



# Standard Test Methods for Analysis of Metal Bearing Ores and Related Materials by Combustion Infrared Absorption Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E 1915; 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 covers the determination of total carbon and sulfur in metal bearing ores and related materials such as tailings and waste rock within the following ranges:

Analyte	Application Range, %	Quantitative Range, %
Total Carbon	0 to 10	0.1 to 10
Total Sulfur	0 to 8.8	0.1 to 8.8

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 are advised to conduct an interlaboratory study in accordance with Practice E 1601 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:

Carbon and Sulfur, Residual from Pyrolysis	Sections 12.7 — 12.12
Carbon and Sulfur, Total	12 — 12.6

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 7.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- D 1193 Specifications for Reagent Water<sup>2</sup>
- E 29 Practice For Using Significant Digits in Test Data to Determine Conformance With Specifications<sup>3</sup>
- E 50 Practices for Apparatus, Reagents and Safety Precautions for Chemical Analysis of Metals<sup>4</sup>
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>4</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.05.

E 882 Guide for Accountability and Quality Control in the Chemical Analysis of Metals<sup>5</sup>

E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, Oxygen and Hydrogen in Steel and in Iron, Nickel and Cobalt Alloys<sup>5</sup>

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method<sup>5</sup>

E 1950 Practice for Reporting Results from Methods of Chemical Analysis<sup>5</sup>

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology E 135.

## 4. Significance and Use

4.1 These test methods are primarily intended to test materials for compliance with compositional specifications and for monitoring. The determination of carbon and sulfur 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 spoils, 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 proper disposal of waste materials.

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 E 882 must be followed.

## 5. Apparatus

5.1 *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

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 03.06.

0.1 to 10 %, using 0.2-g test portions in ores and related materials. Instruments, such as those shown in Test Methods E 1019 and in the section entitled Apparatus for Determination of Total Carbon by Direct Combustion and the section entitled Apparatus for the Determination of Sulfur by Direct Combustion of Practices E 50, that can be shown to give equivalent results may also be used for these test methods.

## 6. 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 conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available<sup>6</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 Reagents:

6.2.1 *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.

6.2.2 *Blank Reference Sample*—Prepare a blank reference sample by pulverizing or grinding 100 g silica (see 6.2.6), passing it through a No. 100 (150- $\mu$ m) sieve, and mixing and storing it in a 250-mL glass bottle. This blank contains 0.00 % carbon and sulfur.

6.2.3 *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.

6.2.4 *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 sulfur.

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

6.2.5 *Calibration Mixtures*—Transfer 4.00, 10.00, 20.00 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 weight 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.

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

6.2.5.2 Commercially produced calibration mixtures, that meet the specifications of 6.2.5, may also be used.

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

<sup>6</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.

## 7. Hazards

7.1 For hazards to be observed in the use of reagents and apparatus in these test methods, refer to Practice E 50. Use care when handling hot crucibles or boats and when operating furnaces to avoid personal injury by either burn or electrical shock.

## 8. Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places as directed in the Rounding Method of Practice E 29.

## 9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E 1601 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.

9.2 *Site-Specific Quantitative Ranges*—An interlaboratory study may be conducted in accordance with Practice E 1601 to establish quantitative ranges for the partial decomposition test methods selected for a particular site. Test samples shall be selected for each lithologic unit containing high and low concentrations 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.

## 10. Sampling and Sample Preparation

10.1 *Materials Safety*—Samples must be prepared, stored and disposed of in accordance with the materials and safety guidelines in Practices E 50.

10.2 *Prepared Sample*—Dry a representative portion of the gross sample at 80°C to constant weight. Pulverize or grind the laboratory sample until 100 % passes a No. 100 (150- $\mu$ m) sieve.

NOTE 1—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 concentrations of carbon or sulfur.

10.3 *Diluted Sample*—If the concentration of sulfur in the test material exceeds 1.75 % for the minimum range instrument, prepare a diluted sample as in 10.3.1.

10.3.1 Weigh  $10.0 \pm 0.1$  g prepared sample and combine with  $40.0 \pm 0.1$  g dry 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.

## 11. Calibration and Standardization

11.1 *Apparatus*—Operate and calibrate the instrument according to the manufacturer's instructions. Resistance furnace instruments require the use of vanadium pentoxide or tungstic acid for the determination of sulfur in these test methods. Use a  $0.200 \pm 0.1$  g weight for all calibration mixtures, reference materials, blank reference materials, test samples and diluted test samples in these test methods.

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

11.2 Ignite the crucibles or boats for test samples and standard samples in a muffle furnace for 1 h at  $550 \pm 10^\circ\text{C}$ .

11.3 *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.

11.3.1 *Linearity Verification*—Measure total carbon and sulfur for the blank reference sample, calibration mixtures, barium sulfate and calcium carbonate in increasing order using the same weight of calibration mixtures selected for test samples, in accordance with the manufacturer's instructions. Record the calibration mixture weights used and the carbon and sulfur results measured by the instrument. Check for linearity by linear regression or by a graphical method to meet a deviation less than 10 % relative for each of the calibration material results at or above a concentration of 0.2 % carbon and sulfur and a correlation coefficient of at least 0.99. Correct any problems with the instrument before proceeding with the analysis of test samples.

11.3.1.1 Linearity may also be verified by the use of barium sulfate and calcium carbonate weights equivalent to the content of the calibration mixtures.

11.3.2 *Blank Sample Precision Verification*—Analyze ten replicates of the blank reference sample. If the standard deviation of the replicate analyses exceeds 0.02 % for carbon or 0.01 % for sulfur, correct any instrumental problems and repeat the blank sample precision verification before proceeding with test method implementation.

11.3.3 *Low Calibration Mixture Precision Verification*—Analyze four replicates of the 0.2 % calibration mixture. If any result for the 0.2 % calibration mixture exceeds the limits shown in Table 1, correct any instrumental problems and repeat the low calibration mixture precision verification before proceeding with test method implementation.

#### 11.4 *Method Quality Control:*

11.4.1 *Calibration Verification*—Analyze a calibration mixture with a concentration greater than or equal to 0.5 % carbon and sulfur prior to and within each group of fifty test samples. If the calibration mixture result exceeds the limits in Table 1, correct any instrumental problems and repeat the linearity

verification before proceeding with analysis of test samples, and discard the results since the last acceptable quality control sample result had been obtained.

11.4.2 *Blank Reference Sample*—Analyze a blank reference sample before analysis of test samples and within each group of fifty test samples. If the result for the blank reference sample exceeds the limits in Table 1 for the 0.0 % calibration mixture, correct any instrumental problems and repeat the analysis of the blank reference sample before proceeding with analysis of test samples, and discard the results since the last acceptable quality control sample result had been obtained.

11.4.3 *Reference Sample*—Analyze a reference sample, certified for total carbon and total sulfur before analysis of test samples for total carbon and sulfur and within each group of fifty test samples. If the difference of the reference sample and the reference value for the reference sample exceeds the limits shown in Table 1 for materials of comparable concentration, correct any instrumental problems and repeat the analysis of the reference material, and discard the results since the last acceptable quality control sample result had been obtained.

11.4.4 *Control Sample*—Analyze the 0.2 % calibration mixture prior to and within each group of fifty test samples. If the result for the control sample exceeds the limits shown in Table 1 for the 0.2 % calibration mixture, correct any instrumental problems and repeat the analysis of the control sample before proceeding with analysis of test samples, and discard the results since the last acceptable quality control sample result had been obtained.

11.4.5 *Standard Addition Sample*—Analyze a standard addition sample prior to analysis of each group of fifty test samples by preparing a duplicate of the first test sample in the group and adding an equal weight of the 0.5 % calibration mixture just prior to determination of carbon and sulfur. Calculate the reference values for the standard addition sample by adding 0.5 % to the carbon and sulfur results for the test sample performed without the standard addition and divide the sum by two. If the difference of any result for the standard addition sample and the reference value exceeds the limits shown in Table 1 for materials of comparable concentration, correct any instrumental problems and repeat the standard addition sample analysis before proceeding with analysis of test samples, and discard the results since the last acceptable quality control sample result had been obtained.

NOTE 2—Add the 0.5 % calibration mixture after the decomposition procedure but before the analysis step for test method quality control of partial decomposition procedures.

## 12. Procedures

### TOTAL CARBON AND SULFUR

12.1 *Scope*—This test method covers the determination of total carbon in the concentration range between 0.1 and 10 % and total sulfur concentrations in the range between 0.1 and 8.8 %.

#### 12.2 *Summary of Test Method:*

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

12.2.2 The amount of carbon dioxide and sulfur dioxide are

**TABLE 1 Calibration Mixture 95 % Confidence Limits from Interlaboratory Testing**

Mixture	Carbon, %		Sulfur, %	
	Minimum	Maximum	Minimum	Maximum
0.0	-0.02	0.04	-0.01	0.01
0.2	0.16	0.25	0.12	0.26
0.5	0.44	0.55	0.42	0.55
1.0	0.92	1.08	0.85	1.14
1.5	1.42	1.59	1.34	1.62
2.0	1.87	2.13	1.78	2.16
BaSO <sub>4</sub>	...	...	12.4	14.5
CaCO <sub>3</sub>	10.9	12.8	...	...