INTERNATIONAL

**Designation: ~~E1019 – 11~~ 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~~<br>Element | ~~Concentration Range, %~~<br>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 (Metal Reference Materials)~~ | ~~0.002 to 0.35~~ |
| Sulfur | 0.002 to 0.35 |
| ~~Sulfur (Potassium Sulfate)~~ | ~~0.001 to 0.600~~ |
| 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

[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.

Current edition approved ~~March 15, 2011~~April 15, 2018. Published ~~June 2011~~June 2018. Originally approved in 1984. Last previous edition approved in ~~2008~~2011 as ~~E1019 – 08.~~E1019 – 11. DOI: ~~10.1520/E1019-11.~~10.1520/E1019-18.

| | |
|---|---|
| ~~Carbon, Total, by the Combustion-Instrumental Measurement Test Method~~ | ~~10 – 20~~ |
| Carbon, Total, by the Combustion and Infrared Absorption or Thermal Conductivity Detection Test Method | 10 – 20 |
| ~~Nitrogen by the Inert Gas Fusion-Thermal Conductivity Test Method~~ | ~~32 – 42~~ |
| 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 Test Method (Calibration with Metal Reference Materials)~~ | ~~55 – 65~~ |
| Sulfur by the Combustion-Infrared Absorption Detection Test Method | 55 – 65 |
| ~~Sulfur by the Combustion-Infrared Absorption Test Method (Potassium Sulfate Calibration)~~ | ~~21 – 31~~ |
| 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~~ safety, health, and ~~health~~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

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.

[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.

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

## 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 ~~for sampling~~to sample the materials, refer to those parts of Practice E1806.

## 8. Rounding Calculated Values

8.1 ~~Calculated values shall be rounded to the desired number of places~~ 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 ~~INSTRUMENTAL MEASUREMENT~~ AND INFRARED ABSORPTION OR THERMAL CONDUCTIVITY DETECTION TEST METHOD

## ~~10. Scope~~

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

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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 ~~carbon~~$CO_2$ ~~dioxide~~ 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 ~~carbon~~$CO_2$ ~~dioxide~~ 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 ~~carbon~~$CO_2$ ~~dioxide~~ is measured by infrared (IR) absorption. ~~Carbon dioxide (CO~~$CO_2$~~)~~ 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 ~~concentration~~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 ~~monitored and~~ measured over a period of time. Refer to Fig. 2~~.~~ for example.

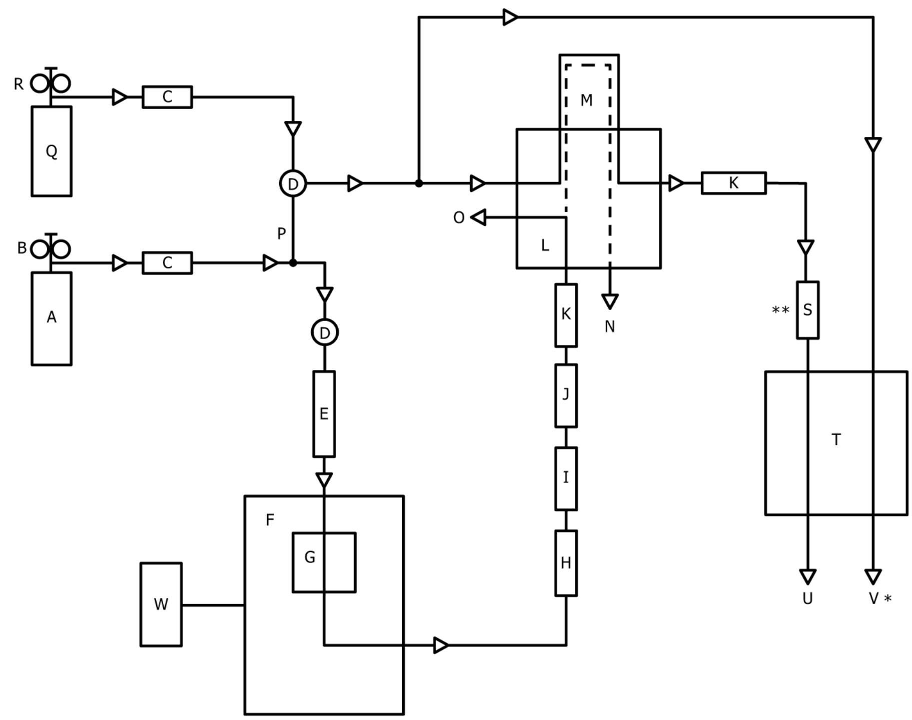
11.1.3 *Infrared (IR) Absorption, Test Method B*—The detector consists of an IR energy source, a separate measure chamber and reference chamber, and a diaphragm acting as one plate of a parallel plate capacitor. During specimen combustion, the flow of $CO_2$ with its oxygen ~~gas~~ carrier gas is routed 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 ~~fixed~~ capacitance creating an electric signal change that is amplified for measurement as $CO_2$. Total carbon, as $CO_2$, is ~~monitored and~~ 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 ~~CO~~ 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 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 ~~concentrations~~amounts of each gas in the closed loop. Total carbon, as $CO_2$ plus CO, is ~~monitored and~~ 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 ~~steels~~ reference materials of known carbon content.

## 12. Interferences

12.1 ~~For the scope of elements typically found in materials to be tested by this method refer to~~ The elements ordinarily present in iron, steel, nickel, and cobalt alloys do not interfere.~~1.1.~~




A—High Purity Oxygen
A—High Purity Oxygen
B—Oxygen Regulator (2 Stage)
C—Sodium Hydroxide Impregnated Clay/Magnesium Perchlorate
C—Sodium Hydroxide Impregnated Clay and Magnesium Perchlorate
D—Secondary Pressure Regulator
E—Flowmeter
F—Induction Furnace
G—Combustion Tube
G—Combustion Tube
H—Dust Trap
I—Manganese Dioxide
J—Heated CO to $CO_2$ Converter (suitable catalyst)
K—Magnesium Perchlorate
K—Magnesium Perchlorate (Note 1 in 14.4)
L—Valve Manifold

M—$CO_2$ Collection Trap
M—$CO_2$ Absorber – Zeolite
N—Furnace Combustion Exhaust
O—Furnace Purge Exhaust
O—Furnace Purge Exhaust
P—Metal Connector To Use Oxygen As Carrier Gas
Q—High Purity Helium
R—Helium Regulator (2 Stage)
S—Chromagraphic Column
S—Chromatographic Column
T—TC Cell/Readout
U—Measure Flowmeter
V—Reference Flowmeter
W—Furnace Power Stat
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 CombustionCombustion/ Thermal Conductivity Detection Test Method**

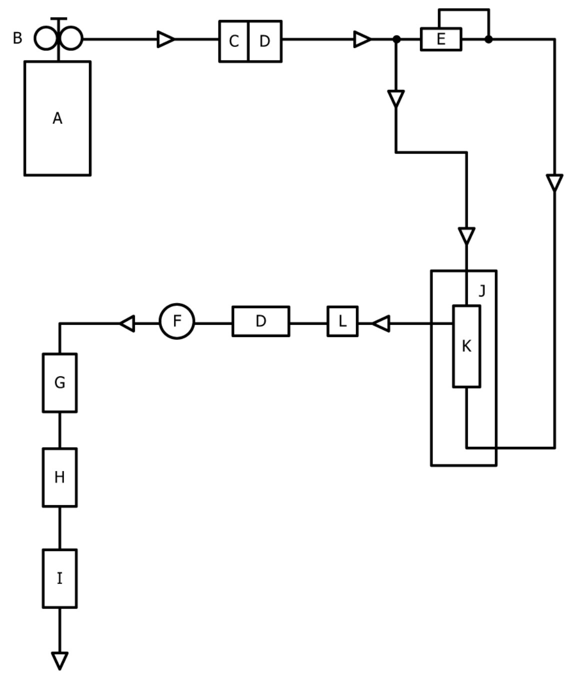


## 13. Apparatus

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. Heating of crucibles is particularly important when 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. Above certain concentrations, as determined by the testing laboratory, the nontreatment of crucibles will have no adverse effect. 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.

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 analyzing for low levels




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**

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),~~ Carbon) Accelerator,* ~~granular~~ granular, 2.00 mm to 0.599 mm (10 mesh to 30 ~~mesh) (~~mesh).~~Note 1).~~

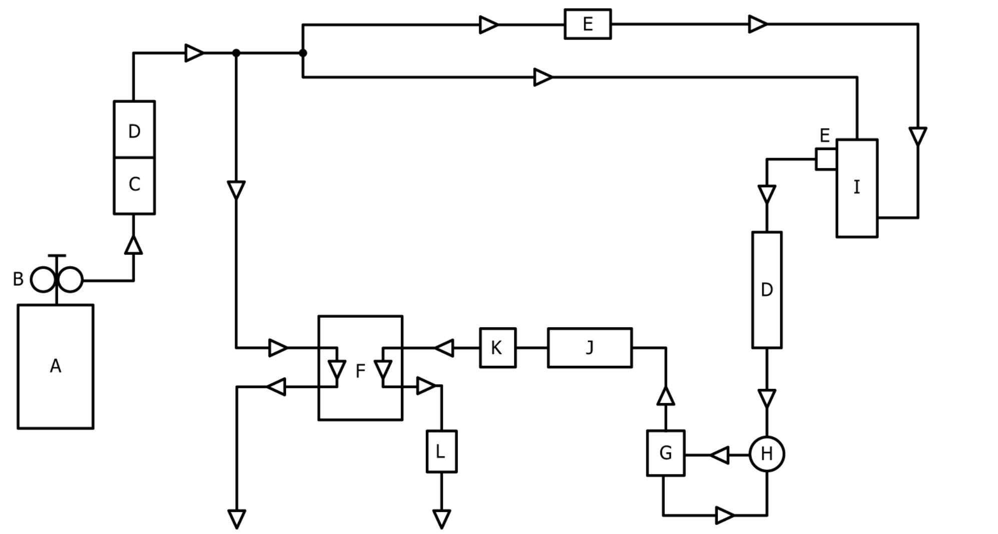
14.3.1 The accelerator should contain no more than 0.001 % carbon. If necessary, wash three times with acetone by decantation to remove organic contaminants and dry at room temperature. The mm (mesh) size is critical to the inductive coupling that heats the sample. Some manufacturers of accelerators 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.

[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 ~~Analar Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K. (http://uk.vwr.com), and~~ 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 Percholorate (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**



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

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

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

14.8 *Tungsten (Low Carbon) Accelerator,* ~~12~~ 1.68 mm to 0.853 mm (12 mesh to 20 ~~mesh (~~mesh). See~~Note 1~~ 14.3.1~~)~~.

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

~~NOTE 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 mesh size is critical to the inductive coupling which heats the sample. Some manufacturers of accelerators may not certify the mesh size on a lot to lot basis. These accelerators may be considered acceptable for use without verifying the mesh size.~~

## 15. Preparation of Apparatus

15.1 Assemble the apparatus as recommended by the manufacturer.

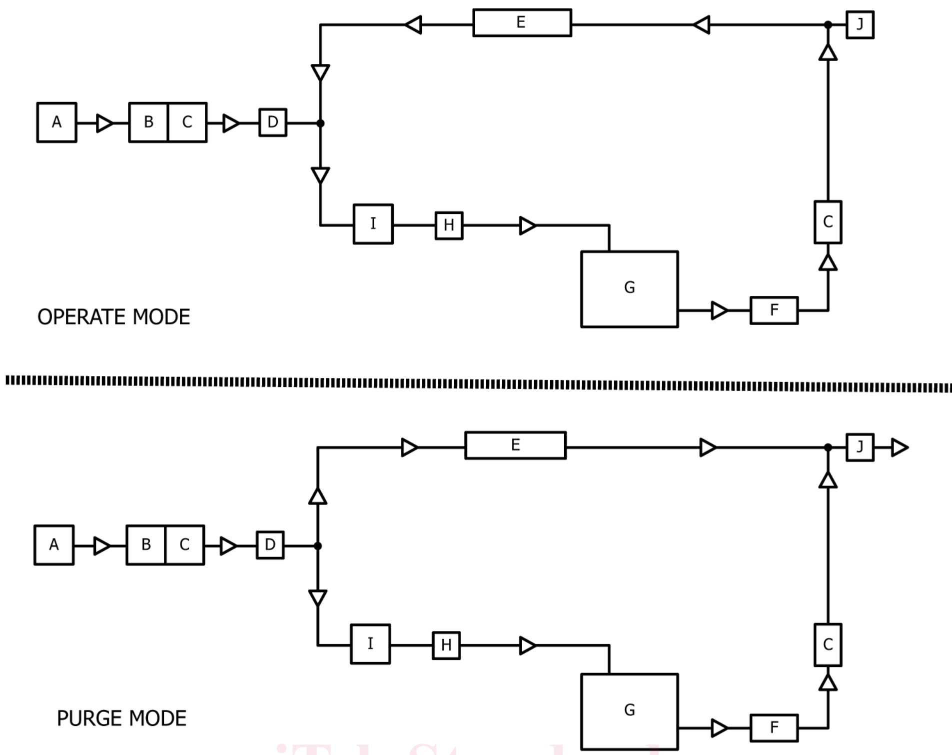
15.2 Test the furnace and analyzer to ensure the absence of leaks and make the required electrical power connections. Prepare the analyzer for operation ~~in a manner consistent with~~ as directed by the manufacturer's instructions. Change the chemical reagents and filters at the intervals recommended 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 ~~40 mesh.~~ 0.422 mm (40 mesh). Specimens will typically be in the 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.

~~16.2 If necessary, wash in acetone or another suitable solvent and dry.~~




A—Oxygen Cylinder
B—Sodium Hydroxide Impregnated Clay
C—Magnesium Perchlorate (Note 1 in 14.4)
~~D—Press Regulator~~
D—Pressure Regulator
E—IR Cell/Readout
F—Dust Trap
G—Furnace
H—Pump
I—Flow Meter
~~J—Exhaust~~
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**



## 17. Calibration

17.1 *Calibration Reference ~~Materials~~Materials:* ~~(Note 2):~~

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 ~~Calibrants~~ A, B, and C, respectively. ~~Some labs may use~~ An accelerator with a certified carbon value ~~as Calibrant~~ 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 ~~Calibrants~~ 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 ~~Calibrants~~ BBB and CCC, respectively.

~~NOTE 2—The uncertainty of results obtained using this test method is dependent on the uncertainty of the values assigned to the calibration reference materials. The homogeneity of the reference materials shall be considered as well, if it was not included in the derivation of the published uncertainty values.~~

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 ~~Calibrant~~ 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 certified percent 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 Calibrant 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 Calibrant A was used, blank values are equal to the total result of the accelerator and Calibrant A minus the certified value of Calibrant 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 (analyzer. Note 3). 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.

NOTE 3—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 Calibrant 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 Calibrant C, to the nearest 1 mg, then place in crucibles. To each, add approximately 1.5 g of accelerator (see Note 517.6.4.1).



17.6.2 Follow the calibration procedure recommended by the manufacturer. Use Calibrant C as the primary calibrant 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 Calibrant C following the calibration procedure. The result should agree with the certified value within a suitable confidence interval (see Note 4). 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 a prediction interval calculated as described in Eq 1, the calibration is acceptable. 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.

$$Test\ Result - t \cdot s \leq Certified\ Value \leq Test\ Result + t - s \tag{1}$$

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

*where:*

$s$ = standard deviation of the analyses run in 17.6,
$n$ = number of analyses (that is, 3 to 5), and
$t$ = Student's $t$ value, which is for $n = 3$, $t = 4.30$; for $n = 4$, $t = 3.18$; for $n = 5$, $t = 2.78$ at *the* 95 % confidence level.

$p$ = 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 calibrants 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 calibrant," 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 standardizationcalibration 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 ~~Calibrant~~ 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 ~~Calibrant~~ 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.

~~NOTE 5—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.~~

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

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

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

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

17.8.1 Weigh four 0.5 g specimens of ~~Calibrant~~ 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 ~~Calibrant~~ CCC as the primary ~~calibrant~~ 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 ~~Calibrant~~ CCC following the calibration procedure. ~~The result should agree with the certified value within a suitable confidence interval (see Note 4).~~ 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 ~~Calibrant~~ 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 ~~Calibrant~~ 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 (~~analyzer.~~Note 6).~~

~~NOTE 6—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 calibrant. Recalibrate as necessary.~~

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 ~~on the furnace pedestal and raise the pedestal into position.~~ 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.

$$Y = mX + b \quad (2)$$

**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 |



where:

$Y$ = measurement response,
~~$M$ = slope,~~
$m$ = slope,
~~$X$ = calibrant concentration, and~~
$X$ = calibration RM mass fraction, and
$b$ = $Y$ intercept.

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 ~~calibrant concentration~~ 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 ~~fraction concentrations~~ fractions directly, including corrections for blank and sample mass, manual calculations by the analyst are not required.

~~NOTE 7—If the analyzer does not compensate for blank and sample mass values, then use the following formula:~~

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

~~where:~~

~~$A$ = DVM (Digital Volt Meter) reading for specimen,~~
~~$B$ = DVM reading for blank,~~
~~$C$ = mass compensator setting, and~~
~~$D$ = specimen mass, g.~~

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

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

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 ~~Tables 1-3~~Table 1 through Table 3. Testing was performed in compliance with Practice E173 (see 9.1).

20.2 *Bias*—~~No information on the bias~~ The accuracy of this ~~method is known because at the time of the interlaboratory study, suitable reference materials were~~test method has been deemed satisfactory based upon the data for the certified reference materials in Table 1~~not~~, Table 2~~available. The~~, and Table 3~~user of this method is encouraged to employ accepted reference materials, if available, to determine the presence or absence of bias.~~ . 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)

## ~~21. Scope~~

~~21.1 This test method covers the determination of sulfur in the range of 0.001 % to 0.01 %. As written, this test method is not applicable to cast iron samples.~~

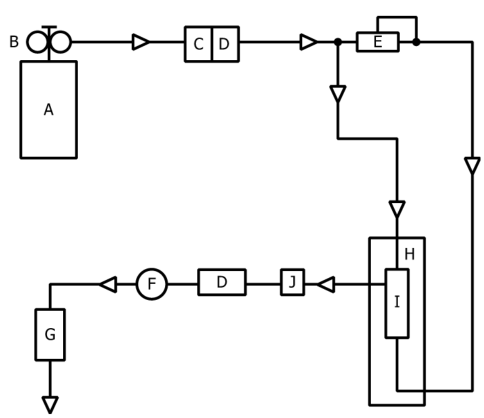
## ~~22. Summary of Test Method~~

~~22.1 The sample is combusted in a stream of oxygen that converts the sulfur in the sample to sulfur dioxide. The sulfur is measured using infrared absorption spectrometry~~

~~22.1.1 *Infrared Absorption Test Method A*—Sulfur dioxide ($SO_2$) 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. Therefore, the absorption of IR energy can be attributed to only $SO_2$ and its concentration is measured as changes in energy at the detector. One cell is used as both a reference and a measure chamber. Total sulfur, as $SO_2$, is monitored and measured over a period of time. Refer to Fig. 5.~~

~~22.1.2 *Infrared Absorption Test Method B*—The combustion is performed in a closed loop where $SO_2$ is detected in an infrared cell. The $SO_2$ is measured with a solid state energy detector, and filters are used to pass the appropriate IR wavelength to the detector. During combustion, the IR absorption properties of the $SO_2$ gas in the chamber causes a loss of energy, therefore a loss in signal results which is proportional to the concentration of the gas in the closed loop. Total sulfur, as $SO_2$, is measured over a period of time. Refer to Fig. 6.~~






A—Oxygen Cylinder
B—Two Stage Regulator
C—Sodium Hydroxide Impregnated Clay
D—Magnesium Perchlorate (Note 1 in 14.4)
E—Regulator
F—Flow Controller
G—IR Cell/Readout
H—Induction Furnace
I—Combustion Area
J—Dust Trap

**FIG. ~~5~~11 Infrared Absorption Detection Test Method A**

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