Designation: E1019 – 18

Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Inert Gas Fusion Techniques[1]

This standard is issued under the fixed designation E1019; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the determination of carbon, sulfur, nitrogen, and oxygen, in steel, iron, nickel, and cobalt alloys having chemical compositions within the following limits:

Element	Mass Fraction Range, %
Aluminum	0.001 to 18.00
Antimony	0.002 to 0.03
Arsenic	0.0005 to 0.10
Beryllium	0.001 to 0.05
Bismuth	0.001 to 0.50
Boron	0.0005 to 1.00
Cadmium	0.001 to 0.005
Calcium	0.001 to 0.05
Carbon	0.001 to 4.50
Cerium	0.005 to 0.05
Chromium	0.005 to 35.00
Cobalt	0.01 to 75.0
Niobium	0.002 to 6.00
Copper	0.005 to 10.00
Hydrogen	0.0001 to 0.0030
Iron	0.01 to 100.0
Lead	0.001 to 0.50
Magnesium	0.001 to 0.05
Manganese	0.01 to 20.0
Molybdenum	0.002 to 30.00
Nickel	0.005 to 84.00
Nitrogen	0.0005 to 0.50
Oxygen	0.0005 to 0.03
Phosphorus	0.001 to 0.90
Selenium	0.001 to 0.50
Silicon	0.001 to 6.00
Sulfur	0.002 to 0.35
Tantalum	0.001 to 10.00
Tellurium	0.001 to 0.35
Tin	0.002 to 0.35
Titanium	0.002 to 5.00
Tungsten	0.005 to 21.00
Vanadium	0.005 to 5.50
Zinc	0.005 to 0.20
Zirconium	0.005 to 2.500

1.2 The test methods appear in the following order:

	Sections
Carbon, Total, by the Combustion and Infrared Absorption or Thermal Conductivity Detection Test Method	10 – 20
Nitrogen by the Inert Gas Fusion and Thermal Conductivity Detection Test Method	32 – 42
Oxygen by the Inert Gas Fusion and Infrared Absorption or Thermal Conductivity Detection Test Method	43 – 54
Sulfur by the Combustion-Infrared Absorption Detection Test Method	55 – 65
Sulfur by the Combustion–Infrared Absorption Test Method (Potassium Sulfate Calibration) – *Discontinued 2018*	21 – 31

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 6.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

[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 E01.01 on Iron, Steel, and Ferroalloys.

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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.

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
E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

3. Terminology

3.1 For definition of terms used in this test method, refer to Terminology E135.

4. Significance and Use

4.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use 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.

5. Apparatus and Reagents

5.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure.

5.2 These methods were originally developed for older technology manual instrumentation with the flow schematics indicated. Current commercially available instruments are more automated and may have slightly different flow schematics and should be capable of producing data meeting or exceeding the precision and bias requirements.

6. Hazards

6.1 For hazards to be observed in the use of certain reagents in this test method, refer to Practices E50.

6.2 Use care when handling hot crucibles and operating furnaces to avoid personal injury by either burn or electrical shock.

7. Sampling

7.1 For procedures to sample the materials, refer to those parts of Practice E1806.

8. Rounding Calculated Values

8.1 Rounding of test results obtained using these test methods shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E173. The Reproducibility R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

TOTAL CARBON BY THE COMBUSTION AND INFRARED ABSORPTION OR THERMAL CONDUCTIVITY DETECTION TEST METHOD

10. Scope

10.1 This test method covers the determination of carbon from 0.005 % to 4.5 %.

11. Summary of Test Method

11.1 The carbon is converted to carbon dioxide (CO_2) by combustion in a stream of oxygen.

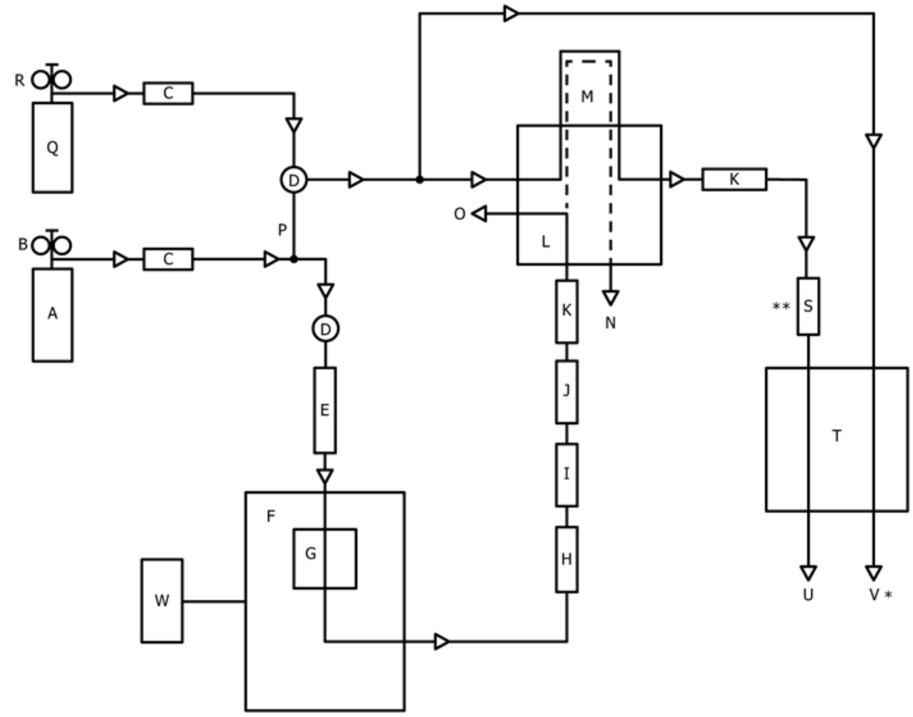
11.1.1 *Thermal Conductivity Test Method*—The CO_2 is absorbed on a suitable grade of zeolite, released by heating the zeolite, and swept by helium or oxygen into a chromatographic column. Upon elution, the amount of CO_2 is measured in a thermistor-type conductivity cell. Refer to Fig. 1 for example.

11.1.2 *Infrared (IR) Absorption, Test Method A*—The amount of CO_2 is measured by infrared (IR) absorption. CO_2 absorbs IR energy at a precise wavelength within the IR spectrum. Energy of this wavelength is absorbed as the gas passes through a cell body in which the IR energy is transmitted. All other IR energy is eliminated from reaching the detector by a precise wavelength filter. Thus, the absorption of IR energy can be attributed to only CO_2 and its amount is measured as changes in energy at the detector. One cell is used as both a reference and a measure chamber. Total carbon, as CO_2, is measured over a period of time. Refer to Fig. 2 for example.

11.1.3 *Infrared (IR) Absorption, Test Method B*—The detector consists of an IR energy source, a separate measure chamber and reference chamber, and a diaphragm acting as one plate of a parallel plate capacitor. During specimen combustion, the flow of CO_2 with its oxygen carrier gas is routed through the measure chamber while oxygen alone passes through the reference chamber. Energy from the IR source passes through both chambers, simultaneously arriving at the diaphragm (capacitor plate). Part of the IR energy is absorbed by the CO_2 present in the measure chamber while none is absorbed passing through the reference chamber. This creates an IR energy imbalance reaching the diaphragm, thus distorting it. This distortion alters the capacitance creating an electric signal change that is amplified for measurement as CO_2. Total carbon, as CO_2, is measured over a period of time. Refer to Fig. 3 for example.

11.1.4 *Infrared (IR) Absorption, Test Method C, Closed Loop*—The combustion is performed in a closed loop, where carbon monoxide (CO) and CO_2 are detected in the same infrared cell. Each gas is measured with a solid state energy detector. Filters are used to pass the appropriate IR wavelength to each detector. In the absence of CO and CO_2, the energy received by each detector is at its maximum. During

[3] The last approved version of this historical standard is referenced on www.astm.org.



A—High Purity Oxygen
B—Oxygen Regulator (2 Stage)
C—Sodium Hydroxide Impregnated Clay and Magnesium Perchlorate
D—Secondary Pressure Regulator
E—Flowmeter
F—Induction Furnace
G—Combustion Tube
H—Dust Trap
I—Manganese Dioxide
J—Heated CO to CO_2 Converter (suitable catalyst)
K—Magnesium Perchlorate (Note 1 in 14.4)
L—Valve Manifold
M—CO_2 Absorber – Zeolite
N—Furnace Combustion Exhaust
O—Furnace Purge Exhaust
P—Metal Connector To Use Oxygen As Carrier Gas
Q—High Purity Helium
R—Helium Regulator (2 Stage)
S—Chromatographic Column
T—TC Cell/Readout
U—Measure Flowmeter
V—Reference Flowmeter
W—Furnace Power Supply

* May be sealed chamber if oxygen is carrier gas.

** Not required if oxygen is carrier gas.

FIG. 1 Apparatus for Determination of Carbon by the Combustion/ Thermal Conductivity Detection Test Method

combustion, the IR absorption properties of CO and CO_2 gases in the chamber cause a loss of energy; therefore a loss in signal results which is proportional to amounts of each gas in the closed loop. Total carbon, as CO_2 plus CO, is measured over a period of time. Refer to Fig. 4 for example.

11.2 This test method is written for use with commercial analyzers, equipped to perform the above operations automatically and calibrated using reference materials of known carbon content.

12. Interferences

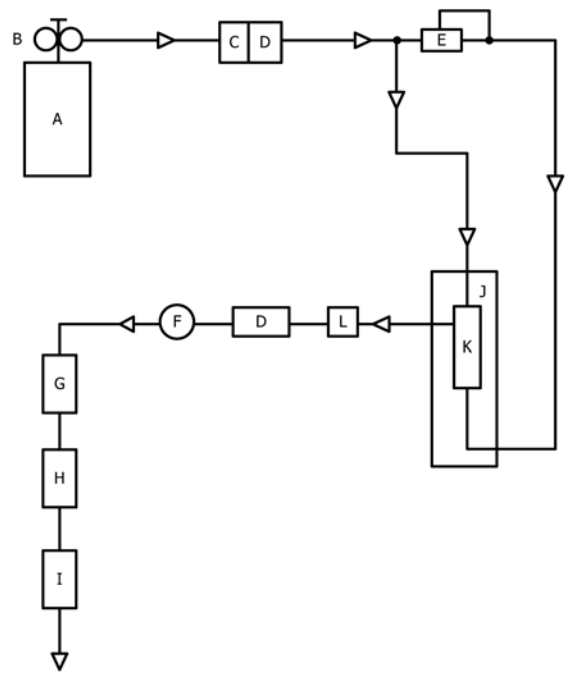
12.1 The elements ordinarily present in iron, steel, nickel, and cobalt alloys do not interfere.

13. Apparatus

13.1 *Combustion and Measurement Apparatus*—See Figs. 1-4 for examples.

13.2 *Crucibles*—Use crucibles that meet or exceed the specifications of the instrument manufacturer and prepare the crucibles by heating in a suitable furnace for not less than 40 min at approximately 1000 °C. Remove from the furnace and cool before use. Crucibles may be stored in a desiccator prior to use.

13.2.1 The analytical ranges for the use of untreated crucibles shall be determined by the testing laboratory and supporting data shall be maintained on file to validate these ranges. Heating of crucibles is particularly important when



A—Oxygen Cylinder
B—Two Stage Regulator
C—Sodium Hydroxide Impregnated Clay
D—Magnesium Percholorate (Note 1 in 14.4)
E—Regulator
F—Flow Controller
G—CO-CO_2 Converter (suitable catalyst)
H—SO_3 Trap
I—CO_2 IR Cell/Readout
J—Induction Furnace
K—Combustion Area
L—Dust Trap

FIG. 2 Infrared Absorption Detection Test Method A

analyzing for low levels of carbon and may not be required if the material to be analyzed has higher levels of carbon such as that found in pig iron (3.5% or greater). Above certain carbon mass fractions, as determined by the testing laboratory, the non-treatment of crucibles will have no adverse effect.

13.3 *Crucible Tongs*—Capable of handling recommended crucibles.

14. Reagents

14.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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.

14.2 *Acetone*—The residue after evaporation shall be < 0.0005 %.

14.3 *Copper (Low Carbon) Accelerator,* granular, 2.00 mm to 0.599 mm (10 mesh to 30 mesh).

14.3.1 The accelerator should contain no more than 0.001 % carbon. If necessary, wash three times with acetone by decantation to remove organic contaminants and dry at room temperature. The mm (mesh) size is critical to the inductive coupling that heats the sample. Some manufacturers of accelerators may not certify the mm (mesh) size on a lot to lot basis. These accelerators may be considered acceptable for use without verifying the mm (mesh) size.

14.4 *Magnesium Perchlorate,* (known commercially as *Anhydrone*) — Use the purity specified by the instrument manufacturer.

NOTE 1—Phosphorus pentoxide may be used by some instrument manufacturers.

14.5 *Oxygen*—Purity as specified by the instrument manufacturer.

14.6 *Platinum or Platinized Silica,* heated to 350 °C for the conversion of CO to CO_2. Use the form specified by the instrument manufacturer.

NOTE 2—Copper oxide may be used by some instrument manufacturers.

14.7 *Sodium Hydroxide on Clay (known commercially as Ascarite II)*—Use the purity specified by the instrument manufacturer.

14.8 *Tungsten (Low Carbon) Accelerator,* 1.68 mm to 0.853 mm (12 mesh to 20 mesh). See 14.3.1.

14.9 *Tungsten-Tin (Low Carbon) Accelerator,* 0.853 mm to 0.422 mm (20 mesh to 40 mesh) or 1.68 mm to 0.853 mm (12 mesh to 20 mesh). See 14.3.1.

15. Preparation of Apparatus

15.1 Assemble the apparatus as recommended by the manufacturer.

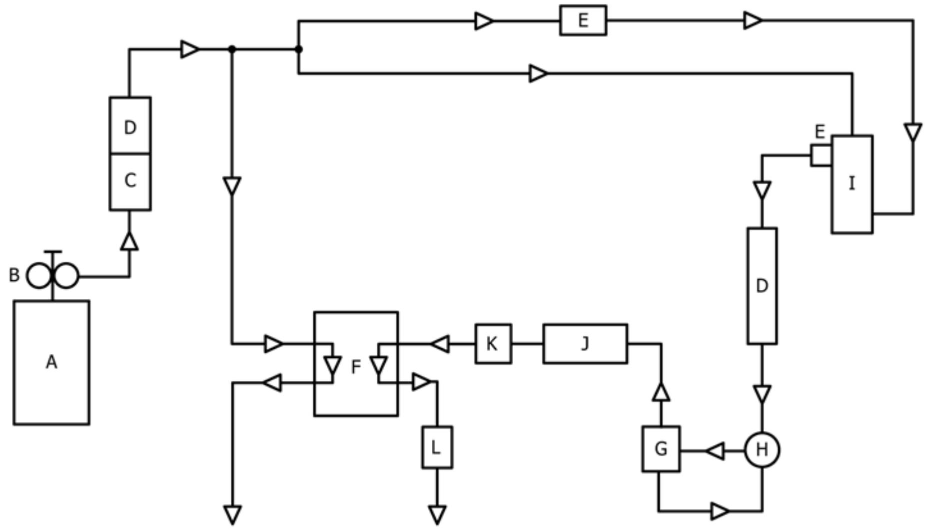
15.2 Test the furnace and analyzer to ensure the absence of leaks and make the required electrical power connections. Prepare the analyzer for operation as directed by the manufacturer's instructions. Change the chemical reagents and filters at the intervals recommended by the instrument manufacturer. Make a minimum of two determinations using the specimen and accelerator as directed in 18.1.2 and 18.1.3 to condition the instrument before attempting to calibrate the system or determine the blank. Avoid the use of reference materials for instrument conditioning.

15.2.1 Approximately 1.5 g of accelerator is typically required for proper combustion. However, the use of 1.5 g of accelerator may not be sufficient for all instruments. The required amount is determined by the instrument used, induction coil spacing, position of the crucible in the induction coil, age and strength of the oscillator tube, and type of crucible being used. Use the amount required to produce proper sample combustion and use the same amount throughout the entire test method.

16. Sample Preparation

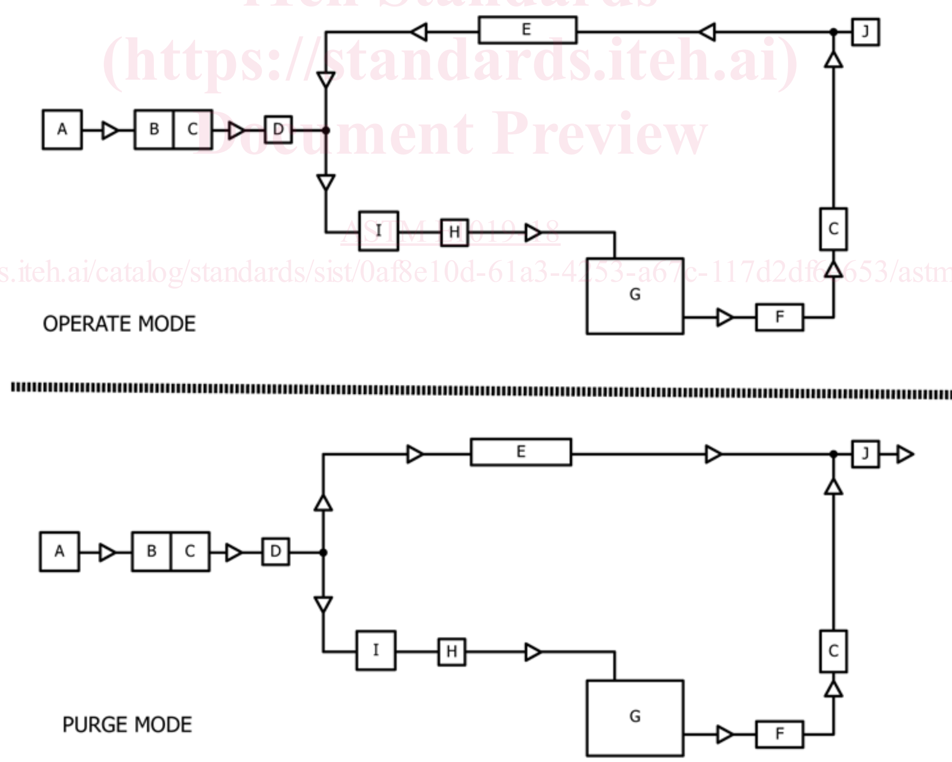
16.1 The specimens should be uniform in size, but not finer than 0.422 mm (40 mesh). Specimens will typically be in the

[4] *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 the *United States Pharmacopeia—National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD (http://www.usp.org/USPNF).



A—Oxygen Cylinder
B—Two Stage Regulator
C—Sodium Hydroxide Impregnated Clay
D—Magnesium Perchlorate (Note 1 in 14.4)
E—Dust Trap
F—IR Cell/Readout
G—Orifice
H—Pressure Regulator
I—Combustion Chamber
J—CO to CO_2 Converter (suitable catalyst)
K—SO_3 Trap (typically cellulose)
L—Measure Flow Rotameter

FIG. 3 Infrared Absorption Detection Test Method B



A—Oxygen Cylinder
B—Sodium Hydroxide Impregnated Clay
C—Magnesium Perchlorate (Note 1 in 14.4)
D—Pressure Regulator
E—IR Cell/Readout
F—Dust Trap
G—Furnace
H—Pump
I—Flow Meter
J—Exhaust
K—CO to CO_2 Converter (suitable catalyst)
L—SO_3 Trap (typically cellulose)

FIG. 4 Infrared Absorption Detection Test Method C—Closed Loop

form of chips, drillings, slugs, or solids. Specimens shall be free of any residual lubricants and cutting fluids. It may be necessary to clean specimens to remove residual lubricants and cutting fluids. Any cleaned specimens shall be rinsed in acetone (or another suitable solvent with low residue, see 14.2) and dried completely before analysis.

17. Calibration

17.1 *Calibration Reference Materials:*

NOTE 3—The accuracy of this test method is largely dependent upon the absence of bias in the values assigned to the reference materials and upon the homogeneity of these materials.

17.1.1 For Range I, 0.005 % to 0.10 % carbon, select three certified reference materials containing approximately 0.005 %, 0.05 %, and 0.10 % carbon and designate them as A, B, and C, respectively. An accelerator with a certified carbon value may be used as A.

17.1.2 For Range II, 0.10 % to 1.25 % carbon, select two certified reference materials containing approximately 0.12 % and 1.00 % carbon and designate them as BB and CC, respectively.

17.1.3 For Range III, 1.25 % to 4.50 % carbon, select two certified reference materials containing approximately 1.25 % and 4.00 % carbon and designate them as BBB and CCC, respectively.

17.1.4 Users may determine that only one or two ranges are necessary for calibration depending on the carbon range of samples to be tested.

17.2 *Adjustment of Response of Measurement System:*

17.2.1 Modern instruments may not require adjustment of the measurement system response prior to calibration. For these instruments proceed directly to 17.3 after the conditioning runs described in 15.2.

17.2.2 Transfer 1.0 g of B, weighed to the nearest 1 mg, and approximately 1.5 g of accelerator to a crucible. Some manufacturers provide scoops that dispense approximately 1.5 g of accelerator. Once it is verified that the scoop delivers this approximate mass, it is acceptable to use this device for routine dispensing of accelerator.

17.2.3 Proceed as directed in 18.1.2 and 18.1.3.

17.2.4 Repeat 17.2.2 and 17.2.3 until the absence of drift is indicated by stable carbon readings being obtained. Consistency is indicated by consecutive runs agreeing within 0.001 % carbon. If using an instrument which requires manual adjustment, adjust the signal to provide a reading within ± 0.003 % of the carbon value for the certified reference material.

17.3 *Determination of Blank Reading—Range I:*

17.3.1 Add approximately 1.5 g of accelerator into a crucible. If required, 1.0 g of A, weighed to the nearest 1 mg, may be added to the crucible.

17.3.2 Proceed as directed in 18.1.2 and 18.1.3.

17.3.3 Repeat 17.3.1 and 17.3.2 a sufficient number of times to establish that low (less than 0.002 % carbon) and stable (± 0.0002 % carbon) readings are obtained. Blank values are equal to the total result of the accelerator. If A was used, blank values are equal to the total result of the accelerator and A minus the value of A.

17.3.4 Record the average value of the last three or more stable blank determinations.

17.3.5 If the blank readings are too high or unstable, determine the cause, correct it, and repeat the steps as directed in 17.3.1 – 17.3.4.

17.3.6 Enter the average blank value in the analyzer. Refer to the manufacturer's instructions for specific instructions on performing this function. Typically the instrument will electronically compensate for the blank value. If the unit does not have this function, the blank value shall be subtracted from the total result prior to any calculation.

17.4 *Determination of Blank Reading—Range II*—Proceed as directed in 17.3.

17.5 *Determination of Blank Reading—Range III:*

17.5.1 Transfer 0.5 g of A, weighed to the nearest 1 mg, and approximately 1.5 g of accelerator to a crucible.

17.5.2 Proceed as directed in 17.3.2 – 17.3.6.

17.6 *Calibration—Range I (0.005 % to 0.10 % Carbon):*

17.6.1 Weigh four 1.0 g specimens of C, to the nearest 1 mg, then place in crucibles. To each, add approximately 1.5 g of accelerator (see 17.6.4.1).

17.6.2 Follow the calibration procedure recommended by the manufacturer. Use C as the primary calibration reference material (RM) and analyze at least three specimens to determine the measurement response to be used in the calibration regression. Treat each specimen, as directed in 18.1.2 and 18.1.3, before proceeding to the next one.

17.6.3 Confirm the calibration by analyzing C following the calibration procedure. If the result agrees with the certified value within the uncertainty provided on the certificate of analysis, the calibration is acceptable. Also, if the certified value falls within a prediction interval calculated as described in Eq 1, the calibration is acceptable (see Note 4). The prediction interval is defined as the range of values either bounded by the mean result and (mean result – p) or by the mean result and (mean result + p). Compare the certified value of the reference material to the appropriate calculated prediction interval. If the certified value of the reference material falls within the prediction interval, there is evidence that the calibration may not be biased. If the value does not fall within the prediction interval there may be calibration bias.

$$p = t \cdot \left(1 + \frac{1}{\sqrt{n}} \right) \cdot s \qquad (1)$$

where:

p = width of the prediction interval,
n = number of replicates used in 17.6.2,
t = student's t chosen for a one-sided interval at the 95 % confidence level for n replicate measurements. For example: t = 2.92 when n = 3 (degrees of freedom = 2); t= 2.35 when n = 4 (degrees of freedom = 3); t= 2.13, when n = 5 (degrees of freedom = 4), and
s = standard deviation of n replicates.

NOTE 4—The procedure for verifying calibration reference materials (RMs) outlined in the original version of this test method required the test result to be compared to "the uncertainty limits of the certified value for the calibration RM," typically interpreted as the range defined by the certified value plus or minus its associated uncertainty. The original version was utilized in the generation of the data in this test method's precision and bias statements. The current method in 17.6.3 for confirming the calibration is statistically rigorous and should be used in general practice. As an option, the laboratory may obtain an estimate of s from a control chart maintained as part of their quality control program. If the control chart contains a large number of measurements ($n > 30$), t may be set equal to 2 at the 95 % confidence level. At its discretion, the laboratory may choose to set a smaller range for the acceptable test result.

17.6.4 Weigh at least two 1.0 g specimens of B, weighed to the nearest 1 mg, and transfer them to crucibles. To each, add approximately 1.5 g of accelerator.

17.6.4.1 The use of 1.5 g of accelerator may not be sufficient for all determinators. The required amount is determined by the analyzer used, induction coil spacing, position of the crucible in the induction coil, age and strength of the oscillator tube, and type of crucible being used. Use the amount required to produce proper sample combustion using the same amount throughout the entire test method.

17.6.5 Treat each specimen as directed in 18.1.2 and 18.1.3 before proceeding to the next one.

17.6.6 Record the results of 17.6.4 and 17.6.5 and compare them to the certified carbon value of B. The result should agree with the certified value within a suitable confidence interval (see Note 4 in 17.6.3). If the result agrees with the certified value within the uncertainty provided on the certificate of analysis, the calibration is acceptable. Also, if the certified value falls within an interval calculated as described in Eq 1, the calibration is acceptable. If not, refer to the manufacturer's instructions for checking the linearity of the system.

17.7 *Calibration—Range II (0.10 % to 1.25 % carbon):*

17.7.1 Proceed as directed in 17.6.1 – 17.6.3, using CC.

17.7.2 Proceed as directed in 17.6.4 – 17.6.6, using BB.

17.8 *Calibration—Range III (1.25 % to 4.50 % carbon):*

17.8.1 Weigh four 0.5 g specimens of CCC, to the nearest 1 mg, and place in crucibles. To each, add approximately 1.5 g of accelerator. Follow the calibration procedure recommended by the manufacturer. Use CCC as the primary calibration RM and analyze at least three specimens to determine the calibration slope. Treat each specimen, as directed in 18.1.2 and 18.1.3, before proceeding to the next one.

17.8.2 Confirm the calibration by analyzing CCC following the calibration procedure. If the result agrees with the certified value within the uncertainty provided on the certificate of analysis, the calibration is acceptable. Also, if the certified value falls within an interval calculated as described in Eq 1, the calibration is acceptable. See Note 4 in 17.6.3.

17.8.3 If not, repeat 17.8.1 and 17.8.2.

17.8.4 Weigh at least two 0.5 g specimens of BBB, weighed to the nearest 1 mg, and transfer to crucibles. To each, add approximately 1.5 g of accelerator.

17.8.5 Treat each specimen as described in 18.1.2 and 18.1.3 before proceeding to the next one.

17.8.6 Record the results of 17.8.4 and 17.8.5 and compare to the certified carbon value of BBB. The result should agree with the certified value within a suitable confidence interval (see Note 4 in 17.6.3). If the result agrees with the certified value within the uncertainty provided on the certificate of analysis, the calibration is acceptable. Also, if the certified value falls within an interval calculated as described in Eq 1, the calibration is acceptable. If not, refer to manufacturer's instructions for checking the linearity of the analyzer.

17.8.7 Verify the calibration when: *(1)* a different lot of crucibles is used, *(2)* a different lot of accelerator is used, *(3)* the system has been in use for 4 h, *(4)* the oxygen supply has been changed, and *(5)* the system has been idle for 1 h. Verification should consist of analyzing at least one specimen of each calibration RM. Recalibrate as necessary.

18. Procedure

18.1 *Procedure—Range I:*

18.1.1 Stabilize the furnace and analyzer as directed in Section 15. Transfer approximately 1.0 g of specimen and approximately 1.5 g of accelerator to a crucible. (See 13.2.)

18.1.2 Place the crucible into the furnace mechanism. Use crucible tongs to handle the crucibles.

18.1.3 Refer to the manufacturer's recommended procedure regarding entry of specimen mass and blank value. Start the analysis cycle.

18.2 *Procedure—Range II*—Proceed as directed in 18.1.

18.3 *Procedure—Range III*—Proceed as directed in 18.1, using a 0.5 g specimen.

19. Calculation

19.1 The calibration function of the equipment shall yield a linear plot described by Eq 2.

$$Y = mX + b \tag{2}$$

where:

Y = measurement response,
m = slope,
X = calibration RM mass fraction, and
b = Y intercept.

TABLE 1 Statistical Information—Carbon, Range I

Test Specimen	Carbon Found, %	Repeatability (R_1, Practice E173)	Reproducibility (R_2, Practice E173)
1. Electrolytic iron (NIST 365, 0.0068 C)	0.007	0.002	0.003
2. Bessemer carbon steel (NIST 8j, 0.081 C)	0.080	0.003	0.006
3. Type 304L stainless steel 18Cr-8Ni (NIST 101f, 0.014 C)	0.014	0.002	0.004
4. Type 446 stainless steel 26Cr (NIST 367, 0.093 C)	0.094	0.003	0.004
5. Nickel steel 36Ni (NIST 126b, 0.090 C)	0.092	0.003	0.004
6. Waspaloy 57Ni-20Cr-14Co-4Mo (NIST 349, 0.080 C)	0.078	0.003	0.004
7. Silicon steel (NIST 131a, 0.004 C)	0.004	0.002	0.002
8. High temperature alloy A286 26Ni-15Cr (NIST 348, 0.044 C)	0.046	0.003	0.004

TABLE 2 Statistical Information—Carbon, Range II

Test Specimen	Carbon Found, %	Repeatability (R_1, Practice E173)	Reproducibility (R_2, Practice E173)
1. Basic open hearth steel (NIST 11h, 0.200 C)	0.201	0.006	0.010
2. Basic open hearth carbon steel (NIST 337, 1.07 C)	1.087	0.039	0.053
3. Low alloy electric furnace steel (NIST 51b, 1.21 C)	1.224	0.039	0.048
4. High temperature nickel alloy (LE 105, 0.130 C)	0.130	0.005	0.008
5. Tool steel 8Co-9Mo-2W-4Cr-2V (NIST 153a, 0.902 C)	0.905	0.023	0.027
6. Type 416 stainless steel (NIST 133b, 0.128 C)	0.126	0.005	0.013
7. Low alloy steel 1Cr (NIST 163, 0.933 C)	0.934	0.016	0.020

TABLE 3 Statistical Information—Carbon, Range III

Test Specimen	Carbon Found, %	Repeatability (R_1, Practice E173)	Reproducibility (R_2, Practice E173)
1. Tool steel (CISRI 150, 1.56 C)	1.550	0.027	0.049
2. Low alloy electric furnace steel (NIST 51b, 1.21 C)	1.228	0.039	0.050
3. Cast iron (LECO 501-105, 2.20 C)	2.202	0.044	0.056
4. Ductile iron (LECO 501-083, 4.24 C)	4.244	0.083	0.091
5. White iron (LECO 501-024, 3.25 C)	3.274	0.064	0.074
6. Iron (BAM 035-1, 1.31 C)	1.314	0.034	0.048
7. Ferritic stainless steel (BAM 228-1, 2.05 C)	2.040	0.027	0.055

Calculation of the calibration function shall be done using a linear least squares regression. Some manufacturers recommend the use of a curve weighting factor where the calibration RM mass fraction is derived as 1/*X*. It is acceptable to use this type of curve weighting.

19.2 Since most modern commercially available instruments calculate mass fractions directly, including corrections for blank and sample mass, manual calculations by the analyst are not required.

19.2.1 If the analyzer does not compensate for blank and sample mass values, then use the following formula:

$$Carbon,\ \% = \left[(A - B) \times C/D\right] \quad (3)$$

where:

A = instrument reading for specimen,
B = instrument reading for blank,
C = mass compensator setting, and
D = specimen mass, g.

20. Precision and Bias[5]

20.1 *Precision*—Nine laboratories cooperated in testing this test method and obtained the data summarized in Table 1 through Table 3. Testing was performed in compliance with Practice E173 (see 9.1).

20.2 *Bias*—The accuracy of this test method has been deemed satisfactory based upon the data for the certified reference materials in Table 1, Table 2, and Table 3. Users are encouraged to use these or similar reference materials to verify that the test method is performing accurately in their laboratories.

SULFUR BY THE COMBUSTION–INFRARED ABSORPTION TEST METHOD (POTASSIUM SULFATE CALIBRATION)

This test method, which consisted of Sections 21 through 31 of this standard, was discontinued in 2018.

NITROGEN BY THE INERT GAS FUSION AND THERMAL CONDUCTIVITY DETECTION TEST METHOD

32. Scope

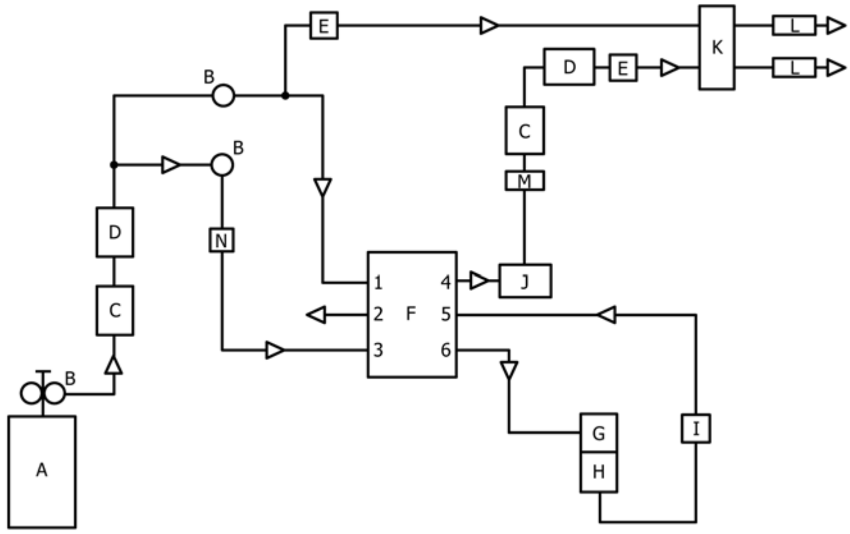
32.1 This test method covers the determination of nitrogen from 0.0010 % to 0.2 %.

32.1.1 The upper limit of the scope has been set at 0.2 % because sufficient numbers of test materials containing higher nitrogen contents were unavailable for testing as directed in Practice E173. However, recognizing that commercial nitrogen determinators are capable of handling higher compositions, this test method provides a calibration procedure up to 0.5 %. Users of this test method are cautioned that use of it above 0.2 % is not supported by interlaboratory testing. In this case, laboratories should perform method validation using reference materials.

33. Summary of Test Method

33.1 The specimen, contained in a small, single-use graphite crucible, is fused under a flowing helium atmosphere at a minimum temperature of 1900 °C. Nitrogen present in the sample is released as molecular nitrogen into the flowing helium stream. The nitrogen is separated from other liberated gases such as hydrogen and CO and is finally measured in a thermal conductivity cell. Refer to Figs. 5-8 for examples.

[5] Supporting data are available from ASTM International Headquarters. Request RR:E01-1093.



A—Helium Supply
B—Pressure Regulator 2 Stage
C—Sodium Hydroxide Impregnated Clay
D—Magnesium Perchlorate (Note 1 in 14.4)
E—Flow Control
F—Flow Manifold
G—Sample Holding Chamber
H—Electrode Furnace (Note 5 in 35.1)
I—Dust Filter
J—Heated Rare Earth Copper Oxide
K—Thermal Conductive Detector/Readout
L—Flow Rotameter
M—Charcoal
N—Flow Restrictor

Manifold Porting

Crucible Degas Flow: 1 *to* 4; 5 *to* 2; 3 *to* 6

Fusion Flow: 1 *to* 6; 5 *to* 4; 3 & 2

FIG. 5 Nitrogen Test Method A—Flow Diagram

33.2 This test method is written for use with commercial analyzers equipped to perform the above operations automatically and calibrated using reference materials of known nitrogen content.

34. Interferences

34.1 The elements ordinarily present in iron, steel, nickel, and cobalt alloys do not interfere.

35. Apparatus

35.1 *Fusion and Measurement Apparatus*—See Figs. 5-8 for examples.

NOTE 5—Some manufacturers may use an induction furnace

35.2 *Graphite Crucibles*—Use the size crucibles recommended by the manufacturer of the instrument. Crucibles shall be composed of high purity graphite.

35.3 *Crucible Tongs*—Capable of handling recommended crucibles.

36. Reagents

36.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall 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.

36.2 *Acetone*—The residue after evaporation shall be <0.0005 %.

36.3 *Copper*—, use the purity and form specified by the instrument manufacturer.

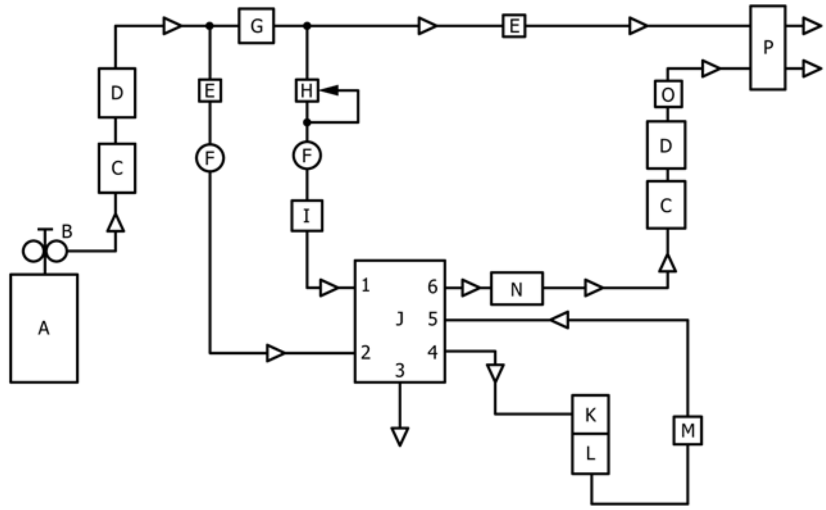
36.4 *Helium,* high-purity (99.99 %).

36.5 *Magnesium Perchlorate,* (known commercially as *Anhydrone*). Use the purity specified by the instrument manufacturer. See Note 1 in 14.4.

36.6 *Rare Earth Copper Oxide*—Use the purity as specified by the instrument manufacturer.

36.7 *Silica,* as specified by the instrument manufacturer.

36.8 *Sodium Hydroxide on Clay (known commercially as Ascarite II)*—Use the purity as specified by the instrument manufacturer.



A—Helium Supply
B—Pressure Regulator 2 Stage
C—Sodium Hydroxide Impregnated Clay
D—Magnesium Perchlorate (Note 1 in 14.4)
E—Flow Restrictor
F—Flow Meter
G—Pressure Meter
H—Needle Valve
I—Optional Gas Doser
J—Flow Manifold
K—Sample Holding Chamber
L—Electrode Furnace (Note 5 in 35.1)
M—Dust Filter
N—Heated Rare Earth Copper Oxide
O—Flow Control
P—Thermal Conductive Detector Readout

Manifold Porting

Crucible Degas Flow: 2 *to* 4; 5 *to* 3; 1 *to* 6
Fusion Flow: 1 *to* 4; 5 *to* 6; 2 & 3 *off*

FIG. 6 Nitrogen Test Method B—Flow Diagram

37. Preparation of Apparatus

37.1 Assemble the apparatus as recommended by the manufacturer.

37.2 Test the furnace and analyzer to ensure the absence of leaks, and make the required electrical power and water connections. Prepare the apparatus for operation as directed in the manufacturer's instructions. Change the chemical reagents and filters at the intervals recommended by the instrument manufacturer. Make a minimum of two determinations using a specimen as directed in 40.2.1 or 40.2.2 to condition the instrument before attempting to calibrate the system or to determine the blank. Avoid the use of reference materials for instrument conditioning.

37.2.1 Many instrument manufacturers provide a cycle time in the analysis parameters to pre-heat the crucible (commonly referred to as degassing or outgassing) before a blank can be determined or a sample can be added to the crucible for analysis. This outgassing removes any absorbed impurities from the crucible. See Figs. 5-8 for examples of crucible degas flow.

38. Sample Preparation

38.1 Practice E1806 provides some guidance for sampling and preparation of steel and iron alloys for gas analysis. Specimens will typically be in the form of chips, drillings, slugs, or solids. Final specimen preparation shall be performed as directed in 38.2 or 38.3.

38.1.1 Size all specimens to permit free introduction through the loading device of the equipment or directly into the graphite crucible.

38.2 If slugs or solid-form specimens are used, cut them with a water-cooled abrasive cut-off wheel or by another means that will prevent overheating. Abrade the surface to remove surface oxidation using a clean file, die grinder, or silicon carbide grinding media. Again, avoid overheating the sample. If specimens are wet ground it will be necessary to rinse specimens in water followed by an acetone rinse. Samples shall be air dried completely prior to analysis.

38.3 Clean, dry chips and millings may be analyzed without additional preparation; however, specimens shall be free of any lubricants and cutting fluids. It may be necessary to clean specimens to remove residual lubricants and cutting fluids. Any