



Designation: D 5016 – 98

Standard Test Method for Sulfur in Ash from Coal, Coke, and Residues from Coal Combustion Using High-Temperature Tube Furnace Combustion Method with Infrared Absorption¹

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1. Scope

1.1 This test method describes a procedure using a high-temperature tube furnace and infrared detection for the rapid determination of sulfur in ash from coal and coke.

1.2 This test method is an alternative for test methods described in Test Methods D 1757.

1.3 The values stated in SI units (Practice E 380) shall be regarded as the standard.

1.4 *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 1757 Test Methods for Sulfur in Ash from Coal and Coke²

D 2795 Test Methods for Analysis of Coal and Coke Ash²

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal²

D 3682 Test Method for Major and Minor Elements in Coal and Coke Ash by Atomic Absorption²

D 4326 Test Method for Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence²

D 4621 Guide for Accountability and Quality Control in the Coal Analysis Laboratory²

D 5142 Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures²

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)³

3. Summary of Test Method

3.1 A weighed test portion is mixed with a promoting agent and ignited in a tube furnace at a minimum operating tempera-

ture of 1350°C in a stream of oxygen (see Note 1). The combustible sulfur contained in the test portion is oxidized to gaseous oxides of sulfur. Moisture and particulates are removed by traps filled with anhydrous magnesium perchlorate. The gas stream is passed through a cell in which sulfur dioxide is measured by an infrared absorption detector. Sulfur dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy is absorbed as the gas passes through the cell body in which the IR energy is being transmitted; thus, at the detector, less energy is received. All other IR energy is eliminated from reaching the detector by a precise wavelength filter. The absorption of IR energy can be attributed only to sulfur dioxide whose concentration is proportional to the change in energy at the detector. One cell is used as both a reference and a measurement chamber. Total sulfur as sulfur dioxide is detected on a continuous basis.

NOTE 1—The promoter aids in the quantitative release of all sulfur present in the test portion as SO₂. Refer to the instrument manufacturer's recommended procedure for using oxidizing agents or promoters commonly referred to as "combustion accelerators."

3.2 This test method is applicable for use with commercially available sulfur analyzers equipped to carry out the operations in 3.1 automatically, and must be calibrated using certified reference materials (CRM's) covering the range of sulfur in the ash samples being analyzed.

4. Significance and Use

4.1 The purpose of this test method is to determine the percent sulfur trioxide (SO₃) portion of the major and minor elements in coal ash.

4.2 This test method may be used to determine the percent sulfur trioxide (SO₃) portion of ash determined by Test Methods D 3174 or D 5142 for coals containing high amounts of calcium. The ash may then be reported on a sulfur trioxide free basis.

5. Apparatus

5.1 *Tube Furnace*, electrically heated, capable of heating 150 to 165-mm length of the hot zone area of the combustion tube (see 5.2) to at least 1350°C. Specific dimensions can vary with manufacturer's design.

5.2 *Combustion Tube*, made of mullite, porcelain, or zircon,

¹ This test method is under the jurisdiction of ASTM Committee D-5 on Coal and Coke and is the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

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² *Annual Book of ASTM Standards*. Vol 05.05.

³ *Annual Book of ASTM Standards*. Vol 14.02.