



Designation: ~~D7679-10~~ Designation: D7679 - 11

Standard Test Method for Sulfur Content in Carbon Black Feedstock Oils¹

This standard is issued under the fixed designation D7679; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

~~1.1 This test method covers the instrumental determination of sulfur content in samples of carbon black feedstock oils. Values obtained represent the total sulfur content.~~

1.1 This test method covers the instrumental determination of sulfur content in samples of carbon black feedstock oils. Values obtained represent the total sulfur content. Two analysis methods are available for use:

Test Method A: High-Temperature Combustion With Infrared Absorption Detection Procedures
Test Method B: X-Ray Fluorescence

Sections 4-10

Sections 11-15

1.2 This test method is applicable to carbon black feedstock oils derived from petroleum, coal, and other sources which include fuel oils, residues, tars, pitches, reclaimed oils, and similar materials that are normally handled as liquids. This test method is applicable to products typically containing 0 to 5 mass % sulfur.

1.3 The results of these tests can be expressed as mass % sulfur.

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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

D1619 [Test Methods for Carbon Black Sulfur Content](#)

D4057 [Practice for Manual Sampling of Petroleum and Petroleum Products](#)

D4177 [Practice for Automatic Sampling of Petroleum and Petroleum Products](#)

3. Summary of Test Method

~~3.1 In this test method, a sample of feedstock oil is weighed in a combustion boat containing either a tungsten oxide based accelerator, sand or aluminum oxide absorbent, and the sulfur content is determined by placing the boat in a tube furnace at 1350°C in an air or oxygen-enriched atmosphere resulting in complete combustion. Sulfur in the sample is completely oxidized to sulfur dioxide. Moisture and particulates are removed from the gas stream by traps filled with anhydrous magnesium perchlorate. The gas stream is then passed through a cell in which sulfur dioxide concentration is measured by an infrared (IR) absorption detector at a precise wavelength in the IR spectrum. The IR absorption detects sulfur dioxide throughout the entire combustion process. The integral of the detector signal is the basis for the total sulfur content of a sample.~~

~~3.2 This test method is for use with commercially available sulfur analyzers equipped to carry out the combustion and measurement operations automatically.~~

~~3.3 The sulfur analyzer shall be calibrated using appropriate calibration standards (see 6.7).~~

~~3.4 The instrument may be configured to analyze more than one element such as a carbon-sulfur analyzer. In this case, the analyzer has individual detectors for each element that are arranged in series.~~

¹ This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.66 on Environment, Health, and Safety.

Current edition approved Dec. 1, 2010. Published January 2011. DOI: 10.1520/D7679-10.

Current edition approved Nov. 1, 2011. Published December 2011. Originally approved in 2010. Last previous edition approved in 2010 as D7679-10. DOI: 10.1520/D7679-11.

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.

4. Significance and Use

4.1 Measuring the total sulfur content of feedstock oil is often a necessary component in calculations for sulfur dioxide emissions.

4.2 The carbon black industry measures sulfur content of feedstock oils along with sulfur content of carbon black products per Test Method D1619 in calculations to determine sulfur dioxide emissions for compliance with governmental reporting requirements.

TEST METHOD A: HIGH-TEMPERATURE COMBUSTION WITH INFRARED ABSORPTION DETECTION PROCEDURES

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5. Apparatus

5.1 There are a number of commercially available instruments designed to measure sulfur content in organic materials including sulfur and carbon-sulfur combustion analyzers. This test method makes no specifications regarding system designs.

5.2 Functionally, however, the following are specified for all instruments:

5.2.1 An analytical balance, or equivalent, that is capable of a weighing sensitivity of 0.1 mg or better resolution.

5.2.2 The combustion tube and boat are made of a suitable material such as mullite, porcelain, or zircon.

5.2.3 The sample is absorbed onto a solid which may include the following materials: (1) tungsten oxide based accelerator; (2) Sea sand; (3) Aluminum oxide. Refer to the instrument manufacturer's recommendations.

5.2.4 The sample is combusted at a temperature of $1350 \pm 15^\circ\text{C}$ in an air or oxygen-enriched atmosphere.

5.2.5 The combustion gas is passed through an absorbent (anhydrous magnesium perchlorate) to remove water vapor.

5.2.6 The detection system measures sulfur dioxide using an IR absorption detector at a wavelength suitable for linear responses with respect to the concentration over the full range of possible concentrations.

6. Reagents

6.1 *Purity of Reagents*—Reagent-grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Magnesium Perchlorate*.

6.3 *Oxygen*, high purity, 99.9 %.

6.4 *Tungsten (VI) Oxide Accelerator*, containing potassium dihydrogen-phosphate (optional).

6.5 *Sea Sand*, purified by acid and calcinated (optional).

6.6 *Aluminum Oxide*, optional.

6.7 *Calibration Standards*.⁴

7. Sampling

7.1 Collect feedstock oil as specified in Practices D4057 or D4177.

7.2 *Sample*—This is the portion or aliquot of the feedstock oil for use in obtaining a result.

7.3 *Preparation*—Warm viscous samples until they are fluid and shake for 5 s.

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁴ Sulfur calibration and reference standards are typically available from the instrument manufacturer, National Institute of Standards and Technology's (NIST), <http://www.nist.gov>, and are also available from Alpha Resources, 3090 Johnson Road, Stevensville, MI 49127, www.alpharesources.com.