



# Standard Test Methods for Analysis of Metal Bearing Ores and Related Materials for Carbon, Sulfur, and Acid-Base Characteristics<sup>1</sup>

This standard is issued under the fixed designation E1915; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 These test methods cover the determination of total carbon and sulfur and acid-base characteristics in metal bearing ores and related materials such as leach residues, tailings, and waste rock within the following ranges:

Analyte	Application Range, %	Quantitative Range, %
Total Carbon	0 to 10	0.08 to 10
Total Sulfur	0 to 8.8	0.023 to 8.8

NOTE 1—The test methods were tested over the following ranges:

Total Carbon	0.01 % to 5.87 %
Total Sulfur	0.0002 % to 4.70 %
Residual Carbon from Pyrolysis	0.002 % to 4.97 %
Residual Sulfur from Pyrolysis	0.014 % to 1.54 %
Pyrolysis Loss Sulfur	0 % to 4.42 %
Hydrochloric Acid Insoluble Carbon	0.025 % to 0.47 %
Hydrochloric Acid Loss Carbon	0 % to 5.78 %
Hydrochloric Acid Insoluble Sulfur	0.012 % to 4.20 %
Acid Neutralization Potential Acidity Titration	-1.0 % to 100 %
Acid Neutralization Potential Acidity Titration Low Range	-1.0 % to 2 % CaCO <sub>3</sub>
Nitric Acid Insoluble Sulfur	0.006 % to 0.924 %
Nitric Acid Loss Sulfur	-0.08 % to 4.19 %
Sodium Carbonate Insoluble Sulfur	0.007 % to 3.78 %

1.2 The quantitative ranges for the partial decomposition test methods are dependent on the mineralogy of the samples being tested. The user of these test methods is advised to conduct an interlaboratory study in accordance with Practice E1601 on the test methods selected for use at a particular mining site, in order to establish the quantitative ranges for these test methods on a site-specific basis.

1.3 The test methods appear in the following order:

	Sections
Carbon and Sulfur, Total	10.1-10.9
Carbon and Sulfur, Residual from Pyrolysis	10.10-10.18
Carbon and Sulfur, Hydrochloric Acid Insoluble	10.19-10.27
Acid Neutralization Potential Acidity Titration	10.28-10.36
Acid Neutralization Potential Acidity Titration Low Range	10.37-10.46
Sulfur, Nitric Acid Insoluble	10.47-10.55
Sulfur, Sodium Carbonate Insoluble	10.56-10.64

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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1.4 The values stated in SI units are to be regarded as standard.

1.5 *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.* Specific warning statements are given in Section 6.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- D1067 Test Methods for Acidity or Alkalinity of Water
- D1193 Specification for Reagent Water
- D1976 Test Method for Elements in Water by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy
- D5673 Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry
- D5744 Test Method for Laboratory Weathering of Solid Materials Using a Humidity Cell
- D6234 Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
- E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
- E1950 Practice for Reporting Results from Methods of Chemical Analysis

<sup>2</sup> 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.

## E2242 Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology E135.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *standardization, v*—analysis of samples with known values or known additions, prior to and within groups of test samples to assure accuracy.

3.2.2 *standardization sample, n*—calibration mixtures or samples with known values or known additions that are analyzed with test samples to assure accuracy of analysis.

### 4. Significance and Use

4.1 These test methods are primarily intended to test materials for compliance with compositional specifications and for monitoring.

4.1.1 The determination of carbon and sulfur and acid neutralization potential in ores and related materials is necessary to classify ores for metallurgical processing and to classify waste materials from the mining and processing of ores such as leach residues, waste rock, and tailings according to their potential to generate acid in the environment. This information is useful during mine development to assist in mining and mineral processing operations and for proper disposal of waste materials.

4.1.1.1 The use of the acid neutralization potential titration low range method is most useful where acidity is present in the samples and when acid potential by titration is desired in the uncertain content range below 2 % CaCO<sub>3</sub>.

4.1.2 These test methods are also used to isolate minerals based on carbon and sulfur contents of metal-bearing ores and related materials so that acid-base accounting can be performed (that is, carbonate mineral acid neutralization potential (ANP) minus sulfide-sulfur mineral acid generation potential (AGP) = net calcium carbonate (NCC)).

4.1.3 Additionally, the carbon hydrochloric acid insoluble test method has utility to identify the amount of organic carbon contained in gold ores so that potential for preg robbing can be identified and rectified through established pretreatment methods prior to cyanidation. **Warning**—Pyrolysis pretreatment at 550 °C has a potential to thermally decompose some carbonate minerals: (1) transition metal carbonates (for example, siderite, FeCO<sub>3</sub>, and rhodochrosite, MnCO<sub>3</sub>) decompose, yielding carbon dioxide, CO<sub>2</sub>, in the range of 220 °C to 520 °C; (2) calcite decomposes slightly between 300 °C and 500 °C, although most decomposition occurs above 550 °C; (3) dolomite decomposes at 800 °C to 900 °C (Hammack, 1994, p. 440).<sup>3</sup>

4.2 These test methods also may be used for the classification of rock to be used in construction, where the potential to generate acid under environmental conditions exists.

4.3 It is assumed that the users of these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices such as those described in Guide E882 must be followed.

### 5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>4</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.

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

### 6. Hazards

6.1 For hazards to be observed in the use of reagents and apparatus in these test methods, refer to Practices E50. Use care when handling hot crucibles or boats and when operating furnaces to avoid personal injury by either burn or electrical shock.

### 7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places as directed in the Rounding Method of Practice E29.

### 8. Interlaboratory Studies

8.1 These test methods have been evaluated in accordance with Practice E1601 unless otherwise noted in the precision and bias section. The lower limit in the scope of these test methods specifies the lowest analyte content that may be analyzed with an acceptable error. A warning statement is included in the scope for test methods not observing this convention.

8.2 *Site-Specific Quantitative Ranges*—An interlaboratory study may be conducted in accordance with Practice E1601 to establish quantitative ranges for the partial decomposition test methods selected for a particular site. Test samples shall be selected for each alteration or lithologic unit, or both, containing high and low contents of carbon and sulfur minerals. Each test sample must be analyzed in rapid succession for total carbon and sulfur followed by the different partial decomposition treatments selected in order to minimize the between-method variation.

<sup>3</sup> Hammack, R. W., "Evolved-Gas Analysis: A Method for Determining Pyrite, Marcasite, and Alkaline-Earth Carbonates," *Environmental Geochemistry of Sulfide Oxidation*, Alpers, C., and Blowes, D., eds., Chapter 28, ACS Symposium Series 550, American Chemical Society, Washington, D.C., 1994, pp. 431–444.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, http://www.usp.org.

## 9. Sampling and Sample Preparation

9.1 *Materials Safety*—Samples must be prepared, stored, and disposed of in accordance with the materials and safety guidelines in Practices E50.

9.2 *Prepared Sample*—Dry a representative portion of the gross sample at 80 °C to constant mass in order to minimize sulfide mineral oxidation. Pulverize or grind the laboratory sample until 100 % passes a No. 100 (150- $\mu$ m) sieve.

NOTE 2—Results from the interlaboratory study suggest that it may be necessary to grind samples to pass a No. 200 (75- $\mu$ m) sieve in order to improve precision for samples containing low contents of carbon or sulfur.

9.3 *Diluted Sample*—If the content of sulfur in the test material exceeds 1.75 % for the minimum range instrument, prepare a diluted sample as in 9.3.1.

9.3.1 Weigh 10.0 g  $\pm$  0.1 g prepared sample and combine with 40.0 g  $\pm$  0.1 g dry silica (SiO<sub>2</sub>). Grind the mixture in a ring and puck mill, or equivalent, until 100 % will pass through a No. 100 (150- $\mu$ m) sieve; mix, and store in a 250-mL glass bottle.

## 10. Procedures

### TOTAL CARBON AND SULFUR

10.1 *Scope*—This test method covers the determination of total carbon in the content range between 0.1 % and 10 % and total sulfur contents in the range between 0.1 % and 8.8 %.

#### 10.2 Summary of Test Method:

10.2.1 The carbon in the test sample is converted to carbon dioxide and the sulfur to sulfur dioxide by combustion in a stream of oxygen.

10.2.2 The amount of carbon dioxide and sulfur dioxide are measured by infrared absorption.

#### 10.3 Apparatus:

10.3.1 *Combustion-Infrared Spectrophotometer*, equipped with a combustion chamber, oxygen carrier stream, and infrared absorption detector, suitable for analysis of sulfur in a minimum range instrument from 0.1 % to 1.75 % or in a maximum range instrument from 0.1 % to 8.8 % and carbon in the range of 0.1 % to 10 %, using 0.2-g test portions of ores and related materials. Instruments, such as those shown in Test Methods E1019 that can be shown to give equivalent results may also be used for these test methods.

#### 10.4 Reagents and Materials:

##### 10.4.1 Reagents:

10.4.1.1 *Barium Sulfate (BaSO<sub>4</sub>)*, Anhydrous, contains 13.74 % sulfur (purity 99.9 % minimum). Dry 100 g at 120 °C for 2 h and store in a 250-mL glass bottle.

10.4.1.2 *Blank Reference Sample*—Prepare a blank reference sample by pulverizing or grinding 100 g of SiO<sub>2</sub> (see 10.4.1.6), to pass through a No. 100 (150- $\mu$ m) sieve, mix, and store in a 250-mL glass bottle. This blank contains 0.00 % carbon and 0.00 % sulfur.

10.4.1.3 *Calcium Carbonate (CaCO<sub>3</sub>)*, Anhydrous, contains 12.00 % carbon (purity 99.9 % minimum). Dry 100 g for 2 h at 120 °C and store in a 250-mL glass bottle.

10.4.1.4 *Calibration Mixture A*—(1 g = 20 mg C and 20 mg S)—Combine 16.67 g CaCO<sub>3</sub>, 14.56 g BaSO<sub>4</sub>, and

68.77 g SiO<sub>2</sub> in a ring and puck grinding mill or equivalent device. Grind until 100 % passes through a No. 100 (150- $\mu$ m) sieve, pass the mixture through the screen to break up any lumps, mix, and store in a glass bottle. This mixture contains 2.00 % carbon and 2.00 % sulfur.

(1) Alternatively, grind the reagents separately, mix, and pass through the screen prior to final mixing.

10.4.1.5 *Calibration Mixtures*—Transfer 4.00 g, 10.00 g, 20.00 g, and 30.00 g of Calibration Mixture A to ring and puck grinding mills or equivalent devices. Add the amount of dried SiO<sub>2</sub> needed to bring the total mass to 40.0 g in each mill, grind to 100 % passing a No. 100 (150- $\mu$ m) sieve, pass the mixture through the screen, mix, and store in 250-mL glass bottles. These mixtures contain: 0.2 %, 0.5 %, 1.0 %, and 1.5 % for both carbon and sulfur.

(1) Alternatively, grind the reagents separately, mix, and pass through the screen prior to final mixing.

(2) Commercially-produced calibration mixtures, which meet these specifications, may also be used.

10.4.1.6 *Silica (SiO<sub>2</sub>)* (purity 99.9 % minimum), Ottawa sand, washed and ignited, containing less than 0.01 % carbon and 0.01 % sulfur. Dry at 120 °C for 2 h and store in a 250-mL glass bottle.

10.4.1.7 *Tungstic Acid (H<sub>2</sub>WO<sub>4</sub>)* (purity 99 % minimum).

10.4.1.8 *Vanadium Pentoxide (V<sub>2</sub>O<sub>5</sub>)* (purity 99 % minimum).

#### 10.4.2 Materials:

10.4.2.1 *Crucibles or boats*, suitable for combustion analyses.

#### 10.5 Calibration and Standardization:

10.5.1 *Apparatus*—Operate and calibrate the instrument in accordance with the manufacturer's instructions. Resistance furnace instruments require the use of V<sub>2</sub>O<sub>5</sub> or H<sub>2</sub>WO<sub>4</sub> for the determination of sulfur in this test method. Use a 0.200 g  $\pm$  0.01 g mass for all calibration mixtures, reference materials, blank reference materials, test samples, and diluted test samples in this test method.

10.5.1.1 Certain instruments may require different sample masses for certain content ranges, which is permissible as long as the precision and bias requirements of these test methods are fulfilled.

10.5.2 Ignite the crucibles or boats for test samples and standard samples in a muffle furnace for 1 h at 550 °C  $\pm$  10 °C.

10.5.3 *Laboratory Test Method Performance Demonstration*—A demonstration of laboratory test method performance must be performed before this test method may be used in a laboratory for the first time. This demonstration is particularly important if the laboratory needs to modify the test method in any way. The demonstration must be repeated whenever the test method is significantly modified.

10.5.3.1 *Linearity Verification*—Measure total carbon and sulfur for the blank reference sample, calibration mixtures, BaSO<sub>4</sub>, and CaCO<sub>3</sub> in increasing order using the same mass of calibration mixtures selected for test samples, in accordance with the manufacturer's instructions. Record the calibration mixture masses used and the carbon and sulfur results from the instrument. Check for linearity by linear regression or by a graphical method to meet a deviation less than 10 % relative