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StandardTest Methods for
Analysis of Metal Bearing Ores and Related Materials for Carbon, Sulfur, and Acid-Base Characteristics[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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 %
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
Sulfur, Nitric Acid Insoluble	10.37-10.45
Sulfur, Sodium Carbonate Insoluble	10.46-10.54

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:*[2]

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

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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. 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. These test methods are also used to speciate 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)). Additionally, the 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, $FeCO_3$, and rhodochrosite, $MnCO_3$) decompose, yielding carbon dioxide, CO_2, 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).[3])

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.[4] 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 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.

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 weight in order to minimize sulfide mineral oxidation. 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.

[3] 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.

[4] *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.

9.3 *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 9.3.1.

9.3.1 Weigh 10.0 g ± 0.1 g prepared sample and combine with 40.0 g ± 0.1 g dry silica (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.

10. Procedures

TOTAL CARBON AND SULFUR

10.1 *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 %.

10.2 *Summary of Test Method:*

10.2.1 The carbon in the test sample is converted to CO_2 and the sulfur to sulfur dioxide by combustion in a stream of oxygen.

10.2.2 The amount of CO_2 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 and in the sections Apparatus for Determination of Total Carbon by Direct Combustion and Apparatus for the Determination of Sulfur by Direct Combustion of Practices E50, 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 ($BaSO_4$), 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.

10.4.1.2 *Blank Reference Sample*—Prepare a blank reference sample by pulverizing or grinding 100 g of SiO_2 (see 10.4.1.6), to pass through a No. 100 (150-μm) 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 ($CaCO_3$), 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.

10.4.1.4 *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 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_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.

(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_2)* (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_2WO_4)* (purity 99 % minimum).

10.4.1.8 *Vanadium Pentoxide (V_2O_5)* (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_2O_5 or H_2WO_4 for the determination of sulfur in this test method. Use a 0.200 g ± 0.1 g weight 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 weights for certain concentration 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 ± 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, $BaSO_4$, and $CaCO_3$ 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 from 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 0.2 % sulfur and a correlation coefficient of at least 0.99. Correct any problems with the instrument before proceeding with the analysis of test samples.

(1) Linearity may also be verified by the use of $BaSO_4$ and $CaCO_3$ weights equivalent to the content of the calibration mixtures.

TABLE 1 Calibration Mixture 95 % Confidence Limits from Interlaboratory Testing

Mixture	Min, % Carbon	Max., % Carbon	Min, % Sulfur	Max., % Sulfur
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	. . .	. . .

10.5.3.2 *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.

10.5.3.3 *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.

10.5.4 *Method Quality Control:*

10.5.4.1 *Calibration Verification*—Analyze a calibration mixture with a concentration greater than or equal to 0.5 % carbon and 0.5 % 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.

10.5.4.2 *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.

10.5.4.3 *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.

10.5.4.4 *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.

10.5.4.5 *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.

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

10.7 *Procedure:*

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

10.7.2 *Test Samples*—Transfer test samples, diluted test samples and standardization samples using 0.200 g ± 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 10.5.1.1).

10.7.3 *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 10.7.2.

10.7.4 *Analysis:*

10.7.4.1 Analyze quality control samples before each batch of test samples and within each group of ten test samples as directed in 10.5.4. 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.

10.7.4.2 Continue analysis until the batch of test samples is completed, or until a quality control sample or duplicate test sample result deviates more than the limits shown in Table 1, for a material of comparable concentration. If the difference of the 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 10.7.4.2.

10.8 *Calculation:*

10.8.1 Calculate the total carbon and sulfur concentrations for the test samples in accordance with the manufacturer's instructions.

10.8.2 Round the results above 0.1 % to the nearest 0.01 % and record as total carbon or sulfur. Enclose results from

TABLE 2 Statistical Information—Total Carbon

Test Material	Number of Laboratories	Carbon Found, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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 Andes- ite	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 Tail- ings (H)	10	5.87	0.055	0.494	8
Refractory Gold Ore (I)	10	5.70	0.038	0.478	8

TABLE 3 Statistical Information—Total Sulfur

Test Material	Number of Laboratories	Sulfur Found, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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 Andes- ite	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 Tail- ings (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

0.03 % to 0.1 % in parentheses and below 0.03 % in parentheses followed by an asterisk in accordance with Practice E1950.

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

10.8.3.1 Alternatively, use a lower sample weight for the analysis as specified in 10.5.1.1.

10.9 *Precision and Bias*:[5,6]

10.9.1 *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.

10.9.2 *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 3—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 E1601. 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

10.10 *Scope*—This test method covers the determination of residual carbon from pyrolysis in the concentration range

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

between 0.1 % and 10 % and residual sulfur from pyrolysis concentrations in the range between 0.1 % and 8.8 %.

10.11 *Summary of Test Method:*

10.11.1 The test sample is ignited in a muffle furnace prior to instrumental analysis where the carbon in the test sample is converted to CO_2 and the sulfur to sulfur dioxide by combustion in a stream of oxygen.

10.11.2 The amount of CO_2 and sulfur dioxide are measured by infrared absorption.

10.12 *Apparatus:*

10.12.1 *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

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[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1023.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1040.

materials. Instruments, such as those shown in Test Methods E1019 and in the sections Apparatus for Determination of Total Carbon by Direct Combustion and Apparatus for the Determination of Sulfur by Direct Combustion of Practices E50, that can be shown to give equivalent results may also be used for these test methods.

10.13 *Reagents and Materials:*

10.13.1 *Reagents:*

10.13.1.1 *Barium Sulfate* (see 10.4.1.1).

10.13.1.2 *Blank Reference Sample* (see 10.4.1.2).

10.13.1.3 *Calcium Carbonate* (see 10.4.1.3).

10.13.1.4 *Calibration Mixture A* (see 10.4.1.4).

10.13.1.5 *Calibration Mixtures* (see 10.4.1.5).

10.13.1.6 *Silica* (see 10.4.1.6).

10.13.1.7 *Tungstic Acid* (see 10.4.1.7).

10.13.1.8 *Vanadium Pentoxide* (see 10.4.1.8).

10.13.2 *Materials:*

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

10.14 *Calibration and Standardization:*

10.14.1 *Apparatus:* Operate and calibrate the instrument in accordance with the manufacturer's instructions. Resistance furnace instruments require the use of vanadium pentoxide or H_2WO_4 for the determination of sulfur in this test method. Use a 0.200 g $\pm$ 0.1 g weight for all calibration mixtures, reference materials, blank reference materials, test samples, and diluted test samples in this test method.

10.14.1.1 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.

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

10.14.3 *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. Carry out the performance demonstration as described in 10.5.3.

10.14.4 *Method Quality Control:*

10.14.4.1 *Calibration Verification*—Analyze a calibration mixture with a concentration greater than or equal to 0.5 % carbon and 0.5 % 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.

10.14.4.2 *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.

10.14.4.3 *Reference Sample*—Analyze a reference sample, certified for total carbon and total sulfur before analysis of test samples for and within each group of fifty test samples and a reference sample certified for pyrolysis residual carbon or sulfur from pyrolysis, if available. 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.

10.14.4.4 *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.

10.14.4.5 *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 4—Add the 0.5 % calibration mixture after the pyrolysis procedure but before the analysis step.

10.15 *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.

NOTE 5—$SO_2(g)$ can be adsorbed by carbonate minerals within a sample and from other samples in a batch, resulting in low pyrolysis loss estimates for sulfide. Pyrolysis pretreatment at 550 °C has a potential to thermally decompose some carbonate minerals:
(*1*) Transition metal carbonates (for example, $FeCO_3$ and $MnCO_3$) decompose, yielding CO_2, 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.

10.16 *Procedure:*

10.16.1 Ignite the crucibles or boats for test samples and standardization samples in a muffle furnace for 1 h at 550 °C $\pm$ 10 °C (see 10.7.1).

10.16.2 *Test Samples*—Transfer test samples, diluted test samples, and standardization samples using 0.200 g $\pm$ 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 10.14.1.1).

10.16.3 *Ignition*—Ignite the crucibles or boats containing the test samples, blank, reference samples for pyrolysis residual carbon and sulfur from pyrolysis and standard addition samples in a muffle furnace for 1 h at 550 °C ± 10 °C. Add the calibration mixture portion for the standard addition sample after pyrolysis and cooling, then mix.

10.16.4 *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 10.16.2.

10.16.5 *Analysis:*

10.16.5.1 Analyze quality control samples before each batch of test samples and within each group of ten test samples as directed in 10.14.4. 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.

10.16.5.2 Continue analysis until the batch of test samples is completed, or until a quality control sample or duplicate test sample result deviates more than the limits shown in Table 1 for a material of comparable concentration. If the difference of the 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 10.16.2.

10.17 *Calculation:*

10.17.1 Calculate the residual carbon and sulfur from pyrolysis concentrations for the test samples in accordance with the manufacturer's instructions.

10.17.2 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, %.

10.17.3 Round the results to the nearest 0.01 % and record as residual carbon from pyrolysis, residual sulfur from pyrolysis, 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 E1950.

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

10.17.4.1 Alternatively, use a lower sample weight for the analysis as specified in 10.14.1.1.

TABLE 6 Residual Carbon From Pyrolysis

Test Material	Number of Laboratories	Carbon Found, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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

10.18 *Precision and Bias:*[7]

10.18.1 *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.

10.18.2 *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 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 E1601. 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

10.19 *Scope*—This test method covers the determination of HCl 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 %.

10.20 *Summary of Test Method:*

10.20.1 The test sample is partially decomposed with HCl prior to instrumental analysis, where the carbon in the test sample is converted to CO_2 and the sulfur to sulfur dioxide by combustion in a stream of oxygen.

10.20.2 The amount of CO_2 and sulfur dioxide are measured by infrared absorption.

10.21 *Apparatus:*

10.21.1 *Combustion-Infrared Spectrophotometer*, equipped with a combustion chamber, oxygen carrier stream, and infrared absorption detector, suitable for analysis of sulfur in a

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[7] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1026.

TABLE 7 Residual Sulfur From Pyrolysis

Test Material	Number of Laboratories	Sulfur Found, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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

TABLE 8 Pyrolysis Loss Sulfur

Test Material	Number of Laboratories	Sulfur Loss, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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

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 and in the sections Apparatus for Determination of Total Carbon by Direct Combustion and Apparatus for the Determination of Sulfur by Direct Combustion of Practices E50, that can be shown to give equivalent results may also be used for these test methods.

10.22 *Reagents and Materials:*

10.22.1 *Reagents:*

10.22.1.1 *Barium Sulfate* (see 10.4.1.1).

10.22.1.2 *Blank Reference Sample* (see 10.4.1.2).

10.22.1.3 *Calcium Carbonate* (see 10.4.1.3).

10.22.1.4 *Calibration Mixture A* (see 10.4.1.4).

10.22.1.5 *Calibration Mixtures* (see 10.4.1.5).

10.22.1.6 *Silica* (see 10.4.1.6).

10.22.1.7 *Tungstic Acid* (see 10.4.1.7).

10.22.1.8 *Vanadium Pentoxide* (see 10.4.1.8).

10.22.2 *Materials:*

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

10.22.2.2 *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.

10.23 *Calibration and Standardization:*

10.23.1 *Apparatus*—Operate and calibrate the instrument in accordance with the manufacturer's instructions. Resistance furnace instruments require the use of V_2O_5 or H_2WO_4 for the determination of sulfur in this test method. Use a 0.200 g $\pm$ 0.1 g weight for all calibration mixtures, reference materials, blank reference materials, test samples and diluted test samples in this test method.

10.23.1.1 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.

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

10.23.3 *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. Carry out the performance demonstration as described in 10.5.3.

10.23.4 *Method Quality Control:*

10.23.4.1 *Calibration Verification*—Analyze a calibration mixture with a concentration greater than or equal to 0.5 % carbon and 0.5 % 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.

10.23.4.2 *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.

10.23.4.3 *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 and a reference sample certified for hydrochloric acid insoluble carbon or sulfur, if available. 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.

10.23.4.4 *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.

10.23.4.5 *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 7—Add the 0.5 % calibration mixture after the hydrochloric acid decomposition procedure but before the analysis step.

10.24 *Interferences:*

10.24.1 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.

10.25 *Procedure:*

10.25.1 Ignite the crucibles or boats for test samples and standardization samples in a muffle furnace for 1 h at 550 °C ± 10 °C (see 10.7.1).

10.25.2 *Test Samples*—Transfer test samples, diluted test samples, blank samples, reference samples for HCl insoluble carbon or sulfur and standard addition samples using 0.200 g ± 0.01 g into a 150-mL beaker and record the weight.

10.25.3 *Decomposition*—Add 25 mL of HCl (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.

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

10.25.5 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 10.23.1.1). Add the calibration mixture portion for the standard addition sample after digestion, and mix.

10.25.6 *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 10.25.2.

10.25.7 *Analysis:*

10.25.7.1 Analyze quality control samples before each batch of test samples and within each group of ten test samples as directed in 10.23.4. 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.

10.25.7.2 Continue analysis until the batch of test samples is completed, or a quality control sample or duplicate test sample result deviates more than the limits shown in Table 1, for a material of comparable concentration. If the difference of the 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 10.25.2.

10.26 *Calculation:*

10.26.1 Calculate the HCl insoluble carbon and sulfur concentrations for the test samples in accordance with the manufacturer's instructions.

10.26.2 Calculate the HCl loss carbon, % D, as follows:

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

where:
E = total carbon result, %, and
F = hydrochloric acid insoluble carbon result, %.

10.26.3 Round the results to the nearest 0.01 % and record as HCl 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 Practice E1950.

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

10.26.4.1 Alternatively, use a lower sample weight for the analysis as specified in 10.23.1.1.

10.27 *Precision and Bias:*

10.27.1 *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.

10.27.2 *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 8—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 E1601. 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.

TABLE 9 Statistical Information Hydrochloric Acid Insoluble Carbon

Test Material	Number of Laboratories	Carbon Found, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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 11 Statistical Information Hydrochloric Acid Loss Carbon

Test Material	Number of Laboratories	Carbon Loss, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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

TABLE 10 Statistical Information Hydrochloric Acid Insoluble Sulfur

Test Material	Number of Laboratories	Sulfur Found, %	Min, SD (S_M, Practice E1601)	Reproducibility Index (R, Practice E1601)	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

ACID NEUTRALIZATION POTENTIAL POTENTIAL ACIDITY TITRATION

10.28 *Scope:*

10.28.1 This test method covers the determination of acid neutralization potential with acidity correction in metal-bearing ores and related materials such as tailings and waste rock within the range of -1.0 % to 100 % expressed as $CaCO_3$.

10.29 *Summary of Test Method:*

10.29.1 A finely ground sample is reacted with a known amount of H_2SO_4, filtered, treated with hydrogen peroxide (H_2O_2) to oxidize the metals present in filtrate, boiled to expel the excess peroxide, and the potential of the sample to neutralize acid is determined by automatic or manual titration of the acid remaining in the filtrate, using standard sodium hydroxide (NaOH).

10.30 *Apparatus:*

10.30.1 *Automatic Titrator*—Capable of determination of the equivalence point for a strong acid neutralization titration within the parameters measured during the interlaboratory testing of this method.

10.31 *Reagents and Materials:*

10.31.1 *Blank Reference Sample* (see 10.4.1.2).

10.31.2 *Calcium Carbonate* (see 10.4.1.3).

10.31.3 *Calcium Carbonate Calibration Mixtures*—Transfer 5.00 g and 50.00 g of Calibration Mixture B to ring and puck grinding mills or equivalent devices. Add the amount of dried SiO_2 needed to bring the total weight to 100.0 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: 1.00 % and 10.0 % $CaCO_3$.

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

10.31.3.2 Commercially-produced calibration mixtures, which meet these specifications, may also be used.

10.31.4 *Calibration Mixture B—(1 g = 200 mg CaCO₃)*—Combine 20.0 g $CaCO_3$ and 80.0 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 20.0 % $CaCO_3$.

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

10.31.4.2 Commercially-produced calibration mixtures, which meet these specifications, may also be used.

10.31.5 *Calibration Mixture C—(1 g = -100 mg CaCO₃)*—Combine 18.37 g $FeSO_4·7H_2O$ and 81.63 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 an equivalent of -10 % $CaCO_3$.

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

10.31.5.2 Commercially-produced calibration mixtures, which meet these specifications, may also be used.