



Designation: ~~D4326-04~~ Designation: D4326 – 11

Standard Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence¹

This standard is issued under the fixed designation D4326; 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.

~~Note—Section 13 Precision and Bias, was corrected and the year date was changed on May 26, 2004.~~

1. Scope

1.1 This test method covers the analysis of the commonly determined major and minor elements in ash from coal or coke using X-ray fluorescence (XRF) techniques.

NOTE 1—Test Method D1757 is used for determination of sulfur.

NOTE 2—Although not included in the present method, the determination of barium, strontium, and manganese may be required to yield adequate totals.

~~1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.~~

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:~~ ASTM Standards:²

D121 Terminology of Coal and Coke

D1757 Test Method for Sulfate Sulfur in Ash from Coal and Coke

D3174 Test Method for Ash in the Analysis Sample of Coal and Coke from Coal

E2 Methods of Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography)³

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

3. Terminology

3.1 Definitions—Definitions applicable to this test method are listed in D121, Terminology of Coal and Coke.

4. Summary of Test Method

~~3.1~~ 4.1 The coal or coke to be analyzed is ashed under standard conditions and ignited to constant weight. Previously ashed materials are ignited to constant weight under standard conditions. The ash is fused with lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$) or other suitable flux and either ground and pressed into a pellet or cast into a glass disk. The pellet or disk is then irradiated by an X-ray beam of short wavelength (high energy). The characteristic X-rays of the atom that are emitted or fluoresced upon absorption of the primary or incident X-rays are dispersed and intensities at selected wavelengths are measured by sensitive detectors. Detector output is related to concentration by calibration curves or by computerized data-handling equipment.

~~3.2~~ 4.2 The K spectral lines are used for all of the elements determined by this procedure.

~~3.3~~ 4.3 All elements are determined as the element and reported as the oxide and include Si, Al, Fe, Ca, Mg, Na, K, P, Ti, Mn, Sr, and Ba.

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 Annual Book of ASTM Standards, Vol 05.05.

For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For

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4. Significance and Use

4.1 A compositional analysis of ash is used in describing the quality of coal for its complete characterization. Ash composition is useful in predicting slagging and fouling characteristics of combusted materials as well as the potential utilization of ash by-products.

4.2 The chemical composition of laboratory prepared coal or coke ash is rarely, if ever, representative of the composition of the mineral matter in the coal because the ashing process can alter some minerals. However, it can approximate the composition of the fly ash and slag resulting from commercial combustion of coal or coke.

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6. Apparatus

6.1

6.1 Ashing Furnace, with air circulation as specified in Test Method D3174 and capable of having its temperature regulated between 700 and 750°C.

6.2

6.2 Fusion Furnace or Fluxing Device, with an operating temperature of at least 1000°C.

6.3

6.3 Fusion Crucibles, either high-purity graphite (22 mm high and 19 mm wide, inside diameter) or platinum-gold alloy of a similar or larger capacity.

6.4

6.4 Pulverizers, including agate, mullite or tungsten carbide mortar, and pestle, minimum capacity 25 mL.

6.5

6.5 Sieves, 250- μm (No. 60) and 75- μm (No. 200) U.S.A standard sieve as specified in Specification E11.

6.6

6.6 Compactor—A press equipped with a gage enabling reproducible pressures (exceeding 1.72×10^8 Pa (25 000 psi)) if pressed pellets are utilized.

6.7

6.7 Excitation Source, with a stable electrical power supply ($\pm 1\%$) and a high-intensity, short-wavelength X-ray capability.

6.8

6.8 Spectrometer—A wavelength or energy dispersive system equipped with a vacuum sample chamber.

6.8.1

6.8.1 Analyzing Crystal (Wavelength Units)—The choice of the analyzing crystal is made on the basis of the element to be determined. An attempt should be made to use the crystal that yields the maximum sensitivity with minimum interferences. The same crystal must be used for standards and unknowns.

6.8.2

6.8.2 Detectors—Scintillation and gas-flow counters are used with wavelength systems while lithium-drifted diodes are used for energy dispersive systems.

6.

7. Reagents

7.1

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, where such specifications are available.³ 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.

³ *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.