



Standard Test Method for Total Sulfur in Coal and Coke Combustion Residues Using a 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 determination of sulfur in coal and coke combustion residues, including lab ashes and residues from coal and coke combustion.

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

D 121 Terminology of Coal and Coke

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

D 3682 Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes

D 3683 Test Method for Trace Elements in Coal and Coke Ash by Atomic Absorption

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

D 4621 Guide for Quality Management in an Organization That Samples or Tests Coal and Coke

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

D 6349 Test Method for Determination of Major and Minor Elements in Coal, Coke, and Solid Residues from Combustion of Coal and Coke by Inductively Coupled Plasma—Atomic Emission Spectrometry

D 6357 Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma Mass Spectrometry, and Graphite Furnace Atomic Absorption

3. Terminology

3.1 For definitions of terms used in these test methods, refer to Terminology D 121.

3.2 Throughout this test method the term ash is used to describe the sample being analyzed. The term ash is to be interpreted as a combustion residue.

4. Summary of Test Method

4.1 A weighed test portion is mixed with a promoting agent and ignited in a tube furnace at an operating temperature of 1450°C in a stream of oxygen. 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

¹ This test method is under the jurisdiction of ASTM Committee D05 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 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.

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.

4.2 This test method is applicable for use with sulfur analyzers equipped to carry out the operations in 4.1 and must be calibrated using reference materials (RMs) covering the range of sulfur in the ash samples being analyzed.

5. Significance and Use

5.1 The percent sulfur content of the ash derived from coal or coke can be calculated to sulfur trioxide content. This information can be used in combination with results from the determination of major, minor and/or trace elements in the same ash to calculate results on a sulfur trioxide free-basis or to calculate total recovered analyte.

6. Interferences

6.1 Known interferences in this test method are some alkaline earth metal ions, including barium and strontium, which form stable sulfate salts that are difficult to decompose. In order to have an accurate analysis of the material all mineral sulfates must be decomposed to yield sulfur dioxide, which is then presented to the IR detection system for measurement. To minimize interferences a promoting agent shall be used to help decompose these salts. These promoting agents can have one or more of the following properties, (1) oxidizing agent, (2) reducing agent and (3) fluxing agent. Oxidizing and reducing agents help decompose the metal sulfates through the oxidation or reduction of the sulfate or metal ions, or both. The fluxing agent helps decompose the metal sulfates by fusing the salt, which leads to the decomposition of sulfates on further heating. Some promoting agents may contain sulfur, which needs to be addressed with their use.

7. Apparatus

7.1 *Tube Furnace*, electrically heated, capable of heating 150 to 165-mm length of the hot zone area of the combustion tube (see 7.2) to at least 1350°C. Specific dimensions can vary with design.

7.2 *Combustion Tube*, made of mullite, porcelain, or zircon, approximately 23-mm inside diameter with a 3-mm thick wall, at least 450 mm long with means to route the gases produced by combustion through the infrared cell.

7.3 *Sample Combustion Boats*, made of iron-free material and of a convenient size suitable for the instrument being used.

7.4 *Boat Puller*, rod of a heat resistant material with a bent or disk end used to insert and remove boats from the combustion tube.

8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used unless otherwise specified. All reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications

exist.^{3,4} Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without affecting the accuracy of the determination.

8.2 *Magnesium Perchlorate (Mg(ClO₄)₂)*—**Warning:** Magnesium perchlorate is a strong oxidizing agent. Do not attempt to regenerate the absorbent. Do not permit contact with organic materials or reducing agents.

8.3 *Promotor*—COM-CAT Combustion promoter⁵.

NOTE 1—COM-CAT⁵ is a promoting agent that is both a fluxing agent and oxidizing agent.

8.4 *Oxygen, 99.5 % Pure*—Compressed gas contained in a cylinder equipped with a suitable pressure regulator and needle valve to control gas flow. **Warning:** Pure oxygen vigorously accelerates combustion. All regulators, lines, and valves shall be free of grease and oil.

9. Hazards

9.1 The user shall ensure acceptable documented safety procedures are in place for the handling of all reagents and test materials and for the operation of laboratory equipment specified for this test method.

10. Calibration Materials and Test Samples

10.1 *Calibration Materials*—Employ RMs for calibration and control. Reference material coal, coke and ash samples can be used for calibration. A RM shall be a Certified Reference Material (CRM) from recognized certifying agencies such as the National Institute for Science and Technology (NIST) or the South African Bureau of Standards (SABS), an External Reference Material (ERM) available from suppliers that provide details of traceability to a recognized CRM with a similar matrix and composition, or an Internal Reference Material (IRM) that has been validated to use through intercomparison with one or more CRMs or ERMs traceable to a CRM.

10.2 For total sulfur in ash determination to complement major and minor elements in ash analysis, the ash sample is prepared in accordance with Test Methods [D 3682](#), [D 4326](#), or [D 6349](#).

10.3 For total sulfur in ash determination to calculate total recovered analyte in conjunction with the determination of trace elements, the ash sample is prepared in accordance with Test Methods [D 3683](#) or [D 6357](#).

10.4 For sulfur correction of ash as determined by Test Methods [D 3174](#) or [D 5142](#), the sample shall consist of the

³ Interested parties are invited to submit information regarding the identification of alternatives to ASTM international Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

⁴ *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*. U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

⁵ The sole source of supply of COM—CAT known to the committee at this time is LECO Corporation, 3000 Lakeview Ave. St Joseph, MI 49085 USA. 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.