Designation: E 1915 – ~~99~~01

Standard Test Methods for Analysis of Metal Bearing Ores and Related Materials by Combustion Infrared Absorption Spectrometry[1]

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 (ε) 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 Carbon	0 to 10	0.08 to 10
~~Total Sulfur~~	~~0 to 8.8~~	~~0.1 to 8.8~~
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 %.

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:

	Sections
Carbon and Sulfur, Hydrochloric Acid Insoluble	12.13 – 12.18
~~Carbon and Sulfur, Residual from Pyrolysis~~	~~12.7 — 12.12~~
Carbon and Sulfur, Residual from Pyrolysis	12.7 – 12.12
~~Carbon and Sulfur, Total~~	~~12 — 12.6~~
Carbon and Sulfur, Total	12.1 – 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[2]

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[1] This test method is under the jurisdiction of ASTM Committee ~~E-1~~ E01 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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E 29 Practice ~~For~~ for Using Significant Digits in Test Data to Determine Conformance ~~W~~ with Specifications[3]
E 50 Practices for Apparatus, Reagents and Safety ~~Precautions~~ Considerations for Chemical Analysis of Metals, Ores, and Related Materials[4]
E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials[4]
E 882 Guide for Accountability and Quality Control in the Chemical Analysis ~~of Metals~~ Laboratory[5]
E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, ~~Oxygen~~ and ~~Hydrogen~~ Oxygen in Steel and in Iron, Nickel and Cobalt Alloys[5]
E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method[5]
E 1950 Practice for Reporting Results from Methods of Chemical Analysis[5]

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 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[6]. 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 reagent water as defined in Type I of Specification D 1193.

6.3 *Reagents*:

6.~~2~~3.1 *Barium Sulfate ($BaSO_4$), 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~~3.2 *Blank Reference Sample*—Prepare a blank reference sample by pulverizing or grinding 100 g silica (see ~~6.2.6), passing it~~ 6.3.6), pass through a No. 100 (150-µm) sieve, and mixing and storing ~~it~~ in a 250-mL glass bottle. This blank contains 0.00 % carbon and sulfur.

6.~~2~~3.3 *Calcium Carbonate ($CaCO_3$), 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~~3.4 *Calibration Mixture A—(1 g = 20 mg C and 20 mg S)*—Combine 16.67 g $CaCO_3$, 14.56 g $BaSO_4$ and 68.77 g SiO_2 in a ring and puck grinding mill or equivalent device. Grind until 100 % passes through a No. 100 (150-µm) 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.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.
[3] *Annual Book of ASTM Standards*, Vol 14.02.
[4] *Annual Book of ASTM Standards*, Vol 03.05.
[5] *Annual Book of ASTM Standards*, Vol 03.06.
[6] *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. Pharmacop~~eutic~~eal Convention, Inc. (USPC), Rockville, MD.

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

6.23.5 *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_2 needed to bring the total weight to 40.0 g in each mill, grind to 100 % passing a No. 100 (150-µm) 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.

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

6.23.5.2 Commercially–produced calibration mixtures, ~~that~~ which meet ~~the specifications of 6.2.5,~~ these specifications, may also be used.

6.23.6 *Silica (SiO_2)*, (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.

6.4 *Materials*:

6.4.1 *Glass Filters*—Fine-porosity glass micro filters, carbon content must be less than 0.15 %, sulfur content must be less than 0.05 % and the filter weight must be less than 0.2 g.

6.4.1.1 Filtering crucibles may also be used if they are shown to provide equivalent results.

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-µm) sieve.

NOTE ~~1~~2—Results from the interlaboratory study suggest that it may be necessary to grind samples to pass a No. 200 (75-µm) 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 ± 0.1 g prepared sample and combine with 40.0 ± 0.1 g dry SiO_2. Grind the mixture in a ring and puck mill, or equivalent, until 100 % will pass through a No. 100 (150-µm) 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 tungstenic acid for the determination of sulfur in these test methods. Use a 0.200 ± 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 ± 10°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~~3—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

TABLE 1 Calibration Mixture 95 % Confidence Limits from Interlaboratory Testing

Mixture	~~Carbon, %~~		~~Sulfur, %~~		
Minimum	Maximum	Minimum	Maximum		
Min., % C	Max., % C	Min., % S	Max., % S		
~~0.0~~	~~– 0.02~~	~~–0.04~~	~~– 0.01~~	~~–0.01~~	
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	
~~$BaSo_4$~~	~~. . .~~	~~. . .~~	~~12.4~~	~~14.5~~	
$BaSO_4$	. . .	. . .	12.4	14.5	
~~$CaCo_3$~~	~~10.9~~	~~12.8~~	~~. . .~~	~~. . .~~	
$CaCO_3$	10.9	12.8	. . .	. . .	

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 measured by infrared absorption.

12.3 *Interferences*—The elements normally present in ores and related materials do not interfere with this test method.

12.4 *Procedure*:

12.4.1 Ignite the crucibles or boats for test samples and standard samples in a muffle furnace for 1 h at 550 ± 10°C, unless it is demonstrated that omission of this step does not degrade the precision and bias of the analysis.

12.4.2 *Test Samples*—Transfer test samples, diluted test samples and standardization samples using 0.200 ± 0.01 g into the crucible or boat used for instrumental analysis and record the weight. Use of a different sample weight may be required on some instruments for some samples (see 11.1.1).

12.4.3 *Duplicate Test Sample*—Analyze a duplicate test sample within each group of fifty test samples. If the difference of the duplicate results exceeds the limits shown in Table 1 for a material of comparable concentration, discard the results since the last acceptable quality control sample result had been obtained, correct any sample preparation or instrumental problems and repeat the analyses from 12.4.2.

12.4.4 *Analysis*:

12.4.4.1 Analyze quality control samples before each batch of test samples and within each group of ten test samples as directed in 11.4. Measure the carbon and sulfur concentrations for quality control samples, test samples and diluted test samples in percent according to the instrument manufacturer's instructions, and record the measurements.

12.4.4.2 Continue analysis until the batch of test samples is completed, a quality control sample or duplicate test sample result deviates more than the limits shown in Table 1, for a material of comparable concentration.

12.5 *Calculation*:

12.5.1 Calculate the total carbon and sulfur concentrations for the test samples according to the manufacturer's instructions.

12.5.2 Round the results above 0.1 % to the nearest 0.01 % and record as total carbon or sulfur. Enclose results from 0.03 to 0.1 % in parentheses and below 0.03 % in parentheses followed by an asterisk in accordance with Guide E 1950.

12.5.3 *Over-Range Results*—If the sulfur result exceeds 1.75 % for the minimum range instrument, discard the result and repeat the procedure from 12.4.2 with the diluted sample. Multiply the diluted test sample result by five and round to the nearest 0.1 %.

12.5.3.1 Alternatively, use a lower sample weight for the analysis as specified in 11.1.1.

12.6 *Precision and Bias*[7]

12.6.1 *Precision*—Eleven laboratories cooperated in testing this test method, providing ten sets of data for carbon and eleven sets of data for sulfur, and obtained the precision data summarized in Tables 2 and 3.

12.6.2 *Bias*—The accuracy of this test method for carbon and sulfur is deemed satisfactory based on the values in Tables 4 and 5. Users are encouraged to employ these or similar reference materials to verify that this test method is performing accurately in their laboratory.

NOTE 4—The user of this test method is cautioned that the method may not be quantitative for reporting above a reproducibility index (R) of 50 % relative, according to Practice E 1601. The user is advised to take this into account, in addition to the mineralogy of the sample, when interpreting the results for this test method.

RESIDUAL CARBON AND SULFUR FROM PYROLYSIS

12.7 *Scope*—This test method covers the determination of residual carbon from pyrolysis in the concentration range between 0.1 and 10 % and residual sulfur from pyrolysis concentrations in the range between 0.1 and 8.8 %.

12.8 *Summary of Test Method*:

12.8.1 The test sample is ignited in a muffle furnace prior to instrumental analysis where the carbon in the test sample is converted to carbon dioxide and the sulfur to sulfur dioxide by combustion in a stream of oxygen.

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

12.9 *Interferences*—The elements normally present in ores and related materials do not interfere with this test method. Use of adequate draft in the muffle furnace is necessary to avoid excessive adsorption of sulfur gasses on the solid phase of the test samples, leading to low sulfur loss by pyrolysis.

12.10 *Procedure*:

12.10.1 Ignite the crucibles or boats for test samples and standard samples in a muffle furnace for 1 h at 550 ± 10°C (see ~~Note 1).~~ 12.4.1).

12.10.2 *Test Samples*—Transfer test samples, diluted test samples and standard addition samples using 0.200 ± 0.01 g into the crucible or boat used for instrumental analysis and record the weight. Use of a different sample weight may be required on some instruments for some samples (see 11.1.1).

12.10.3 *Ignition*—Ignite the crucibles or boats containing the test samples and standard addition samples in a muffle furnace for one hour at 550 ± 10°C.

[7] Supporting data have been filed at ASTM Headquarters. Request RR: E01-1023.

TABLE 2 Statistical Information — Total Carbon

Test Material	Number of Laboratories	Carbon Found, %	~~SD, m~~Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
~~Blank~~	~~7~~	~~0.01~~	~~0.004~~	~~0.03~~	~~300~~
Blank	7	0.012	0.004	0.034	300
~~Ottawa sand~~	~~10~~	~~0.021~~	~~0.011~~	~~0.048~~	~~230~~
Ottawa Sand	10	0.021	0.011	0.0477	230
~~Inert diorite~~	~~7~~	~~0.05~~	~~0.005~~	~~0.04~~	~~80~~
Inert Diorite	7	0.050	0.005	0.037	74
~~Inert andesite~~	~~7~~	~~0.09~~	~~0.004~~	~~0.05~~	~~56~~
Inert Andesite	7	0.090	0.004	0.054	59
~~Autoclave feed ore~~	~~10~~	~~0.086~~	~~0.016~~	~~0.12~~	~~133~~
Autoclave Feed Ore	10	0.086	0.016	0.115	133
~~Calibration mixture 0.1~~	~~7~~	~~0.12~~	~~0.007~~	~~0.05~~	~~42~~
Calibration Mixture 0.1	7	0.117	0.007	0.049	42
~~Duluth waste rock~~	~~10~~	~~0.142~~	~~0.017~~	~~0.11~~	~~79~~
Duluth Waste Rock	10	0.142	0.017	0.112	79
~~Spiked andesite~~	~~6~~	~~0.29~~	~~0.008~~	~~0.05~~	~~17~~
Spiked Andesite	6	0.292	0.008	0.051	17
~~Reclamation tailings~~	~~10~~	~~0.462~~	~~0.025~~	~~0.22~~	~~48~~
Reclamation Tailings	10	0.462	0.025	0.223	48
~~Vinini waste rock~~	~~10~~	~~0.771~~	~~0.024~~	~~0.18~~	~~23~~
Vinini Waste Rock	10	0.771	0.024	0.180	23
~~Pit rock~~	~~10~~	~~0.80~~	~~0.025~~	~~0.117~~	~~15~~
Pit Rock	10	0.800	0.025	0.117	15
~~Diorite gneiss~~	~~10~~	~~1.04~~	~~0.032~~	~~0.17~~	~~16~~
Diorite Gneiss	10	1.04	0.032	0.170	16
~~Zinc plant tailings~~	~~10~~	~~5.87~~	~~0.055~~	~~0.494~~	~~8~~
Zinc Plant Tailings	10	5.87	0.055	0.494	8
~~Refractory gold ore~~	~~10~~	~~5.70~~	~~0.038~~	~~0.478~~	~~8~~
Refractory Gold Ore	10	5.70	0.038	0.478	8

12.10.4 *Duplicate Test Sample*—Analyze a duplicate test sample within each group of fifty test samples. If the difference of the duplicate results exceeds the limits shown in Table 1 for a material of comparable concentration, discard the results since the last acceptable quality control sample result had been obtained, correct any sample preparation or instrumental problems and repeat the analyses from 12.10.2.

12.10.5 *Analysis*:

12.10.5.1 Analyze quality control samples before each batch of test samples and within each group of ten test samples as directed in 11.4. Measure the carbon and sulfur concentrations for quality control samples, test samples and diluted test samples in percent according to the instrument manufacturer's instructions and record the measurements.

12.10.5.2 Continue analysis until the batch of test samples is completed, a quality control sample or duplicate test sample result deviates more than the limits shown in Table 1 for a material of comparable concentration.

12.11 *Calculation*:

12.11.1 Calculate the residual carbon and sulfur from pyrolysis concentrations for the test samples according to the manufacturer's instructions.

12.11.2 Calculate the pyrolysis loss sulfur, %, A, as follows:

$$A = B - C \quad (1)$$

where:

B = total sulfur result, %, and

C = residual sulfur from pyrolysis result, %.

12.11.3 Round the results to the nearest 0.01 % and record as pyrolysis residual carbon, pyrolysis residual sulfur, or pyrolysis loss sulfur, at or above the lower scope limit established during interlaboratory testing. Report results below the lower scope limits

TABLE 3 Statistical Information — Total Sulfur

Test Material	Number of Laboratories	Sulfur Found, %	~~SD, m~~Min., SD(S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
~~Blank~~	~~7~~	~~0.0002~~	~~0.002~~	~~0.01~~	~~5000~~
Blank	7	0.0002	0.002	0.010	5000
~~Ottawa sand~~	~~11~~	~~0.004~~	~~0.003~~	~~0.013~~	~~311~~
Ottawa Sand	11	0.004	0.003	0.0133	312
~~Diorite gneiss~~	~~11~~	~~0.014~~	~~0.007~~	~~0.039~~	~~283~~
Diorite Gneiss	11	0.014	0.007	0.039	283
~~Calibration mixture 0.1~~	~~7~~	~~0.09~~	~~0.004~~	~~0.02~~	~~22~~
Calibration Mixture 0.1	7	0.095	0.004	0.024	25
~~Inert andesite~~	~~7~~	~~0.18~~	~~0.005~~	~~0.10~~	~~56~~
Inert Andesite	7	0.176	0.005	0.095	54
~~Inert diorite~~	~~7~~	~~0.19~~	~~0.004~~	~~0.08~~	~~42~~
Inert Diorite	7	0.190	0.004	0.081	43
~~Pit rock~~	~~11~~	~~0.285~~	~~0.014~~	~~0.068~~	~~24~~
Pit Rock	11	0.285	0.014	0.068	24
~~Spiked andesite~~	~~6~~	~~0.34~~	~~0.02~~	~~0.06~~	~~18~~
Spiked Andesite	6	0.336	0.005	0.055	16
~~Vinini waste rock~~	~~11~~	~~0.761~~	~~0.019~~	~~0.269~~	~~35~~
Vinini Waste Rock	11	0.761	0.019	0.269	35
Refractory gold ore	11	1.50	0.052	0.326	22
~~Duluth waste rock~~	~~11~~	~~1.57~~	~~0.024~~	~~0.186~~	~~12~~
Duluth Waste Rock	11	1.57	0.024	0.186	12
~~Zinc plant tailings~~	~~11~~	~~3.79~~	~~0.072~~	~~0.423~~	~~11~~
Zinc Plant Tailings	11	3.79	0.072	0.423	11
~~Reclamation tailings~~	~~11~~	~~4.04~~	~~0.053~~	~~0.462~~	~~11~~
Reclamation Tailings	11	4.04	0.053	0.462	11
~~Autoclave feed ore~~	~~11~~	~~4.70~~	~~0.067~~	~~0.648~~	~~14~~
Autoclave Feed Ore	11	4.70	0.067	0.648	14

TABLE 4 Bias Information—Total Carbon

Test Material	Reference Carbon, %	Difference Carbon, %	Source	Description
Diorite gneiss	1.0 ± 0.1 Provisional	0.040	CANMET	SY-4 Diorite gneiss

TABLE 5 Bias Information—Total Sulfur

Test Material	Reference Sulfur, %	Difference Sulfur, %	Source	Description
Diorite gneiss	0.015 ± 0.004 Provisional	–0.001	CANMET	SY-4 Diorite gneiss
Pit rock	0.298 ± 0.015 Recommended	–0.013	CANMET	NBM-1 pit rock
Refractory gold ore	1.466 ± 0.044 Certified	0.034	NIST	SRM-886 refractory gold ore

enclosed in parentheses and below the null limit followed by an asterisk in accordance with Guide E 1950.

12.11.4 *Over-Range Results*—If the sulfur result exceeds 1.75 % for the minimum range instrument, discard the result and repeat the procedure from 12.1~~1~~0.2 with the diluted sample. Multiply the diluted test sample result by five and round to the nearest 0.1 %.

12.11.4.1 Alternatively, use a lower sample weight for the analysis as specified in 11.1.1.

12.12 *Precision and Bias*[8]:

12.12.1 *Precision*—Nine laboratories cooperated in testing this test method, providing seven sets of data for carbon and nine sets of data for sulfur, and obtained the precision data summarized in Tables 6-8.

12.12.2 *Bias*—No information on the bias of this test method is known because at the time of the interlaboratory study, suitable reference materials were not available. The user of this test method is encouraged to employ accepted reference materials, if available, to determine the presence or absence of bias.

NOTE 5—The user of this test method is cautioned that the method may not be quantitative for reporting above a reproducibility index (R) of 50 % relative, according to Practice E 1601. The user is advised to take this into account, in addition to the mineralogy of the sample, when interpreting the results for this test method.

HYDROCHLORIC ACID INSOLUBLE CARBON AND SULFUR

12.13 *Scope*—This test method covers the determination of hydrochloric acid insoluble carbon in the concentration range of 0.1 to 10 % and hydrochloric acid insoluble sulfur concentrations in the range of 0.1 to 8.8 %.

12.14 *Summary of Test Method*:

12.14.1 The test sample is partially decomposed with hydrochloric acid prior to instrumental analysis, where the carbon in the test sample is converted to carbon dioxide and the sulfur to sulfur dioxide by combustion in a stream of oxygen.

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

12.15 *Interferences*:

12.15.1 The elements normally present in ores and related materials do not interfere with this test method. Use of a halogen trap may be necessary for some commercially available instruments.

12.16 *Procedure*:

12.16.1 Ignite the crucibles or boats for test samples and standard samples in a muffle furnace for 1 h at 550° ± 10° C (see 12.4.1).

12.16.2 *Test Samples*—Transfer test samples, diluted test samples and standard addition samples using 0.200 ± 0.01 g into a 150–mL beaker and record the weight.

[8] Supporting data have been filed at ASTM Headquarters. Request RR: E01–1026.

TABLE 6 Residual Carbon From Pyrolysis

Test Material	Number of Laboratories	Carbon Found, %	~~SD, m~~Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
~~Ottawa sand~~	~~7~~	~~0.002~~	~~0.014~~	~~0.053~~	~~2449~~
Ottawa Sand	7	0.002	0.014	0.053	2449
~~Inert diorite~~	~~7~~	~~0.011~~	~~0.006~~	~~0.061~~	~~530~~
Inert Diorite	7	0.011	0.006	0.061	530
~~Autoclave feed ore~~	~~7~~	~~0.024~~	~~0.009~~	~~0.051~~	~~210~~
Autoclave Feed Ore	7	0.024	0.009	0.051	210
~~Inert andesite~~	~~7~~	~~0.030~~	~~0.009~~	~~0.061~~	~~204~~
Inert Andesite	7	0.030	0.009	0.061	204
~~Duluth waste rock~~	~~7~~	~~0.11~~	~~0.009~~	~~0.071~~	~~66~~
Duluth Waste Rock	7	0.107	0.009	0.071	66
~~Vinini waste rock~~	~~7~~	~~0.13~~	~~0.009~~	~~0.087~~	~~67~~
Vinini Waste Rock	7	0.131	0.009	0.087	67
~~Reclamation tailings~~	~~7~~	~~0.22~~	~~0.011~~	~~0.10~~	~~47~~
Reclamation Tailings	7	0.216	0.011	0.101	47
~~Pit rock~~	~~7~~	~~0.36~~	~~0.010~~	~~0.26~~	~~73~~
Pit Rock	7	0.359	0.010	0.261	73
~~Diorite gneiss~~	~~7~~	~~0.93~~	~~0.015~~	~~0.13~~	~~13~~
Diorite Gneiss	7	0.931	0.015	0.125	13
~~Refractory gold ore~~	~~7~~	~~4.84~~	~~0.076~~	~~0.75~~	~~16~~
Refractory Gold Ore	7	4.84	0.076	0.752	16
~~Zinc plant tailings~~	~~7~~	~~4.97~~	~~0.047~~	~~1.92~~	~~37~~
Zinc Plant Tailings	7	4.97	0.047	1.82	37

TABLE 7 Residual Sulfur From Pyrolysis

Test Material	Number of Laboratories	Sulfur Found, %	~~SD, m~~Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
~~Ottawa sand~~	~~9~~	~~0.014~~	~~0.009~~	~~0.029~~	~~204~~
Ottawa Sand	9	0.014	0.009	0.029	204
~~Diorite gneiss~~	~~9~~	~~0.11~~	~~0.038~~	~~0.16~~	~~153~~
Diorite Gneiss	9	0.107	0.038	0.164	153
~~Inert andesite~~	~~8~~	~~0.20~~	~~0.019~~	~~0.18~~	~~90~~
Inert Andesite	8	0.196	0.019	0.176	90
~~Pit rock~~	~~9~~	~~0.23~~	~~0.037~~	~~0.19~~	~~82~~
Pit Rock	9	0.229	0.037	0.187	82
~~Inert diorite~~	~~9~~	~~0.24~~	~~0.016~~	~~0.19~~	~~77~~
Inert Diorite	9	0.244	0.016	0.187	77
~~Autoclave feed ore~~	~~9~~	~~0.29~~	~~0.022~~	~~0.32~~	~~112~~
Autoclave Feed Ore	9	0.288	0.022	0.323	112
~~Vinini waste rock~~	~~9~~	~~0.43~~	~~0.015~~	~~0.16~~	~~38~~
Vinini Waste Rock	9	0.425	0.015	0.162	38
~~Refractory gold ore~~	~~9~~	~~0.71~~	~~0.032~~	~~0.24~~	~~34~~
Refractory Gold Ore	9	0.710	0.032	0.244	34
~~Duluth waste rock~~	~~9~~	~~0.71~~	~~0.056~~	~~0.27~~	~~38~~
Duluth Waste Rock	9	0.714	0.056	0.275	38
~~Zinc plant tailings~~	~~9~~	~~1.24~~	~~0.042~~	~~1.45~~	~~117~~
Zinc Plant Tailings	9	1.24	0.042	1.45	117
~~Reclamation tailings~~	~~9~~	~~1.54~~	~~0.025~~	~~0.44~~	~~28~~
Reclamation Tailings	9	1.54	0.025	0.435	28

12.16.3 *Decomposition*—Add 25 mL of hydrochloric acid (1 + 4) to the beaker and let stand at room temperature for 30 min. Cover with a watch glass and place the beaker on a hot plate and gently boil for 10 min. Cool.

12.16.4 *Filtration*—Filter through a glass filter, wash with water at least three times and discard filtrate.

12.16.5 Transfer filter and solids to the crucible or boat used for instrumental analysis. Use of a different sample weight may be required on some instruments for some samples (see 11.1.1).

12.16.6 *Duplicate Test Sample*—Analyze a duplicate test sample within each group of fifty test samples. If the difference of the duplicate results exceeds the limits shown in Table 1, for a material of comparable concentration, discard the results since the last acceptable quality control sample result had been obtained, correct any sample preparation or instrumental problems and repeat the analyses from 12.16.2.

12.16.7 *Analysis*:

12.16.7.1 Analyze quality control samples before each batch of test samples and within each group of ten test samples as directed in 11.4. Measure the carbon and sulfur concentrations for quality control samples, test samples and diluted test samples in percent according to the instrument manufacturer's instructions and record the measurements.

12.16.7.2 Continue analysis until the batch of test samples is completed, a quality control sample or duplicate test sample result deviates more than the limits shown in Table 1, for a material of comparable concentration.

12.17 *Calculation*:

12.17.1 Calculate the hydrochloric acid insoluble carbon and sulfur concentrations for the test samples according to the manufacturer's instructions.

12.17.2 Calculate the hydrochloric acid loss, %. *D*, as follows:

$$D = E - F \tag{2}$$

where:

E = total carbon result, %, and

F = hydrochloric acid insoluble carbon result, %.

12.17.3 Round the results to the nearest 0.01 % and record as hydrochloric acid insoluble carbon and sulfur, or hydrochloric acid loss carbon, at or above the lower scope limit established during interlaboratory testing. Enclose results below the lower scope limits in parentheses and below the null limit followed by an asterisk, in accordance with Guide E 1950.

12.17.4 *Over-Range Results*—If the sulfur result exceeds 1.75 % for the minimum range instrument, discard the result and

TABLE 8 Pyrolysis Loss Sulfur

Test Material	Number of Laboratories	Sulfur Loss, %	~~SD, m~~Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
~~Diorite gneiss~~	~~9~~	~~– 0.11~~	~~0.038~~	~~0.20~~	~~186~~
Diorite Gneiss	9	- 0.106	0.038	0.197	- 186
~~Inert diorite~~	~~9~~	~~– 0.06~~	~~0.015~~	~~0.14~~	~~224~~
Inert Diorite	9	- 0.063	0.015	0.143	- 224
~~Inert andesite~~	~~8~~	~~– 0.04~~	~~0.018~~	~~0.16~~	~~406~~
Inert Andesite	8	- 0.041	0.018	0.165	- 406
~~Ottawa sand~~	~~9~~	~~– 0.02~~	~~0.009~~	~~0.07~~	~~420~~
Ottawa Sand	9	- 0.017	0.009	0.070	- 420
~~Pit rock~~	~~9~~	~~0.04~~	~~0.035~~	~~0.23~~	~~536~~
Pit Rock	9	0.042	0.035	0.225	536
~~Vinini waste rock~~	~~9~~	~~0.32~~	~~0.024~~	~~0.25~~	~~77~~
Vinini Waste Rock	9	0.322	0.024	0.248	77
~~Refractory gold ore~~	~~9~~	~~0.76~~	~~0.059~~	~~0.37~~	~~49~~
Refractory Gold Ore	9	0.763	0.059	0.373	49
~~Duluth waste rock~~	~~9~~	~~0.86~~	~~0.058~~	~~0.38~~	~~44~~
Duluth Waste Rock	9	0.863	0.058	0.384	44
~~Reclamation tailings~~	~~9~~	~~2.50~~	~~0.062~~	~~0.60~~	~~24~~
Reclamation Tailings	9	2.50	0.062	0.599	24
~~Zinc plant tailings~~	~~9~~	~~2.53~~	~~0.082~~	~~1.21~~	~~48~~
Zinc Plant Tailings	9	2.53	0.082	1.21	48
~~Autoclave feed ore~~	~~9~~	~~4.42~~	~~0.076~~	~~0.70~~	~~16~~
Autoclave Feed Ore	9	4.42	0.076	0.696	16

repeat the procedure from 12.16.2 with the diluted sample. Multiply the diluted test sample result by five and round to the nearest 0.1 %.

12.17.4.1 Alternatively, use a lower sample weight for the analysis as specified in 11.1.1.

12.18 *Precision and Bias*

12.18.1 *Precision*—Eight laboratories cooperated in testing this test method, providing eight sets of data for carbon and eight sets of data for sulfur, and obtained the precision data summarized in Table 9, Table 10, and Table 11.

12.18.2 *Bias*—No information on the bias of this test method is known because at the time of the interlaboratory study, suitable reference materials were not available. The user of this test method is encouraged to employ accepted reference materials, if

TABLE 9 Statistical Information Hydrochloric Acid Insoluble Carbon

Test Material	Number of Laboratories	Carbon Found, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Ottawa Sand (D)	8	0.025	0.010	0.053	209
Pit Rock (G)	8	0.054	0.009	0.092	169
Inert Diorite (K)	8	0.056	0.009	0.095	169
Reclamation Tailings (C)	8	0.068	0.011	0.067	99
Autoclave Feed Ore (A)	8	0.078	0.009	0.060	77
Zinc Plant Tails (H)	8	0.082	0.010	0.186	229
Diorite Gneiss (F)	8	0.122	0.013	0.103	85
Duluth Waste Rock (B)	8	0.133	0.014	0.094	70
Vinini Waste Rock (E)	8	0.222	0.021	0.131	59
Refractory Gold Ore (I)	8	0.470	0.009	0.389	83

TABLE 10 Statistical Information Hydrochloric Acid Insoluble Sulfur

Test Material	Number of Laboratories	Sulfur Found, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Ottawa Sand (D)	8	0.012	0.004	0.044	358
Diorite Gneiss (F)	8	0.021	0.003	0.064	308
Inert Diorite (K)	8	0.164	0.008	0.080	49
Pit Rock (G)	8	0.252	0.039	0.136	54
Vinini Waste Rock (E)	8	0.653	0.033	0.392	60
Duluth Waste Rock (B)	8	0.863	0.089	0.709	82
Refractory Gold Ore (I)	8	1.22	0.067	1.21	99
Reclamation Tails (C)	8	2.96	0.166	1.70	58
Zinc Plant Tails (H)	8	3.12	0.185	4.28	137
Autoclave Feed Ore (A)	8	4.20	0.114	0.994	24

TABLE 11 Statistical Information Hydrochloric Acid Loss Carbon

Test Material	Number of Laboratories	Carbon Loss, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Ottawa Sand (D)	7	-0.009	0.010	0.047	- 536
Duluth Waste Rock (B)	7	0.021	0.015	0.100	478
Autoclave Feed Ore (A)	7	0.023	0.009	0.094	412
Reclamation Tails (C)	7	0.413	0.014	0.103	25
Vinini Waste Rock (E)	7	0.573	0.020	0.128	22
Pit Rock (G)	7	0.740	0.014	0.128	17
Diorite Gneiss (F)	7	0.933	0.016	0.142	15
Refractory Gold Ore (I)	7	5.30	0.042	0.335	6
Zinc Plant Tails (H)	7	5.78	0.046	0.406	7

available, to determine the presence or absence of bias.

NOTE 6—The user of this test method is cautioned that the method may not be quantitative for reporting above a reproducibility index (R) of 50 % relative, in accordance with Practice E 1601. The user is advised to take this into account, in addition to the mineralogy of the sample, when interpreting the results for this test method.

13. Keywords

13.1 carbon content; ores; related materials; sulfur content