



Designation: D 2795 – 95

## Standard Test Methods for Analysis of Coal and Coke Ash<sup>1</sup>

This standard is issued under the fixed designation D 2795; 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 These test methods cover the analysis of coal and coke ash for the commonly determined major elements.

1.2 The test methods appear in the following order:

	Sections
Silicon Dioxide (SiO <sub>2</sub> )	10 to 12
Aluminum Oxide (Al <sub>2</sub> O <sub>3</sub> )	13 to 15
Ferric Oxide (Fe <sub>2</sub> O <sub>3</sub> )	16 to 18
Titanium Dioxide (TiO <sub>2</sub> )	19 to 22
Phosphorus Pentoxide (P <sub>2</sub> O <sub>5</sub> )	23 to 25
Calcium Oxide (CaO), and Magnesium Oxide (MgO)	26 to 29
Sodium Oxide (Na <sub>2</sub> O) and Potassium Oxide (K <sub>2</sub> O)	30 to 33

NOTE 1—Test Methods D 1757D 1757 is used for determination of sulfur.

1.3 The values stated in SI units (Practice E 380E 380) shall be regarded as the standard. The values given in parentheses are for information only.

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 346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis<sup>2</sup>

D 1193 Specification for Reagent Water<sup>3</sup>

D 1757 Test Methods for Sulfur in Ash from Coal and Coke<sup>2</sup>

D 2013 Test Method for Preparing Coal Samples for Analysis<sup>2</sup>

D 3173 Test Method for Moisture in the Analysis Sample of Coal and Coke<sup>2</sup>

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal<sup>2</sup>

D 3180 Practice for Calculating Coal and Coke Analyses

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-5 on Coal and Coke and are the direct responsibility of Subcommittee D05.29 on Major Elements in Ash and Trace Elements of Coal.

Current edition approved Sept. 10, 1995. Published April 1996. Originally published as D 2795 – 69. Last previous edition D 2795 – 86 (1991)<sup>ε1</sup>.

<sup>2</sup> Annual Book of ASTM Standards, Vol 05.05.

<sup>3</sup> Annual Book of ASTM Standards, Vol 11.01.

from As-Determined to Different Bases<sup>2</sup>

D 5142 Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures<sup>2</sup>

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)<sup>4</sup>

### 3. Summary of Test Methods

3.1 The coal or coke to be analyzed is ashed under standard conditions and ignited to constant weight. Two solutions are prepared from the ash. Solution A is obtained by fusing the ash with sodium hydroxide (NaOH) followed by a final dissolution of the melt in dilute hydrochloric acid (HCl). Solution B is prepared by decomposition of the ash with sulfuric (H<sub>2</sub>SO<sub>4</sub>), hydrofluoric (HF), and nitric (HNO<sub>3</sub>) acids. Solution A is used for the analysis of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>, and Solution B for the remaining elements.

3.2 The two solutions are analyzed by a combination of methods: (1) spectrophotometric procedures are used for SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>, and P<sub>2</sub>O<sub>5</sub>; (2) chelatometric titration for CaO and MgO; and (3) flame photometry for Na<sub>2</sub>O and K<sub>2</sub>O. See Fig. 1 for a general outline of the methods and procedures used for each determination.

### 4. Significance and Use

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

4.2 It should be noted that chemical composition of laboratory prepared coal ash may not exactly represent the composition of mineral matter in the coal, or the composition of the fly ash and slags resulting from the commercial scale burning of the coal.

### 5. Apparatus

5.1 *Balance*, sensitive to 0.1 mg.

5.2 *Crucibles*—Nickel crucibles of 50-cm<sup>3</sup> capacity shall be used for NaOH fusion of the ash, and platinum crucibles of 30-cm<sup>3</sup> capacity shall be used for decomposition of the ash with HF.

<sup>4</sup> Annual Book of ASTM Standards, Vol 14.02.

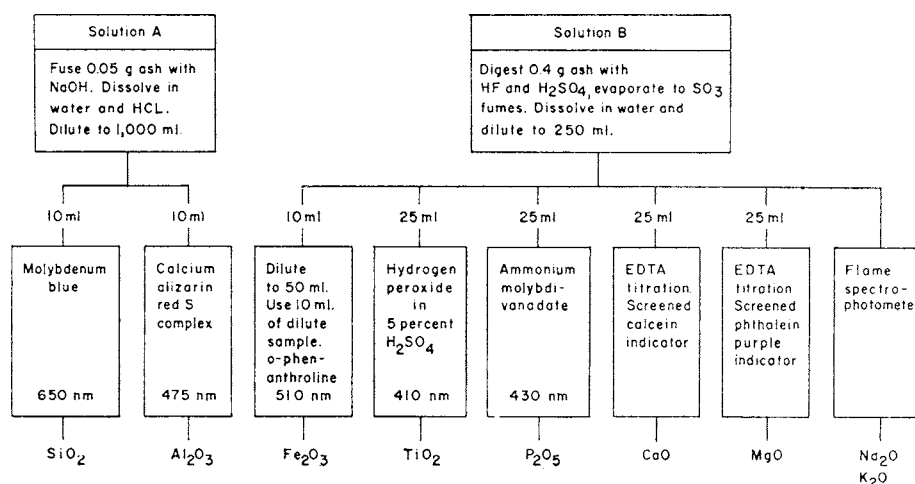


FIG. 1 Outline of Methods for Analysis of Coal Ash

### 5.3 Emission Flame Photometer.

5.4 *Muffle Furnace*—Electrically heated muffle furnace with good air circulation and capable of maintaining a temperature of approximately 750°C.

5.5 *Absorption Spectrophotometer*, visible region 380 to 780 nm.

5.6 *Sieves*, 150 and 250-µm (No. 100 and No. 60) U.S.A. standard.

## 6. Purity of Reagents and Materials

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.<sup>5</sup> 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 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water as defined in specification D 1193D 1193.

## 7. Sample

7.1 Prepare the analysis sample in accordance with Test Method D 2013D 2013 or Practice D 346D 346 by pulverizing the material to pass 250 µm (No. 60) sieve.

7.2 Analyze separate test portions for moisture and ash contents in accordance with Test Methods D 3173, D 3174, or D 5142D 3173D 3174D 5142 so that calculations to other bases can be made.

## 8. Preparation of Coal Ash and Coke Ash

8.1 *Procedure*—Prepare 3 to 5 g of ash from the analysis sample. Spread the coal or coke in a layer not over 6 mm (¼

in.) in depth in a fireclay or porcelain roasting dish. Place in a cold muffle furnace and heat gradually so that the temperature reaches 500°C in 1 h and 750°C in 2 h. Ignite to constant weight ( $\pm 0.001$  g, Note 2), at 750°C. Allow the ash to cool, transfer to an agate mortar, and grind to pass a 150-µm (No. 100) U.S.A. standard sieve. Reignite the ash at 750°C for 1 h, cool rapidly, and immediately weigh portions for analysis. If samples are stored, reignite the ash before weighing or determine loss on ignition at 750°C on a separate sample weighed out at the same time as the analysis sample. Thoroughly mix each sample before weighing.

NOTE 2—Coke may be ignited to constant weight at a temperature not exceeding 950°C if difficulty in ashing is encountered.

## 9. Preparation of Analysis Solutions (Sample, Standards, and Blank)

### 9.1 Reagents and Materials:

9.1.1 *Hydrochloric Acid (1 + 1)*—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water.

9.1.2 *Hydrofluoric Acid (sp gr 1.15)*—Concentrated hydrofluoric acid (HF).

9.1.3 *National Institute of Standards and Technology (NIST) Sample No. 99a Soda Feldspar*.<sup>6</sup>

9.1.4 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO<sub>3</sub>).

9.1.5 *Sodium Hydroxide (NaOH) pellets*.

9.1.6 *Sulfuric Acid (1 + 1)*—Mix carefully while stirring 1 volume of concentrated sulfuric acid (H<sub>2</sub>SO<sub>4</sub>, sp gr 1.84) into 1 volume of water.

### 9.2 Procedure:

9.2.1 The methods described are for typical ash samples; however, different dilutions or aliquots than those specified may be preferable to attain suitable concentrations for proper intensities for various constituents. Colors developed as specified are stable unless otherwise stated. Although the methods

<sup>5</sup> 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.

<sup>6</sup> National Institute of Standards and Technology Sample No. 99a, soda feldspar contains 65.2 % SiO<sub>2</sub> and 20.5 % Al<sub>2</sub>O<sub>3</sub>. This reagent is used for standardization purposes only.