Designation: E 1915 – 05

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	0.08 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 %.
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 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, Total	12.1-12.6
Carbon and Sulfur, Residual from Pyrolysis	12.7-12.12
Carbon and Sulfur, Hydrochloric Acid Insoluble	12.13-12.18
Sulfur, Nitric Acid Insoluble	12.19-12.24
Sulfur, Sodium Carbonate Insoluble	12.25-12.30

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:* [2]

D 1193 Specification for Reagent Water
D 5744 Test Method for Accelerated Weathering of Solid Materials Using a Modified Humidity Cell
D 6234 Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure
E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E 50 Practices for Apparatus, Reagents and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel and Cobalt Alloys
E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
E 1950 Practice for Reporting Results from Methods of Chemical Analysis
E 2242 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 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

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[1] 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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[2] 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.

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.[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.

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*:

6.3.1 *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.

6.3.2 *Blank Reference Sample*—Prepare a blank reference sample by pulverizing or grinding 100 g silica (see 6.3.6), pass through a No. 100 (150-μm) sieve, and mixing and storing in a 250-mL glass bottle. This blank contains 0.00 % carbon and sulfur.

6.3.3 *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.

6.3.4 *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.

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

6.3.5 *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.

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

6.3.5.2 Commercially–produced calibration mixtures, which meet these specifications, may also be used.

6.3.6 *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.

6.3.7 *Sodium Carbonate Solution (10 %)*—Transfer 100 g of sodium carbonate (purity: 99.0 % minimum) to 500-mL of water in a 1-L volumetric flask. Dilute to the mark and mix.

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 Practices 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

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[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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 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 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 in accordance with 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

TABLE 1 Calibration Mixture 95 % Confidence Limits from Interlaboratory Testing

Mixture	Min., % C	Max., % C	Min., % S	Max., % S
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
$CaCO_3$	10.9	12.8	. . .	. . .

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 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 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*:

TABLE 2 Statistical Information — Total Carbon

Test Material	Number of Laboratories	Carbon Found, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Blank	7	0.012	0.004	0.034	300
Ottawa Sand (D)	10	0.021	0.011	0.0477	230
Inert Diorite (K)	7	0.050	0.005	0.037	74
Inert Andesite (J)	7	0.090	0.004	0.054	59
Autoclave Feed Ore (A)	10	0.086	0.016	0.115	133
Calibration Mixture 0.1	7	0.117	0.007	0.049	42
Duluth Waste Rock (B)	10	0.142	0.017	0.112	79
Spiked Andesite	6	0.292	0.008	0.051	17
Reclamation Tailings (C)	10	0.462	0.025	0.223	48
Vinini Waste Rock (E)	10	0.771	0.024	0.180	23
Pit Rock (G)	10	0.800	0.025	0.117	15
Diorite Gneiss (F)	10	1.04	0.032	0.170	16
Zinc Plant Tailings (H)	10	5.87	0.055	0.494	8
Refractory Gold Ore (I)	10	5.70	0.038	0.478	8

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 in accordance with 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 in accordance with 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 Practice 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*[4]

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.

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[4] Supporting data have been filed at ASTM International Headquarters. Request RR: E01-1023 and RR: E01–1040.

TABLE 3 Statistical Information — Total Sulfur

Test Material	Number of Laboratories	Sulfur Found, %	Min., SD(S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Blank	7	0.0002	0.002	0.010	5000
Ottawa Sand (D)	11	0.004	0.003	0.0133	312
Diorite Gneiss (F)	11	0.014	0.007	0.039	283
Calibration Mixture 0.1	7	0.095	0.004	0.024	25
Inert Andesite (J)	7	0.176	0.005	0.095	54
Inert Diorite (K)	7	0.190	0.004	0.081	43
Pit Rock (G)	11	0.285	0.014	0.068	24
Spiked Andesite	6	0.336	0.005	0.055	16
Vinini Waste Rock (E)	11	0.761	0.019	0.269	35
Refractory Gold Ore (I)	11	1.50	0.052	0.326	22
Duluth Waste Rock (B)	11	1.57	0.024	0.186	12
Zinc Plant Tailings (H)	11	3.79	0.072	0.423	11
Reclamation Tailings (C)	11	4.04	0.053	0.462	11
Autoclave Feed Ore (A)	11	4.70	0.067	0.648	14

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, 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.

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.

TABLE 4 Bias Information—Total Carbon

Test Material	Reference Carbon, %	Difference Carbon, %	Source	Description
Diorite Gneiss (F)	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 (F)	0.015 ± 0.004 Provisional	−0.001	CANMET	SY-4 Diorite gneiss
Pit Rock (G)	0.298 ± 0.015 Recommended	−0.013	CANMET	NBM-1 pit rock
Refractory Gold Ore (I)	1.466 ± 0.044 Certified	0.034	NIST	SRM-886 refractory gold ore

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 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.

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 in accordance with 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 in accordance with the manufacturer's instructions.

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

$$A = B - C \tag{1}$$

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

TABLE 6 Residual Carbon From Pyrolysis

Test Material	Number of Laboratories	Carbon Found, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Ottawa Sand (D)	7	0.002	0.014	0.053	2449
Inert Diorite (K)	7	0.011	0.006	0.061	530
Autoclave Feed Ore (A)	7	0.024	0.009	0.051	210
Inert Andesite (J)	7	0.030	0.009	0.061	204
Duluth Waste Rock (B)	7	0.107	0.009	0.071	66
Vinini Waste Rock (E)	7	0.131	0.009	0.087	67
Reclamation Tailings (C)	7	0.216	0.011	0.101	47
Pit Rock (G)	7	0.359	0.010	0.261	73
Diorite Gneiss (F)	7	0.931	0.015	0.125	13
Refractory Gold Ore (I)	7	4.84	0.076	0.752	16
Zinc Plant Tailings (H)	7	4.97	0.047	1.82	37

TABLE 7 Residual Sulfur From Pyrolysis

Test Material	Number of Laboratories	Sulfur Found, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Ottawa Sand (D)	9	0.014	0.009	0.029	204
Diorite Gneiss (F)	9	0.107	0.038	0.164	153
Inert Andesite (J)	8	0.196	0.019	0.176	90
Pit Rock (G)	9	0.229	0.037	0.187	82
Inert Diorite (K)	9	0.244	0.016	0.187	77
Autoclave Feed Ore (A)	9	0.288	0.022	0.323	112
Vinini Waste Rock (E)	9	0.425	0.015	0.162	38
Refractory Gold Ore (I)	9	0.710	0.032	0.244	34
Duluth Waste Rock (B)	9	0.714	0.056	0.275	38
Zinc Plant Tailings (H)	9	1.24	0.042	1.45	117
Reclamation Tailings (C)	9	1.54	0.025	0.435	28

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 12.10.2 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*[5]

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.

TABLE 8 Pyrolysis Loss Sulfur

Test Material	Number of Laboratories	Sulfur Loss, %	Min., SD (S_M, E 1601)	Reproducibility Index (R, E 1601)	R_{rel}, %
Diorite Gneiss (F)	9	- 0.106	0.038	0.197	- 186
Inert Diorite (K)	9	- 0.063	0.015	0.143	- 224
Inert Andesite (J)	8	- 0.041	0.018	0.165	- 406
Ottawa Sand (D)	9	- 0.017	0.009	0.070	- 420
Pit Rock (G)	9	0.042	0.035	0.225	536
Vinini Waste Rock (E)	9	0.322	0.024	0.248	77
Refractory Gold Ore (I)	9	0.763	0.059	0.373	49
Duluth Waste Rock (B)	9	0.863	0.058	0.384	44
Reclamation Tailings (C)	9	2.50	0.062	0.599	24
Zinc Plant Tailings (H)	9	2.53	0.082	1.21	48
Autoclave Feed Ore (A)	9	4.42	0.076	0.696	16

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, 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.

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.

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