



**Designation: D 1757 – 96 (Reapproved 2002)**

## **Standard Test Method for Sulfate Sulfur in Ash from Coal and Coke<sup>1</sup>**

This standard is issued under the fixed designation D 1757; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### **1. Scope**

1.1 This test method pertains to the determination of sulfate sulfur in coal or coke ash.

1.2 The values stated in SI units (Practice E 380) shall be regarded as the 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:**

D 1193 Specification for Reagent Water<sup>2</sup>

D 2795 Test Methods for Analysis of Coal and Coke Ash<sup>3</sup>

D 3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal<sup>3</sup>

D 3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke<sup>3</sup>

D 3682 Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes<sup>3</sup>

D 4326 Test Method for Major and Minor Elements in Coal and Coke Ash by X-Ray Fluorescence<sup>3</sup>

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

#### **2.2 British Standard:**

BS 1016, Part 14 Analysis of Coal Ash and Coke Ash<sup>5</sup>

### **3. Summary of Test Methods**

3.1 *Test Method A—Modified British Method*—A specified quantity of ash is digested in boiling dilute hydrochloric acid solution to which bromine water is added to convert sulfite that

may be present to the sulfate form. After neutralization and precipitation of iron with ammonium hydroxide, the mixture is filtered and sulfate in the filtrate is determined gravimetrically as barium sulfate ( $\text{BaSO}_4$ ).

3.2 *Test Method B—Eschka Mixture*—A specified quantity of ash and Eschka mixture are ignited together in air. The sulfates are dissolved in hot water and separated from undissolved ash residue and magnesium oxide by filtration. Sulfate in the filtrate is determined gravimetrically as barium sulfate ( $\text{BaSO}_4$ ).

### **4. Significance and Use**

4.1 Laboratory furnace temperature and furnace ventilation have an influence on  $\text{SO}_3$  retention in laboratory ash. Consequently, sulfur in ash as determined in the laboratory cannot be assumed to be equivalent to sulfur present in the mineral matter in coal or to the retention of sulfur in ash produced under the conditions of commercial utilization.

4.2 The sulfate sulfur determined by these test methods can be used to calculate the sulfur trioxide portion of ash so that the ash content or ash composition can be reported on a sulfur trioxide free basis.

### **5. Interferences**

5.1 Barium in coal ashes can result in incomplete recovery of sulfate sulfur.

5.2 High iron content can be encountered in coal and coke ash and can introduce error if, during the sulfate precipitation, the iron is either partially adsorbed by the  $\text{BaSO}_4$  precipitate or coprecipitated as iron sulfate.

### **6. Apparatus**

6.1 *Muffle Furnace*, electric, capable of maintaining a temperature of  $800 \pm 25^\circ\text{C}$  for igniting barium sulfate.

6.2 *Crucibles or Capsules*, porcelain, platinum, alundum, or silica of 10 to 15-mL capacity, for igniting  $\text{BaSO}_4$ .

### **7. Purity of Reagents**

7.1 *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,

<sup>1</sup> 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.06.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.04.

<sup>5</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.