boat, ignite, and titrate following steps **8.9** to **8.12**.

9. Calculation

9.1 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.

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

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

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.

10. Precision and Bias

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

11. Keywords

11.1 combustion; graphite; sulfur; titration

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