



Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys¹

This standard is issued under the fixed designation E 1019; 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 methods² cover the determination of carbon, sulfur, nitrogen, and oxygen, in steel and in iron, nickel, and cobalt alloys having chemical compositions within the following limits:

Element	Concentration 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
Columbium	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 Standards)	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 Nov. 10, 2000. Published January 2001. Originally published as E 1019 – 84. Last previous edition E 1019 – 94^{ε1}.

² Some of these test methods represent revisions of test methods covered by ASTM Methods E 350, E 351, E 352, E 353, and E 354 which appear in the *Annual Book of ASTM Standards*, Vol 03.05.

Carbon, Total, by the Combustion-Instrumental Measurement Method	9-19
Nitrogen by the Inert Gas Fusion-Thermal Conductivity Method	31-41
Oxygen by the Inert Gas Fusion Method	42-53
Sulfur by the Combustion-Infrared Absorption Method (Calibration with Metal Standards)	54-64
Sulfur by the Combustion-Infrared Absorption Method (Potassium Sulfate Calibration)	20-30

1.3 *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 hazards statements are given in Section 5.

2. Referenced Documents

2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications³
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴
- E 1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition⁵
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁶

3. Significance and Use

3.1 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.

4. Apparatus and Reagents

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

5. Hazards

5.1 For hazards to be observed in the use of certain reagents

³ *Annual Book of ASTM Standards*, Vol 14.02.

⁴ *Annual Book of ASTM Standards*, Vol 03.05.

⁵ *Annual Book of ASTM Standards*, Vol 03.06.

⁶ Discontinued 1998; see 1997 *Annual Book of ASTM Standards*, Vol 03.06.

in this test method, refer to Practices E 50.

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

6. Sampling

6.1 For procedures for sampling the materials, refer to those parts of Practice E 1806.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places as directed in Practice E 29.

8. Interlaboratory Studies

8.1 These test methods have been evaluated in accordance with Practice E 173.

TOTAL CARBON BY THE COMBUSTION INSTRUMENTAL MEASUREMENT METHOD

9. Scope

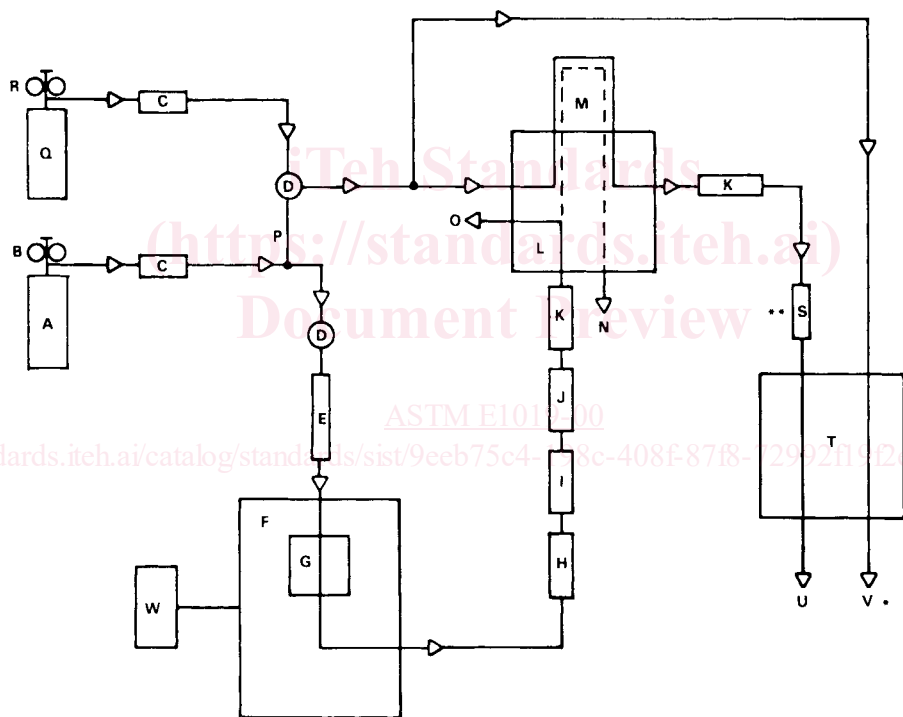
9.1 This test method covers the determination of carbon in concentrations from 0.005 to 4.5 %.

10. Summary of Test Method

10.1 The carbon is converted to carbon dioxide by combustion in a stream of oxygen.

10.1.1 *Thermal Conductivity Method*—The carbon 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 dioxide is measured in a thermistor-type conductivity cell. Refer to Fig. 1.

10.1.2 *Infrared (IR) Absorption, Method A*—The amount of carbon dioxide is measured by infrared (IR) absorption. Carbon dioxide (CO₂) absorbs IR energy at a precise wavelength



- A—High Purity Oxygen
- B—Oxygen Regulator (2 Stage)
- C—Sodium Hydroxide Impregnated Clay/Magnesium Perchlorate
- D—Secondary Pressure Regulator
- E—Flowmeter
- F—Induction Furnace
- G—Combustion Tube
- H—Dust Trap
- I—Manganese Dioxide
- J—Heated CO to CO₂ Converter
- K—Magnesium Perchlorate
- L—Valve Manifold

- M—CO₂ Collection Trap
- 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 Stat

* 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 Method

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 CO₂ 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 carbon, as CO₂, is monitored and measured over a period of time. Refer to Fig. 2.

10.1.3 *Infrared (IR) Absorption, 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 CO₂ with its oxygen gas carrier 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 CO₂ 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 CO₂. Total carbon, as CO₂, is monitored and measured over a period of time. Refer to Fig. 3.

10.1.4 *Infrared (IR) Absorption, Method C, Closed Loop*—The combustion is performed in a closed loop, where CO and CO₂ 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 CO₂, the energy received by each detector is maximum. During combustion, the IR absorption properties of CO and CO₂ gases in the chamber cause a loss of energy; therefore a loss in signal results which is proportional to concentrations of each gas in the closed loop. Total carbon, as CO₂ plus CO, is monitored and measured over a period of time. Refer to Fig. 4.

10.2 This test method is written for use with commercial analyzers, equipped to carry out the above operations automatically and calibrated using steels of known carbon content.

11. Interferences

11.1 The elements ordinarily present do not interfere.

12. Apparatus

12.1 *Combustion and Measurement Apparatus*—See Figs. 1-4.

12.2 *Crucibles*—Use crucibles that meet or exceed the specifications of those recommended by the manufacturer of the instrument used and preheat in a suitable furnace for not less than 40 min at more than 1000°C. Remove from the furnace and cool in air for 90 ± 15 s before use. Crucibles may be placed in a desiccator for periods of 1 h prior to use.

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

13. Reagents

13.1 *Acetone*—The residue after evaporation must be <0.0005 %.

13.2 *Copper (Low Carbon)*, granular (10 to 30 mesh) (Note 1).

13.3 *Oxygen, Ultra High Purity* (purity: 99.95 % minimum)—Other grades of oxygen may be used if low and consistent blank readings are obtained, or the oxygen may be purified as described in Practices E 50.

13.4 *Platinum or Platinized Silica*, heated to 350°C for the conversion of carbon monoxide to carbon dioxide.

13.5 *Tungsten (Low Carbon)*, 12 to 20 mesh (Note 1).

13.6 *Tungsten-Tin (Low Carbon)*, 20 to 40 mesh.

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.

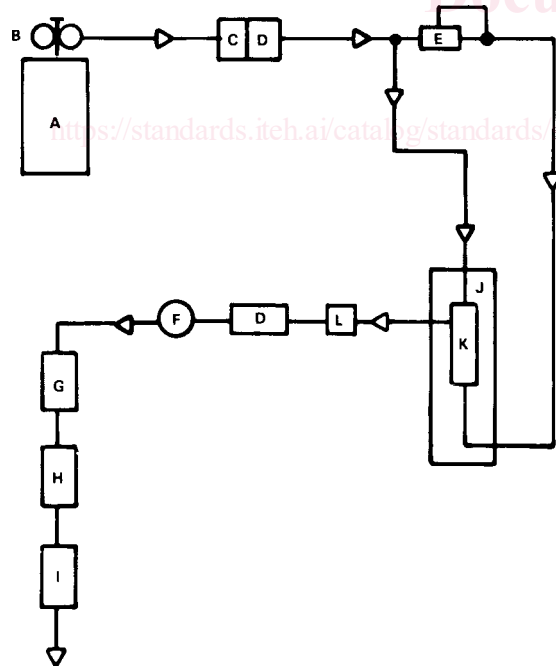
14. Preparation of Apparatus

14.1 Assemble the apparatus as recommended by the manufacturer.

14.2 Test the furnace and analyzer to ensure the absence of leaks and make the required electrical power connections. Prepare the analyzer for operation according to the manufacturer's instructions. Make a minimum of two determinations using the specimen and accelerator as directed in 17.1.2 and 17.1.3 before attempting to calibrate the system or determine the blank.

15. Sample Preparation

15.1 The sample should be uniform in size, but not finer than 40 mesh.



A—Oxygen Cylinder
B—Two Stage Regulator
C—Sodium Hydroxide Impregnated Clay
D—Magnesium Perchlorate
E—Regulator
F—Flow Controller
G—CO-CO₂ Converter
H—SO₃ Trap
I—CO₂ IR Cell/Readout
J—Induction Furnace
K—Combustion Area
L—Dust Trap

FIG. 2 Infrared Absorption Method A

E 1019

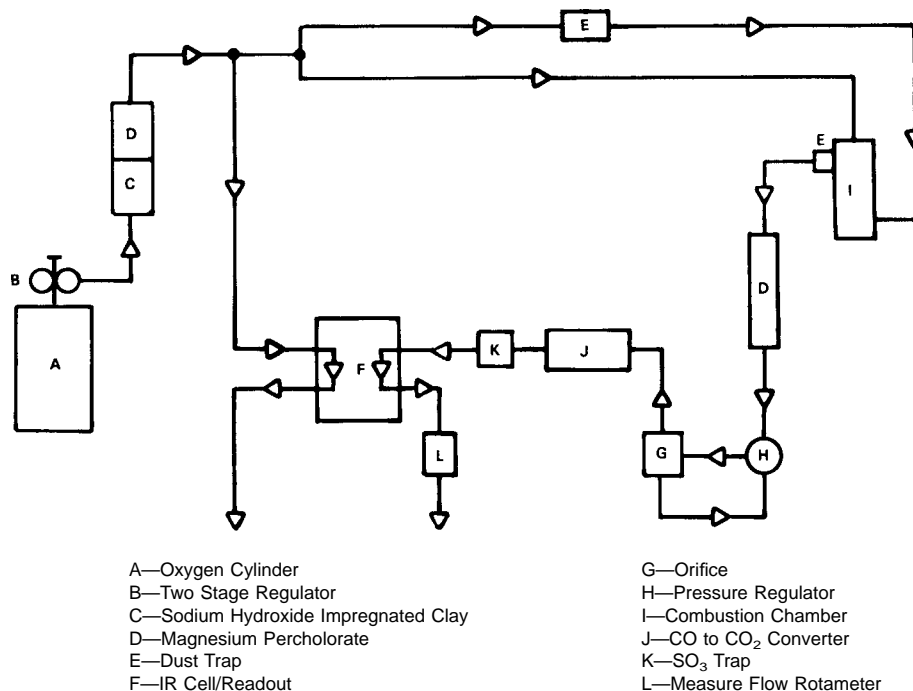


FIG. 3 Infrared Absorption Method B

15.2 Wash in acetone and dry at 70 to 100°C.

16. Calibration

16.1 *Calibration Standards* (Note 2):

16.1.1 For Range I, 0.005 to 0.10 % carbon, select three primary standard reference materials containing approximately 0.005, 0.05, and 0.10 % carbon and designate them as Standards A, B, and C, respectively.

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

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

NOTE 2—The accuracy of this test method is dependent upon the accuracy of the test methods used to certify the carbon concentration in the calibration primary standard reference materials, as well as upon their homogeneity.

16.2 *Adjustment of Response of Measurement System:*

16.2.1 Transfer 1.0 g of Standard B, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible.

16.2.2 Proceed as directed in 17.1.2 and 17.1.3.

16.2.3 Repeat 16.2.1 and 16.2.2 until the absence of drift is indicated. Adjust the signal to provide a reading within ± 0.003 of the certified percent carbon value for the primary standard reference material.

16.3 *Determination of Blank Reading—Range I:*

16.3.1 Transfer 1.0 g of Standard A, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, into a preburned crucible.

16.3.2 Proceed as directed in 17.1.2 and 17.1.3.

16.3.3 Repeat 16.3.1 and 16.3.2 a sufficient number of times to establish that low (less than 0.020 mg of carbon) and consistent (± 0.002 mg of carbon) readings are obtained. Blank values are equal to the total result of the accelerator and Standard A minus the certified value for the primary standard reference material.

16.3.4 Record the average value of at least three blank determinations.

16.3.5 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 16.3.1-16.3.4.

16.3.6 Enter the average blank value in the analyzer (Note 3); refer to manufacturer's instructions. This mechanism will electronically compensate for the blank value.

NOTE 3—If the unit does not have this function, the blank value must be subtracted from the total result prior to any calculation.

16.4 *Determination of Blank Reading—Range II*—Proceed as directed in 16.3.

16.5 *Determination of Blank Reading—Range III:*

16.5.1 Transfer 0.5 g of Standard A, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible.

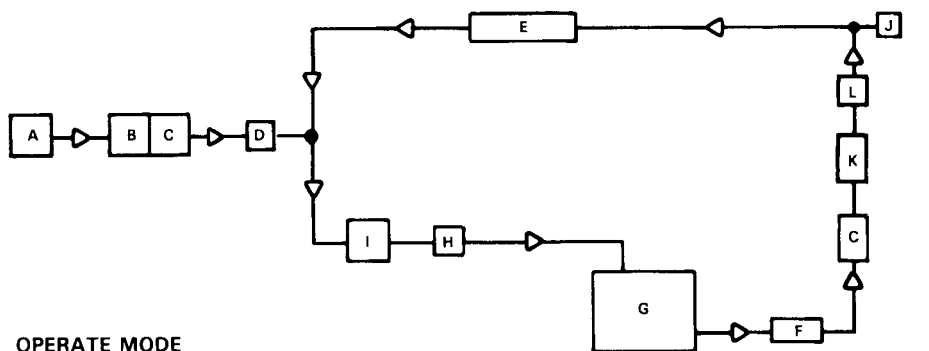
16.5.2 Proceed as directed in 16.3.2-16.3.6.

16.6 *Calibration—Range I (0.005 to 0.10 % carbon):*

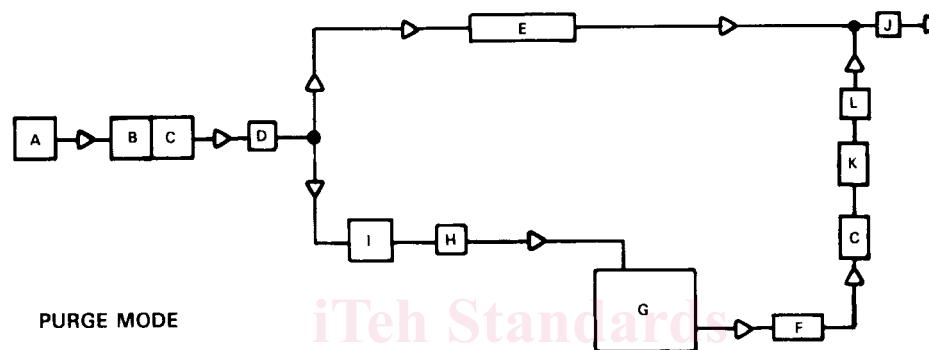
16.6.1 Weigh four 1.0 g specimens of Standard C, to the nearest 1 mg, the place in preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

16.6.2 Follow the calibration procedure recommended by the manufacturer. Use Standard C as the primary calibration standard and analyze at least three specimens to determine the calibration slope. Treat each specimen, as directed in 17.1.2

E 1019



OPERATE MODE



PURGE MODE

- | | |
|-------------------------------------|-----------------------------------|
| A—Oxygen Cylinder | G—Furnace |
| B—Sodium Hydroxide Impregnated Clay | H—Pump |
| C—Magnesium Perchlorate | I—Flow Meter |
| D—Press Regulator | J—Exhaust |
| E—IR Cell/Readout | K—CO to CO ₂ Converter |
| F—Dust Trap | L—SO ₃ Trap |

FIG. 4 Infrared Absorption Method C—Closed Loop

and 17.1.3, before proceeding to the next one.

16.6.3 Confirm the calibration by analyzing Standard C following the calibration procedure. The value should be within the allowable limits of the certified value for the primary standard reference material. If not, repeat 16.6.1 and 16.6.2.

16.6.4 Weigh at least two 1.0 g specimens of Standard B, weighed to the nearest 1 mg, and transfer them to preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

16.6.5 Treat each specimen as directed in 17.1.2 and 17.1.3 before proceeding to the next one.

16.6.6 Record the results of 16.6.4 and 16.6.5 and compare them to the certified carbon value of Standard B. The results should be within the allowable limits of the primary standard reference material. If not, refer to the manufacturer's instructions for checking the linearity of the system.

NOTE 4—Repeat the calibration when: (1) a different lot of crucibles is used, (2) a different lot of accelerator is used, (3) the system has not been in use for 1 h, and (4) the oxygen supply has been changed.

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

16.7.1 Proceed as directed in 16.6.1-16.6.3, using Standard CC.

16.7.2 Proceed as directed in 16.6.4-16.6.6, using Standard BB.

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

16.8.1 Weigh four 0.5 g specimens of Standard CCC, to the nearest 1 mg, and place in preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

16.8.2 Follow the calibration procedure recommended by the manufacturer. Use Standard CCC as the primary calibration standard and analyze at least three specimens to determine the calibration slope. Treat each specimen, as directed in 17.1.2 and 17.1.3, before proceeding to the next one.

16.8.3 Confirm the calibration by analyzing Standard CCC following the calibration procedure. The value should be within the allowable limits of the certified value for the primary standard reference material. If not, repeat 16.8.1 and 16.8.2.

16.8.4 Weigh at least two 0.5 g specimens of Standard BBB, weighed to the nearest 1 mg, and transfer to preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

16.8.5 Treat each specimen as described in 17.1.2 and 17.1.3 before proceeding to the next one.

16.8.6 Record the results of 16.8.4 and 16.8.5 and compare to the certified carbon value of the Standard BBB. The results should be within the allowable limits of the primary standard reference material. If not, refer to manufacturer's instructions for checking the linearity of the analyzer (Note 4).

17. Procedure

17.1 Procedure—Range I:

17.1.1 Stabilize the furnace and analyzer as directed in Section 14. Transfer 1.0 g of specimen, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible. (See 12.2.)

17.1.2 Place the crucible on the furnace pedestal and raise the pedestal into position. Use crucible tongs to handle the preburned crucibles.

17.1.3 Start the analysis cycle; refer to the manufacturer's recommended procedure regarding entry of specimen weight and blank value.

17.2 Procedure—Range II—Proceed as directed in 17.1.

17.3 Procedure—Range III—Proceed as directed in 17.1, using a 0.5 g specimen.

18. Calculation

18.1 Since most commercially available instruments calculate percent concentrations directly, including corrections for blank and sample weight, calculations by the analyst are not required.

NOTE 5—If the analyzer does not compensate for blank and sample weight values, then use the following formula:

$$\text{Carbon, \%} = [(A - B) \times C/D] \quad (1)$$

where:

A = DVM reading for specimen,

B = DVM reading for blank,

C = weight compensator setting, and

D = specimen weight, g.

19. Precision and Bias ⁷

19.1 Precision—Nine laboratories cooperated in testing this method and obtained the data summarized in Tables 1-3. Testing was performed in compliance with Practice E 173.

19.2 Bias—No information on the accuracy of this test method is known. The accuracy of this test method may be judged, however, by comparing accepted reference values with the corresponding arithmetic average obtained by interlaboratory testing.

SULFUR BY THE COMBUSTION-IRFRA-RED ABSORPTION METHOD (POTASSIUM SULFATE CALIBRATION)

20. Scope

20.1 This test method covers the determination of sulfur in

⁷ Supporting data are available from ASTM Headquarters. Request RR:E03-1038.

the range of 0.001 to 0.01 %. As written, this test method is not applicable to cast iron samples.

21. Summary of Test Method

21.1 The sample is combusted in a stream of oxygen that converts the sulfur in the sample to sulfur dioxide. The sulfur is then determined by infrared absorption.

21.1.1 *Infrared Absorption Method A*—Sulfur dioxide (SO₂) absorbs infrared (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 SO₂ 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 SO₂, is monitored and measured over a period of time. Refer to Fig. 5.

21.1.2 *Infrared Absorption Method B*—The combustion is performed in a closed loop where SO₂ is detected in an infrared cell. The SO₂ 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 SO₂ 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 SO₂, is monitored and measured over a period of time. Refer to Fig. 6.

21.1.3 *Infrared Absorption Method C*—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 SO₂ with its oxygen gas carrier 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 SO₂ 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 SO₂. Total SO₂ is monitored and measured over a period of time. Refer to Fig. 7.

22. Interferences

22.1 The elements ordinarily present do not interfere.

23. Apparatus

23.1 *Combustion and Measurement Apparatus*—See Figs. 5-7.

TABLE 1 Statistical Information—Carbon, Range I

Test Specimen	Carbon Found, %	Repeatability (R ₁ , E 173)	Reproducibility (R ₂ , E 173)
1. Electrolytic iron (NBS 365, 0.0068 C)	0.007	0.002	0.003
2. Bessemer carbon steel (NBS 8j, 0.081 C)	0.080	0.003	0.006
3. Type 304L stainless steel 18Cr-8Ni (NBS 101f, 0.014 C)	0.014	0.002	0.004
4. Type 446 stainless steel 26Cr (NBS 367, 0.093 C)	0.094	0.003	0.004
5. Nickel steel 36Ni (NBS 126b, 0.090 C)	0.092	0.003	0.004
6. Waspaloy 57Ni-20Cr-14Co-4Mo (NBS 349, 0.080 C)	0.078	0.003	0.004
7. Silicon steel (NBS 131a, 0.004 C)	0.004	0.002	0.002
8. High temperature alloy A286 26Ni-15Cr (NBS 348, 0.044 C)	0.046	0.003	0.004

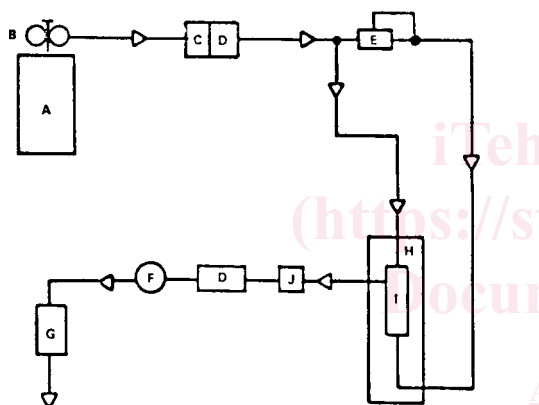
E 1019

TABLE 2 Statistical Information—Carbon, Range II

Test Specimen	Carbon Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
1. Basic open hearth steel (NBS 11h, 0.200 C)	0.201	0.006	0.010
2. Basic open hearth carbon steel (NBS 337, 1.07 C)	1.087	0.039	0.053
3. Low alloy electric furnace steel (NBS 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 (NBS 153a, 0.902 C)	0.905	0.023	0.027
6. Type 416 stainless steel (NBS 133b, 0.128 C)	0.126	0.005	0.013
7. Low alloy steel 1Cr (NBS 163, 0.933 C)	0.934	0.016	0.020

TABLE 3 Statistical Information—Carbon, Range III

Test Specimen	Carbon Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
1. Tool steel (CISRI 150, 1.56 C)	1.550	0.027	0.049
2. Low alloy electric furnace steel (NBS 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



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

FIG. 5 Infrared Absorption Method A

23.2 *Crucibles*—Use crucibles recommended by the manufacturer of the instrument, or equivalent, and preheat in a suitable furnace for not less than 1 h at more than 1100°C. Remove from furnace and cool in air for 90 ± 15 s before use. Preheated crucibles may also be stored in a desiccator prior to use.

23.3 *Micropipet*, (50 µL).

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

23.5 *Tin Capsules*—Approximate dimensions: diameter 6 mm, length 20 mm. Wash twice with acetone and dry at 90°C for 4 h prior to use.

24. Reagents

24.1 *Acetone*—The residue after evaporation must be <0.0005 %.

24.2 *Iron* (purity, 99.8 % minimum)—Must be free of sulfur or of low known sulfur content.

24.3 *Oxygen, Ultra High Purity* (purity: 99.95 % min)—Other grades of oxygen may be used if low and consistent blank readings are obtained, or the oxygen may be purified as described in Practices E 50.

24.4 *Potassium Sulfate* (K₂SO₄)—Dry 20 g of K₂SO₄ at 105 to 110°C for 1 h to a constant weight. Cool in a desiccator.

24.5 *Tungsten Accelerator (Low Sulfur)*, 12 to 20-mesh.

NOTE 6—The accelerator should contain no more than 0.001 % sulfur. 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 that heats the sample.

25. Preparation of Apparatus

25.1 Assemble the apparatus as recommended by the manufacturer.

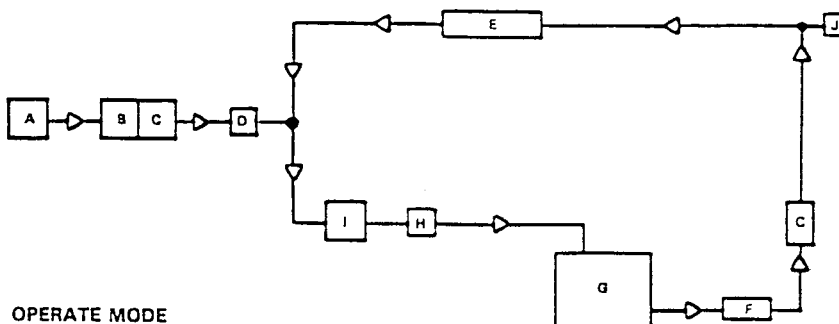
25.2 Test the furnace and analyzer to ensure the absence of leaks, and make the required electrical power connections. Prepare the analyzer for operation according to manufacturer's instructions. Make a minimum of two determinations using the specimen and accelerator as directed in 28.2 and 28.3 before attempting to calibrate the system or determine the blank.

26. Sample Preparation

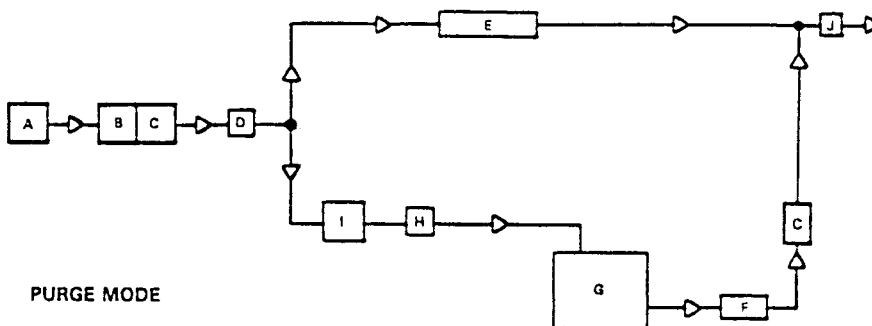
26.1 The sample should be uniform in size but not finer than 40 mesh.

26.2 Wash in acetone and dry at 70 to 100°C.

E 1019



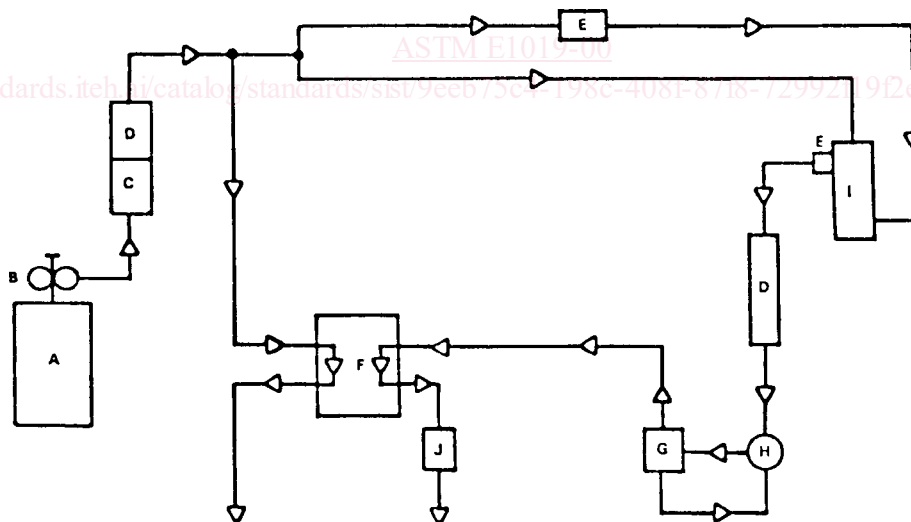
OPERATE MODE



PURGE MODE

- | | |
|-------------------------------------|--------------|
| A—Oxygen Cylinder | F—Dust Trap |
| B—Sodium Hydroxide Impregnated Clay | G—Furnace |
| C—Magnesium Perchlorate | H—Pump |
| D—Press Regulator | I—Flow Meter |
| E—IR Cell/Readout | J—Exhaust |

FIG. 6 Infrared Absorption Method B



- | | |
|-------------------------------------|--------------------------|
| A—Oxygen Cylinder | F—IR Detector/Readout |
| B—Two Stage Regulator | G—Orifice |
| C—Sodium Hydroxide Impregnated Clay | H—Pressure Regulator |
| D—Magnesium Perchlorate | I—Combustion Chamber |
| E—Dust Trap | J—Measure Flow Rotameter |

FIG. 7 Infrared Absorption Method C

27. Calibration

27.1 Calibration Standards:

27.1.1 Weigh to the nearest 0.0001 g the following masses of K_2SO_4 :

Sulfur Solution	K ₂ SO ₄ (g)	Sulfur Concentration (mg/mL)
A	0.1087	0.2
B	0.2718	0.5
C	0.5435	1.0
D	1.0870	2.0
H	0.0000	0.0

27.1.2 Dissolve each mass of K₂SO₄ with 50 mL of water in five 100-mL beakers.

27.1.3 Transfer quantitatively each solution to five 100-mL volumetric flasks. Dilute to volume and mix.

27.1.4 Using a pipet, transfer 50 µL of the following sulfur solutions to individual tin capsules. Prepare the number of replicates indicated and then proceed as directed in 27.1.5.

Sulfur Solution	S (µg)	S, % in the Test Portion	Number of Replicates
H	0	0.0000	5
A	10	0.0010	2
B	25	0.0025	5
C	50	0.0050	2
D	100	0.0100	5

27.1.5 Dry the tin capsules slowly at about 90°C to full-dryness, and cool in a desiccator. Compress the top part of the tin capsule before use.

27.2 Adjustment of Response of Measurement System:

27.2.1 Transfer one dried capsule of sulfur solution B to a preburned crucible. Add 1.0 g of pure iron, weighed to the nearest 5 mg, and 1.5 g of tungsten, weighed to the nearest 5 mg, to the crucible. Proceed as directed in 28.2 and 28.3.

27.2.2 Repeat 27.2.1 until the absence of drift is indicated. (Prepare more capsules of sulfur solution B if necessary.) Adjust the signal to provide a reading of 0.0025 ± 0.0003 % sulfur.

27.3 Determination of Blank Reading:

27.3.1 Transfer one dried capsule of sulfur solution H to a preburned crucible. Add 1.0 g of pure iron, weighed to the nearest 5 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to the crucible. Proceed as directed in 28.2 and 28.3.

27.3.2 Repeat 27.3.1 a sufficient number of times to establish that low (less than 5 µg of sulfur) and consistent (±2 µg of sulfur) readings are obtained. Blank values are equal to the total result of accelerator, iron, and capsule of solution H minus the sulfur contained in 1.0 g of iron.

27.3.3 Record the average value of at least three blank determinations.

27.3.4 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 27.3.1-27.3.3. Prepare more capsules of sulfur solution H if necessary.

27.3.5 Enter the average blank value in the analyzer (Note 7). Refer to manufacturer's instructions. This mechanism will electronically compensate for the blank value.

NOTE 7—If the unit does not have this function, the blank value must be subtracted from the total result prior to any calculation.

27.4 Calibration:

27.4.1 Transfer four dried capsules of sulfur solution D to preburned crucibles. Add 1.0 g of pure iron, weighed to the nearest 5 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to each crucible.

27.4.2 Follow calibration procedure recommended by the

manufacturer using dried capsules of sulfur solution D as the primary standard, analyzing at least three specimens to determine the calibration slope. Treat each capsule as directed in 28.2 and 28.3 before proceeding to the next one.

27.4.3 Confirm the calibration by analyzing a capsule of sulfur solution D after the calibration procedure. The value should be 0.0100 ± 0.0005 % sulfur. If not, repeat 27.4.1 and 27.4.2.

27.4.4 Transfer two dried capsules of sulfur solution A, B, and C to preburned crucibles. Add 1.0 g of pure iron, weighed to the nearest 5 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to each crucible.

27.4.5 Treat each capsule as directed in 28.2 and 28.3 before proceeding to the next one.

27.4.6 Record the results of 27.4.5 and compare them to the theoretical sulfur values of solution A, B, and C. If they are not within 0.0003 % of the theoretical concentrations of sulfur in the test portions, refer to the manufacturer's instructions for checking the linearity of the system.

NOTE 8—Repeat the calibration when:

- (1) a different lot of crucibles is used,
- (2) a different lot of accelerator is used,
- (3) the system has not been in use for 1 h, and
- (4) the oxygen supply has been changed.

28. Procedure

28.1 Stabilize the furnace and analyzer as directed in Section 25. Transfer 1.0 g of specimen, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible. (See 23.2.)

28.2 Place the crucible on the furnace pedestal and raise the pedestal into position. Use crucible tongs to handle the preburned crucibles.

28.3 Start the analysis cycle; refer to manufacturer's recommended procedure regarding entry of specimen weight and blank value.

29. Calculation

29.1 Since most commercially available instruments calculate percent concentrations directly, including corrections for blank and sample weight, calculations by the analyst are not required.

NOTE 9—If the analyzer does not compensate for blank and sample weight values, then use the following formula:

$$\text{Sulfur, \%} = \frac{(A - B) \times C}{D} \quad (2)$$

where:

- A = DVM reading for specimen,
- B = DVM reading for blank,
- C = weight compensator setting, and
- D = specimen weight, g.

30. Precision and Bias ⁸

30.1 *Precision*—Twenty-five laboratories participated in testing this method under the auspices of WG-3 of ISO

⁸ Supporting data are available from ASTM Headquarters. Request RR: E03-1041.

Committee TC 17/SC 1 and obtained the data summarized in Table 4. All testing meets the requirements of Practice E 173.

30.2 *Bias*—No information on the accuracy of this test method is known. The accuracy of this test method may be judged, however, by comparing accepted reference values with the corresponding arithmetic average obtained by interlaboratory testing.

NITROGEN BY THE INERT GAS FUSION THERMAL CONDUCTIVITY METHOD

31. Scope

31.1 This test method covers the determination of nitrogen (*N*) in concentrations from 0.001 to 0.2 % (Note 10).

NOTE 10—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 in accordance with Practice E 173. However, recognizing that commercial nitrogen determinators are capable of handling higher concentrations, 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.

32. Summary of Test Method

32.1 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 carbon monoxide and is finally measured in a thermal conductivity cell. Refer to Figs. 8-11.

32.2 This test method is written for use with commercial analyzers equipped to carry out the above operations automatically and calibrated using standard samples of known nitrogen content.

33. Interferences

33.1 The elements ordinarily present do not interfere.

34. Apparatus

34.1 *Fusion and Measurement Apparatus*— See Fig. 8.

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

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

35. Reagents

35.1 *Acetone*, low-residue.

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

35.3 *Magnesium Perchlorate*, (known commercially as *Anhydrone*).

35.4 *Sodium Hydroxide*, on clay, (known commercially as *Ascarite II*).

36. Preparation of Apparatus

36.1 Assemble the apparatus as recommended by the manufacturer.

36.2 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 in accordance with the manufacturer's instructions. Make a minimum of two determinations using a specimen as directed in 39.2.1 or 39.2.2 before attempting to calibrate the system or to determine the blank.

37. Sample Preparation

37.1 Use chips, drillings, or solid form specimens prepared as directed in 37.2 or 37.3 (Note 11).

NOTE 11—Size all specimens to permit free introduction through the loading device of the equipment or directly into the graphite crucible.

37.2 If a solid specimen is used, cut it with a water-cooled abrasive cut-off wheel or by another means that will prevent overheating. Using a file, abrade the surface of the specimen to be used for analysis to remove oxides and other impurities, rinse in acetone, air dry, and weigh.

37.3 If drillings or chips are used, prepare as directed in Practice E 1806.

38. Calibration

38.1 *Calibration Standards:*

38.1.1 For Range I, 0.0005 to 0.10 % nitrogen, select five certified reference materials containing approximately 0.002, 0.01, 0.03, 0.05, and 0.10 % nitrogen and designate them as standards, A, B, C, D, and E, respectively (Note 12).

38.1.2 For Range II, 0.10 to 0.50 % nitrogen, select three certified reference materials containing approximately 0.10, 0.30, and 0.50 % nitrogen and designate them as standards AA, BB, and CC, respectively (Note 12).

NOTE 12—The accuracy of the test method is largely dependent upon the accuracy of the methods used to certify the nitrogen concentration in the calibration of certified reference materials, as well as upon their homogeneity. Thus, calibration specimens should be NBS Standard Reference Materials (SRM) or other certified reference materials.

38.2 *Adjustment of Response of Measurement System:*

38.2.1 Prepare a 1.0 g specimen of standard C weighed to the nearest 1 mg as directed in 37.2 or 37.3.

38.2.2 Proceed as directed in 39.2.1 or 39.2.2.

38.2.3 Repeat 38.2.1 and 38.2.2 and adjust as recommended by the manufacturer until the absence of drift is indicated.

38.3 *Determination of Blank Reading—Ranges I and II:*

TABLE 4 Statistical Information—Sulfur

Test Specimen	Sulfur Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
Low alloy steel (JK 24, 0.0010 S)	0.0010	0.00045	0.00051
Stainless steel (NBS 348, 0.0020 S)	0.00198	0.0005	0.00064
Silicon steel (IRSID 114-1, 0.0037 S)	0.00322	0.00051	0.0007
Plain carbon steel (JSS 240-8, 0.0060 S)	0.00549	0.00055	0.00099
Stainless steel (JSS 652-7, 0.0064 S)	0.00615	0.00084	0.00087