Designation: D3682 – 21

Standard Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes by Atomic Spectrometry[1]

This standard is issued under the fixed designation D3682; 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.

1. Scope

1.1 This test method covers the analysis of the commonly determined major and minor elements in combustion residues from coal utilization processes.

1.2 Use Test Method D5016 for determination of sulfur.

1.3 *Units*—The values stated in SI units are to be regarded as standard. The values given in parentheses are provided for information only and are not considered 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D121 Terminology of Coal and Coke
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D1193 Specification for Reagent Water
D2013 Practice for Preparing Coal Samples for Analysis
D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D5016 Test Method for Total Sulfur in Coal and Coke Combustion Residues Using a High-Temperature Tube Furnace Combustion Method with Infrared Absorption
D7348 Test Methods for Loss on Ignition (LOI) of Solid Combustion Residues
D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D121.

4. Summary of Test Method

4.1 The combustion residue to be analyzed is ignited in air at 750 °C to a constant mass. The ash is fused within lithium tetraborate ($Li_2B_4O_7$) followed by a final dissolution of the melt in either dilute hydrochloric acid (HCl) or dilute nitric acid (HNO_3). The solution is analyzed by atomic absorption for applicable elements.

5. Significance and Use

5.1 A compositional analysis of the ash in coal is often useful in the total description of the quality of the coal. Knowledge of ash composition is also useful in predicting the behavior of ashes and slags in combustion chambers. Utilization of the ash by-products of coal combustion sometimes depends on the chemical composition of the ash.

5.2 Note that the chemical composition of laboratory-prepared coal ash may not exactly represent the composition of mineral matter in the coal or the composition of fly ash and slag resulting from commercial-scale burning of the coal.

6. Apparatus

6.1 *Ashing Furnace,* with an adequate air circulation and capable of having its temperature regulated at 500 °C and 750 °C.

6.2 *Fusion Furnace,* with an operating temperature of 1000 °C.

[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.

Current edition approved Dec. 1, 2021. Published January 2022. Originally approved in 1978. Last previous edition approved in 2013 as D3682 – 13. DOI: 10.1520/D3682-21.

[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.

6.3 *Platinum Vessels (Dish or Crucible),* 35 mL to 85 mL capacity. Graphite crucibles with 10 mL to 15 mL capacity may also be used (Note 1).

NOTE 1—The use of graphite crucibles and subsequent dissolution of fused beads from them was not investigated; however, their successful use in similar methods has been reported.[3]

6.4 *Stirring Hotplate and Bars,* operating temperature of 200 °C.

6.5 *Flame Atomic Absorption Spectrophotometer*—Any dual-channel instrument using a deuterium (D_2) arc background corrector or other comparable simultaneous background correction system equipped with air/acetylene and nitrous oxide/acetylene burner heads.

6.6 *Hollow Cathode or Electrodeless Discharge Lamp,* for each element to be defined.

6.7 *Deuterium Continuum Lamp.*

6.8 *Compressed Air*—Appropriate pressure reducing regulator with base connections (see instrument manufacturer's instructions).

6.9 *Acetylene Gas and Regulator*—A cylinder of acetylene equipped with a two-gauge, two-stage pressure-reducing regulator (see instrument manufacturer's instructions).

6.10 *Nitrous Oxide Gas and Regulator*—A cylinder of nitrous oxide equipped with a two-gauge, two-stage pressure-reducing regulator (see instrument manufacturer's instructions).

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.[4] 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. The lithium tetraborate and lanthanum chloride reagents in particular should be examined for alkali and alkaline earth contamination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water as defined in Specification D1193.

7.3 *Aluminum Stock Solution* (1000 µg/mL aluminum).

7.4 *Calcium Stock Solution* (1000 µg/mL calcium).

7.5 *Iron Stock Solution* (1000 µg/mL iron).

7.6 *Lanthanum Chloride Solution* (175 g/L lanthanum chloride ($LaCl_3$) or equivalent 10 % mass concentration lanthanum).

7.7 *Fluxing Agent* - Lithium Tetraborate—($Li_2B_4O_7$), or mixtures of lithium tetraborate ($Li_2B_4O_7$) and anhydrous lithium metaborate ($LiBO_2$).

7.8 *Magnesium Stock Solution* (1000 µg/mL magnesium).

7.9 *Potassium Stock Solution* (1000 µg/mL potassium).

7.10 *Silicon Stock Solution* (200 µg/mL silicon) (Note 2).

NOTE 2—A standard stock solution can be prepared by fusing 0.1070 g of reignited spectrographic grade silica (SiO_2) with 1 g of lithium tetraborate, dissolving in solvent acid, and diluting to 250 mL as described for sample preparation in 9.3.1 and 9.3.2. This solution is 200 µg/mL silicon. Preferable standard preparations for silica are made by fusion and dilution of ash sample(s) of known composition in accordance with 9.3.1 and 9.3.2. The standard sample(s) should have a composition(s) similar to the unknown.

7.11 *Sodium Stock Solution* (1000 µg/mL sodium).

7.12 *Titanium Stock Solution* (1000 µg/mL titanium).

7.13 *Solvent Acid*

7.13.1 *Hydrochloric Acid (HCl)*—Concentrated hydrochloric acid, 12 N, specific gravity (sp) 1.19.

7.13.2 *Nitric Acid (HNO_3)*—Concentrated nitric acid, 16 N, sp 1.42.

7.13.3 Dilute 50 mL of concentrated hydrochloric acid (sp gr 1.19) or 50 mL of concentrated nitric acid (sp gr 1.42) to 1000 mL. Either acid solution may be used, but whichever is chosen should be used throughout the subsequent solution preparations.

8. Sample Preparation

8.1 *Coal and Coke*—Prepare the analysis sample in accordance with Practice D2013 for coal or Practice D346 for coke by pulverizing the material to pass a 250 µm (No. 60) U.S.A. standard sieve.

8.1.1 Analyze separate test portions for moisture and ash contents in accordance with Test Methods D3173, D3174, or D7582, so that calculations to other bases can be made.

8.2 *Laboratory Ashing of Coal and Coke Analysis Sample*—Prepare the ash from a thoroughly mixed analysis sample of coal or coke (8.1). Spread the coal and coke in a layer not over 6 mm in depth in a porcelain, quartz, or fused silica roasting dish. Place the dish in a cold muffle furnace and heat gradually so that the temperature reaches 500 °C ± 10 °C at the end of 1 h. Continue the gradual heating until the temperature rises from 500 °C ± 10 °C to 750 °C ± 15 °C at the end of 1 h. Maintain the 750 °C temperature until the test specimen reaches a constant mass or for an additional 2 h. Allow the dish to cool, transfer to an agate mortar, and grind to pass a 75 µm (No. 200) U.S.A. standard sieve. Reignite the ash at 750 °C for 1 h, cool rapidly, and determine the mass of portions for analysis.

8.3 *Solid Combustion Residue*—Dry a representative portion of the solid residue to constant mass at 107 °C ± 3 °C. Determine the moisture loss during this drying step if it is desirable to calculate results to an as-received basis. Crush the

[3] Muter, R. B., and Nice, L. L., "Major and Minor Constituents in Siliceous Materials by Atomic Absorption Spectroscopy," Advances in Chemistry Series 141, *Trace Elements in Fuels,* American Chemical Society, Washington, DC, 1975, pp. 57–65.

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.