



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 Reference Materials)	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:

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 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.

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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 6.*

2. Referenced Documents³

2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
- E 1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals

3. Terminology

3.1 For definition of terms used in this test method, refer to Terminology E 135.

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

³ 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.

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.

6. Hazards

6.1 For hazards to be observed in the use of certain reagents in this test method, refer to Practices E 50.

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 the materials, refer to those parts of Practice E 1806.

8. Rounding Calculated Values

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

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E 173 (discontinued 1997). The Reproducibility R_2 of E 173 corresponds to the Reproducibility Index R of E 1601. The Repeatability R_1 of E 173 corresponds to the Repeatability Index r of E 1601.

TOTAL CARBON BY THE COMBUSTION INSTRUMENTAL MEASUREMENT METHOD

10. Scope

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

11. Summary of Test Method

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

11.1.1 *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.

11.1.2 *Infrared (IR) Absorption, Method A*—The amount of carbon dioxide is measured by infrared (IR) absorption. Carbon dioxide (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 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.

11.1.3 *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 CO_2 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 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.

11.1.4 *Infrared (IR) Absorption, Method C, Closed Loop*—The combustion is performed in a closed loop, where 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 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 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.

11.2 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.

12. Interferences

12.1 The elements ordinarily present do not interfere.

13. Apparatus

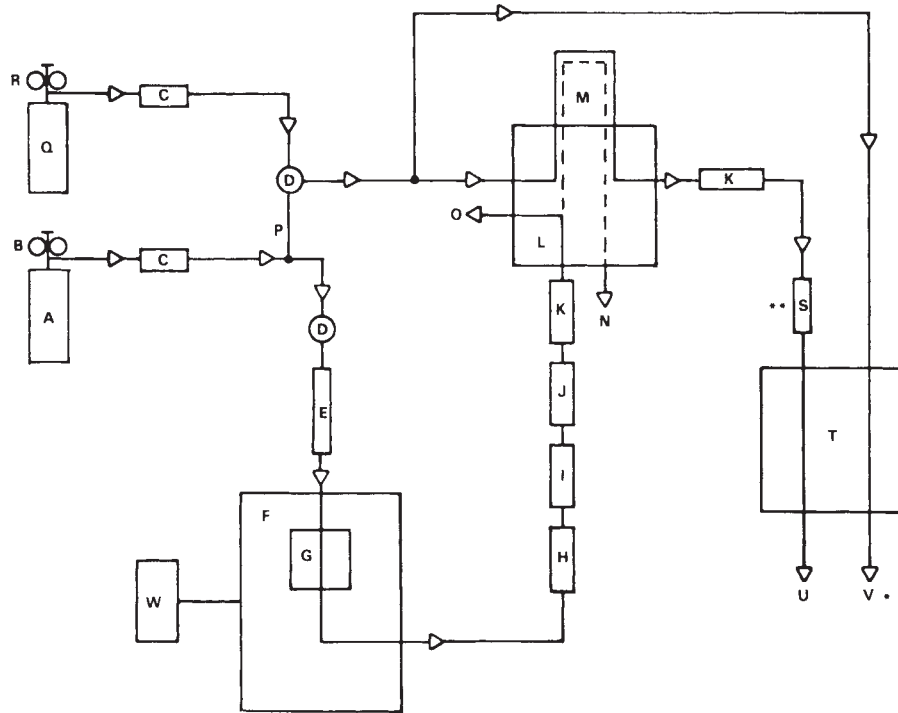
13.1 *Combustion and Measurement Apparatus*—See Figs. 1-4.

13.2 *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.

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,



- | | |
|---|---|
| <p>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</p> | <p>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</p> |
|---|---|

* 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

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 must be <0.0005 %.

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

14.4 *Magnesium Perchlorate*, (known commercially as *Anhydron*).

14.5 *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.

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

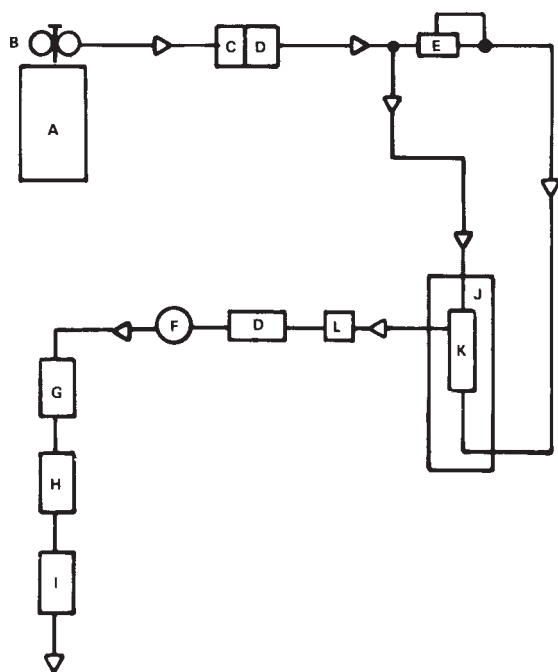
14.7 *Sodium Hydroxide*, on clay (known commercially as *Ascarite II*).

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

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

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., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

NOTE 1—The accelerator should contain no more than 0.001 % carbon. If necessary, wash three times with acetone by decantation to remove



- | | |
|-------------------------------------|-----------------------------------|
| A—Oxygen Cylinder | G—CO-CO ₂ Converter |
| B—Two Stage Regulator | H—SO ₃ Trap |
| C—Sodium Hydroxide Impregnated Clay | I—CO ₂ IR Cell/Readout |
| D—Magnesium Perchlorate | J—Induction Furnace |
| E—Regulator | K—Combustion Area |
| F—Flow Controller | L—Dust Trap |

FIG. 2 Infrared Absorption Method A

organic contaminants and dry at room temperature. The mesh size is critical to the inductive coupling which heats the sample.

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 according to the manufacturer's instructions. Make a minimum of two determinations using the specimen and accelerator as directed in 18.1.2 and 18.1.3 before attempting to calibrate the system or determine the blank.

16. Sample Preparation

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

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

17. Calibration

17.1 *Calibration Reference Materials* (Note 2):

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.

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 must be considered as well, if it was not included in the derivation of the published uncertainty values.

17.2 Adjustment of Response of Measurement System:

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

17.2.2 Proceed as directed in 18.1.2 and 18.1.3.

17.2.3 Repeat 17.2.1 and 17.2.2 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 certified reference material.

17.3 Determination of Blank Reading—Range I:

17.3.1 Transfer 1.0 g of Calibrant A, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, into a preburned 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 % of carbon) and consistent (± 0.0002 % of carbon) readings are obtained. Blank values are equal to the total result of the accelerator and Calibrant A minus the certified value for the certified reference material.

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

17.3.5 If the blank readings are too high or inconsistent, 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 (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.

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 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned 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, the place in preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

17.6.2 Follow the calibration procedure recommended by the manufacturer. Use Calibrant C as the primary calibrant 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.

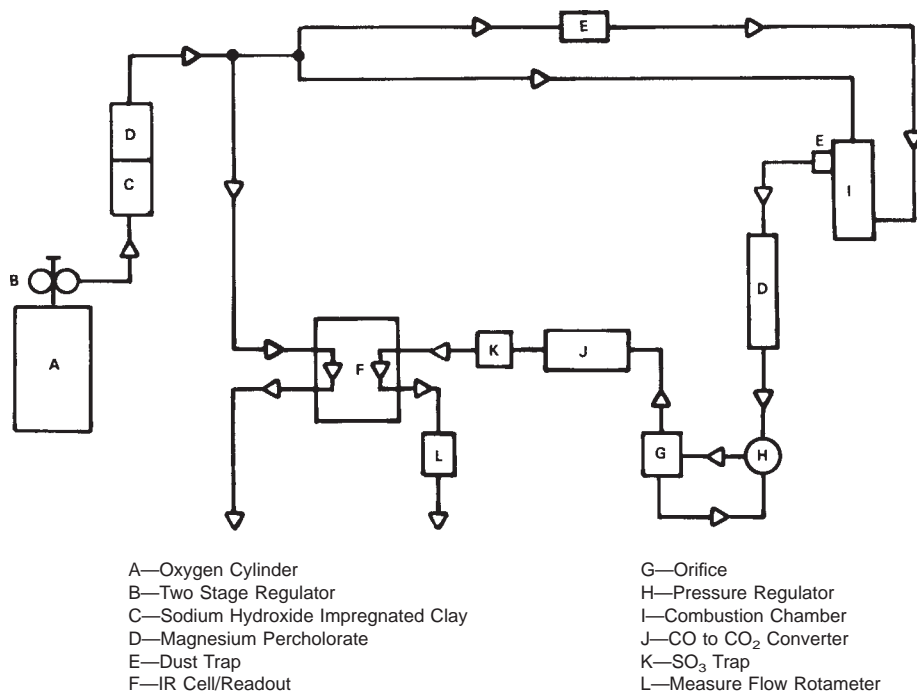


FIG. 3 Infrared Absorption Method B

17.6.3 Confirm the calibration by analyzing Calibrant C following the calibration procedure. The value should be within the allowable limits of the certified value for the certified reference material. If not, repeat 17.6.1 and 17.6.2.

17.6.4 Weigh at least two 1.0 g specimens of Calibrant 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.

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 results should be within the allowable limits of the certified 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.

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 preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

17.8.2 Follow the calibration procedure recommended by the manufacturer. Use Calibrant CCC as the certified reference material 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.

17.8.3 Confirm the calibration by analyzing Calibrant CCC following the calibration procedure. The value should be within the allowable limits of the certified value for the certified reference material. 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 preburned crucibles. To each, add 1.5 g of accelerator, weighed to the nearest 5 mg.

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 the Calibrant BBB. The results should be within the allowable limits of the certified reference material. If not, refer to manufacturer's instructions for checking the linearity of the analyzer (Note 4).

18. Procedure

18.1 Procedure—Range I:

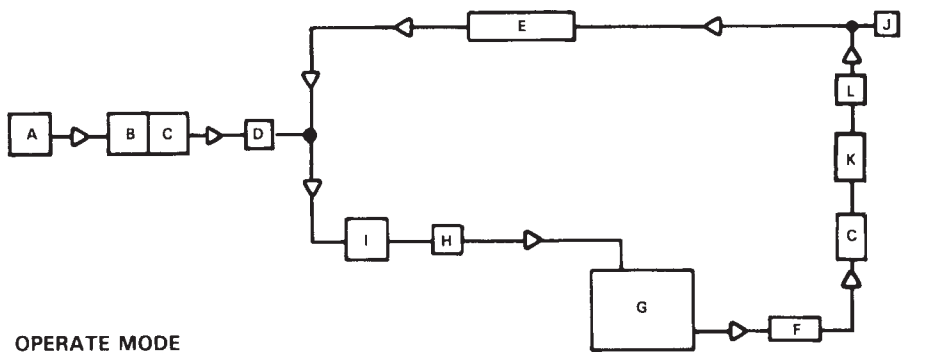
18.1.1 Stabilize the furnace and analyzer as directed in Section 15. Transfer 1.0 g of specimen, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible. (See 13.2.)

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

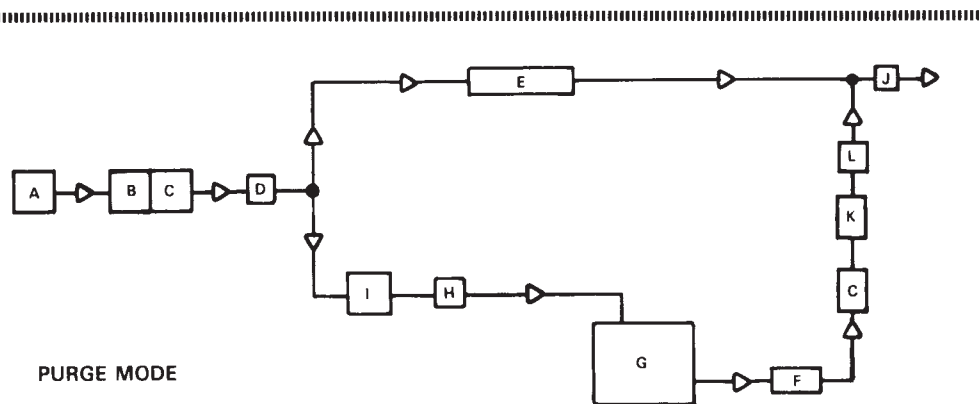
18.1.3 Start the analysis cycle; refer to the manufacturer's recommended procedure regarding entry of specimen weight and blank value.

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.



OPERATE MODE



PURGE MODE

- A—Oxygen Cylinder
- B—Sodium Hydroxide Impregnated Clay
- C—Magnesium Perchlorate
- D—Press Regulator
- E—IR Cell/Readout
- F—Dust Trap
- G—Furnace
- H—Pump
- I—Flow Meter
- J—Exhaust
- K—CO to CO₂ Converter
- L—SO₃ Trap

FIG. 4 Infrared Absorption Method C—Closed Loop

TABLE 1 Statistical Information—Carbon, Range I

Test Specimen	Carbon Found, %	Repeatability (<i>R</i> ₁ , E 173)	Reproducibility (<i>R</i> 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 (SRM 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

TABLE 2 Statistical Information—Carbon, Range II

Test Specimen	Carbon Found, %	Repeatability (<i>R</i> ₁ , E 173)	Reproducibility (<i>R</i> ₂ , E 173)
1. Basic open hearth steel (SRM 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 (SRM 133b, 0.128 C)	0.126	0.005	0.013
7. Low alloy steel 1Cr (SRM 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

19. Calculation

19.1 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.

20. Precision and Bias⁵

20.1 *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.

20.2 *Bias*—Information on the accuracy of this test method is incomplete at this time. The user is cautioned to verify by the use of certified reference materials, if available, that the accuracy of this test method is adequate for the contemplated use.

SULFUR BY THE COMBUSTION-IR INFRARED ABSORPTION 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

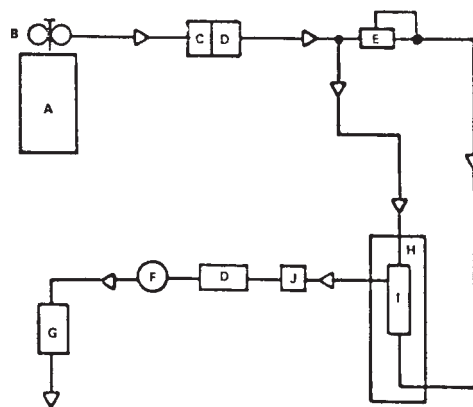
22.1 The sample is combusted in a stream of oxygen that converts the sulfur in the sample to sulfur dioxide. The sulfur is then determined by infrared absorption.

22.1.1 *Infrared Absorption Method A*—Sulfur dioxide (SO_2) 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 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 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 monitored and measured over a period of time. Refer to Fig. 6.

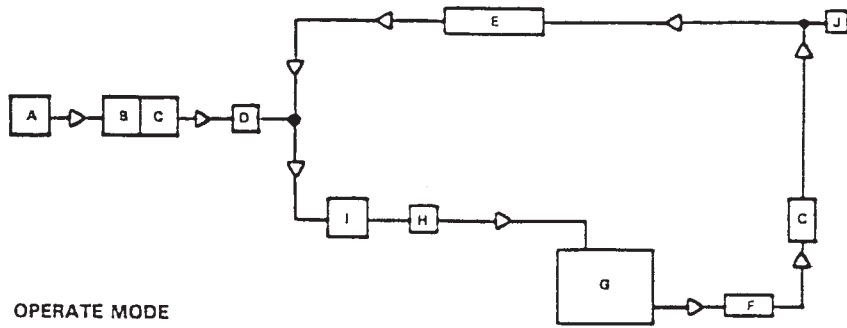
22.1.3 *Infrared Absorption Method C*—The detector consists of an IR energy source, a separate measure chamber and reference chamber, and a diaphragm acting as one plate of a parallel plate capacitor. During specimen combustion, the flow of SO_2 with its oxygen gas carrier is routed through the measure 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 SO_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 SO_2 . Total SO_2 is monitored and measured over a period of time. Refer to Fig. 7.



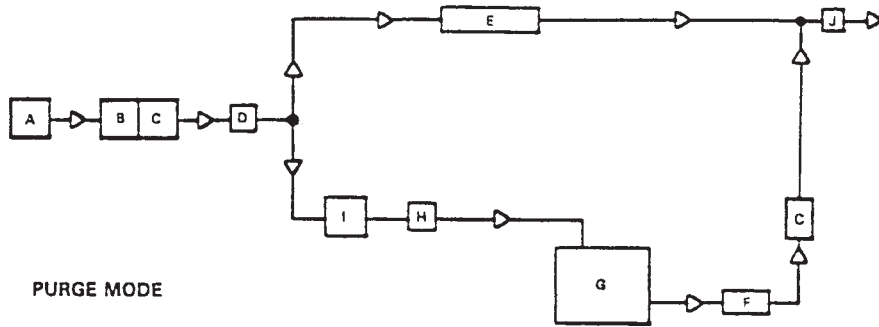
A—Oxygen Cylinder
 B—Two Stage Regulator
 C—Sodium Hydroxide Impregnated Clay
 D—Magnesium Perchlorate
 E—Regulator
 F—Flow Controller
 G—IR Cell/Readout
 H—Induction Furnace
 I—Combustion Area
 J—Dust Trap

FIG. 5 Infrared Absorption Method A

⁵ Supporting data are available from ASTM International Headquarters. Request RR:E03-1038.



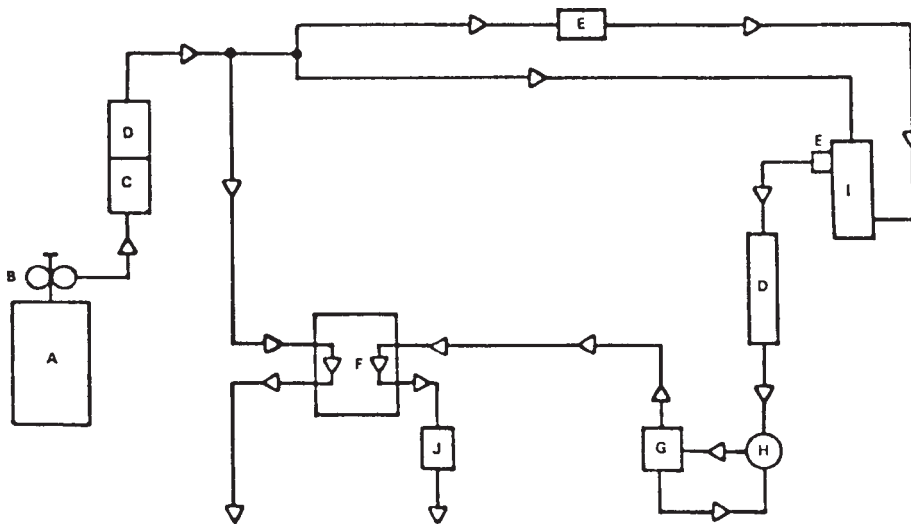
OPERATE MODE



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

23. Interferences

23.1 The elements ordinarily present do not interfere.

24. Apparatus

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

24.2 *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.

24.3 *Micropipet*, (50 μ L).

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

24.5 *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.

25. Reagents

25.1 *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.

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

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

25.4 *Magnesium Perchlorate*, (known commercially as *Anhydron*).

25.5 *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.

25.6 *Potassium Sulfate* (K_2SO_4)—Dry 20 g of K_2SO_4 at 105 to 110°C for 1 h to a constant weight. Cool in a desiccator.

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

25.8 *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.

26. Preparation of Apparatus

26.1 Assemble the apparatus as recommended by the manufacturer.

26.2 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 29.2 and 29.3 before attempting to calibrate the system or determine the blank.

27. Sample Preparation

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

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

28. Calibration

28.1 *Calibration Reference Materials*:

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

Sulfur Solution	K_2SO_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

28.1.2 Dissolve each portion of K_2SO_4 in 50 mL of water in five 100-mL beakers.

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

28.1.4 Using a pipet, transfer 50 μ L of the following sulfur solutions to individual tin capsules. Prepare the number of replicates indicated and then proceed as directed in 28.1.5.

Sulfur Solution	S (μ g)	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

28.1.5 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.

28.2 *Adjustment of Response of Measurement System*:

28.2.1 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 29.2 and 29.3.

28.2.2 Repeat 28.2.1 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.

28.3 *Determination of Blank Reading*:

28.3.1 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 29.2 and 29.3.

28.3.2 Repeat 28.3.1 a sufficient number of times to establish that low (less than 5 μ g of sulfur) and consistent (± 2 μ g of sulfur) readings are obtained. Blank values are equal to the total result of accelerator, iron, and capsule of solution H.

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

28.3.4 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 28.3.1-28.3.3. Prepare more capsules of sulfur solution H if necessary.

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

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

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

28.4 Calibration:

28.4.1 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.

28.4.2 Follow calibration procedure recommended by the manufacturer using dried capsules of sulfur solution D as the primary calibrant, analyzing at least three specimens to determine the calibration slope. Treat each capsule as directed in 29.2 and 29.3 before proceeding to the next one.

28.4.3 Confirm the calibration by analyzing a capsule of sulfur solution D after the calibration procedure. The value should be 0.0100 % \pm 0.0005 % sulfur. If not, repeat 28.4.1 and 28.4.2.

28.4.4 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.

28.4.5 Treat each capsule as directed in 29.2 and 29.3 before proceeding to the next one.

28.4.6 Record the results of 28.4.5 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.

29. Procedure

29.1 Stabilize the furnace and analyzer as directed in Section 26. 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 24.2.)

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

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

NOTE 9—This procedure is for analysis of steel samples and a new blank must be determined using 1.5 g of accelerator only. Refer to section 62.3.

30. Calculation

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

NOTE 10—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.

31. Precision and Bias ⁶

31.1 *Precision*—Twenty-five laboratories participated in testing this method under the auspices of WG-3 of ISO Committee TC 17/SC 1 and obtained the data summarized in Table 4. All testing meets the requirements of Practice E 173.

31.2 *Bias*—Information on the accuracy of this test method is incomplete at this time. The user is cautioned to verify by the use of certified reference materials, if available, that the accuracy of this test method is adequate for the contemplated use.

NITROGEN BY THE INERT GAS FUSION THERMAL CONDUCTIVITY METHOD

32. Scope

32.1 This test method covers the determination of nitrogen (N) in concentrations from 0.0010 % to 0.2 % (Note 11).

NOTE 11—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.

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

⁶ Supporting data are available from ASTM International Headquarters. Request RR: E03-1041.

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.

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

34. Interferences

34.1 The elements ordinarily present do not interfere.

35. Apparatus

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

35.2 *Graphite Crucibles*—Use the size crucibles recommended by the manufacturer of the instrument. Crucibles must 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*, low-residue.

36.3 *Copper*.

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

36.5 *Magnesium Perchlorate*, (known commercially as *Anhydron*).

36.6 *Rare Earth Copper Oxide*, as recommended by the instrument manufacturer.

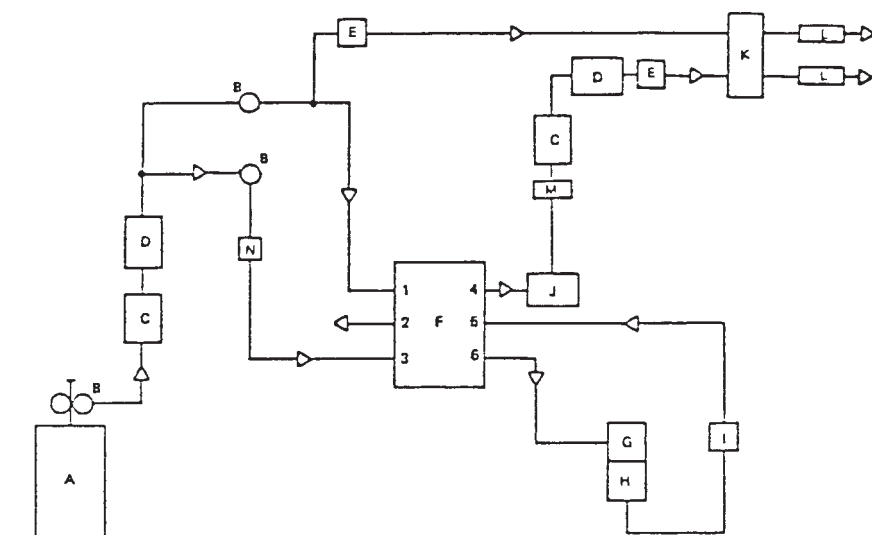
36.7 *Silica*, as recommended by the instrument manufacturer.

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

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 in accordance with the manufacturer's instructions. Make a minimum of two determinations using a specimen as directed in 40.2.1 or 40.2.2 before attempting to calibrate the system or to determine the blank.

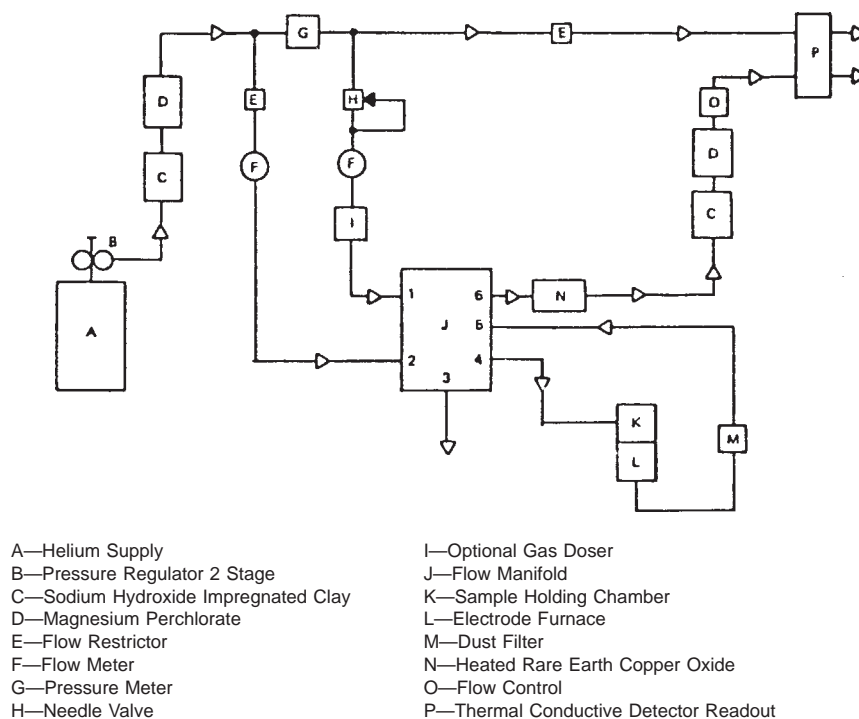


- A—Helium Supply
- B—Pressure Regulator 2 Stage
- C—Sodium Hydroxide Impregnated Clay
- D—Magnesium Perchlorate
- E—Flow Control
- F—Flow Manifold
- G—Sample Holding Chamber
- H—Electrode Furnace
- I—Dust Filter
- J—Heated Rare Earth Copper Oxide
- K—Thermal Conductive Detector/Readout
- L—Flow Rotameter
- 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. 8 Nitrogen Method A Flow Diagram



Manifold Porting

Crucible Degas Flow $\left\{ \begin{array}{l} 2 \text{ to } 4 \\ 5 \text{ to } 3 \\ 1 \text{ to } 6 \end{array} \right.$
 Fusion Flow $\left\{ \begin{array}{l} 1 \text{ to } 4 \\ 5 \text{ to } 6 \\ 2 \text{ \& } 3 \text{ off} \end{array} \right.$

FIG. 9 Nitrogen Method B Flow Diagram

38. Sample Preparation

38.1 Use chips, drillings, or solid form specimens prepared as directed in 38.2 or 38.3 (Note 12).

NOTE 12—Size all specimens to permit free introduction through the loading device of the equipment or directly into the graphite crucible.

38.2 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.

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

39. Calibration

39.1 Reference Materials:

39.1.1 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 Calibrants A, B, C, D, and E, respectively (Note 13).

39.1.2 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 Calibrants AA, BB, and CC, respectively (Note 13).

NOTE 13—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 must be considered as well, if it was not included in the derivation of the published uncertainty values.

39.2 Adjustment of Response of Measurement System:

39.2.1 Prepare a 1.0 g specimen of Calibrant C weighed to the nearest 1 mg as directed in 38.2 or 38.3.

39.2.2 Proceed as directed in 40.2.1 or 40.2.2.

39.2.3 Repeat 39.2.1 and 39.2.2 and adjust as recommended by the manufacturer until the absence of drift is indicated.

39.3 Determination of Blank Reading—Ranges I and II:

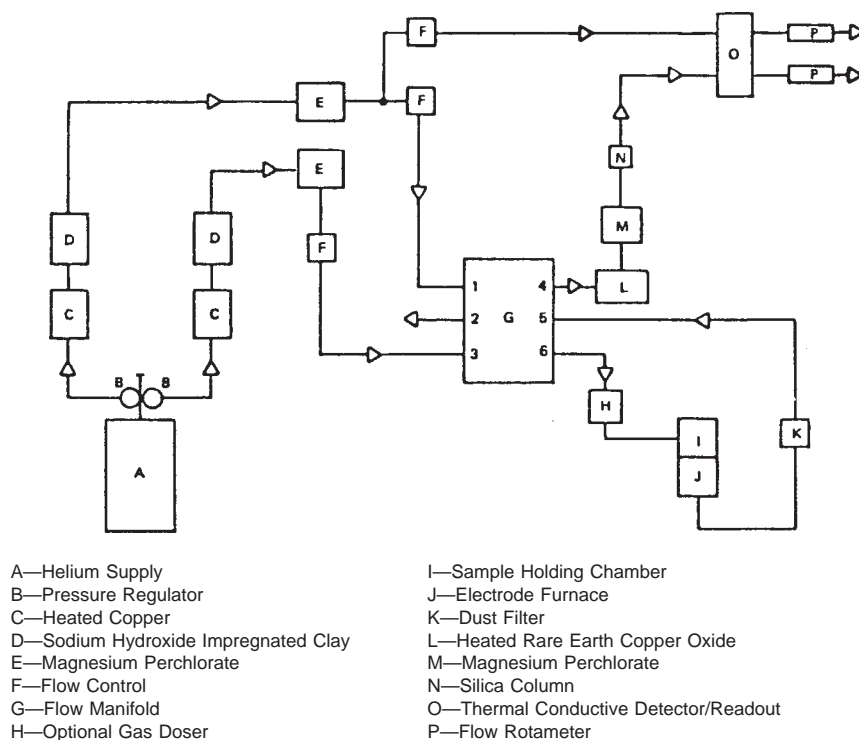
39.3.1 If the instrument is equipped with an electronic blank compensator, adjust to zero, and proceed with the determination of the blank value.

39.3.2 Make three blank determinations as directed in 40.2.1 or 40.2.2 with the sample omitted. Use a fresh crucible each time (Note 14).

NOTE 14—If the loading device is used to analyze the unknown or calibration sample, see Note 12.

39.3.3 If the blank values exceed 0.0003 % or a spread of three consecutive values exceeds 0.0003 %, then determine the cause, make necessary corrections, and repeat 39.3.1 and 39.3.2.

39.3.4 Record the average value of at least three blank readings.



Manifold Porting

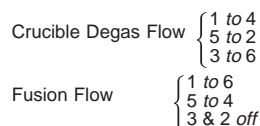


FIG. 10 Nitrogen/Oxygen Method A Flow Diagram

39.3.5 Enter the average blank value in the appropriate mechanism of the analyzer (Note 15); refer to manufacturer's instructions. This mechanism will electronically compensate for the blank value.

NOTE 15—If the unit does not have this function, the average blank value must be subtracted from the DVM readings for reference materials and specimens (Note 19).

39.4 Calibration Procedure—Range I:

39.4.1 Prepare four 1.0 g specimens of Calibrant D weighed to the nearest 1 mg, as directed in 38.2 or 38.3.

39.4.2 Follow the calibration procedure recommended by the manufacturer using Calibrant D as certified reference material. Analyze at least three reference material specimens to determine calibration slope. Treat each specimen as directed in 40.2.1 or 40.2.2 before proceeding to the next one.

39.4.3 Confirm the calibration by analyzing Calibrant D after calibration. The value should be within the allowable limits of the certified value. If not, repeat 39.4.1 and 39.4.2.

39.4.4 Prepare two 1.0 g specimens each of Calibrants A, B, C, and E weighed to the nearest 1 mg, as directed in 38.2 or 38.3.

39.4.5 Treat each specimen as directed in 40.2.1 or 40.2.2 before proceeding to the next one.

39.4.6 Record the results and compare them to the actual certified nitrogen values of the certified reference material.

They should be within the allowable limits of the respective certified reference material. If not, refer to the manufacturer's instructions for checking the linearity of the analyzer.

NOTE 16—Repeat the calibration when: (1) a different lot of crucibles is used, (2) the system has not been in use for 1 h, or (3) the carrier gas supply has been changed.

39.5 Calibration Procedure—Range II:

39.5.1 Proceed as directed in 39.4.1-39.4.3 using Calibrant CC.

39.5.2 Proceed as directed in 39.4.4-39.4.6 using Calibrants AA and BB (Note 16).

40. Procedure

40.1 Assemble the apparatus and condition it as directed in Section 37.

40.2 Procedure—Ranges I and II:

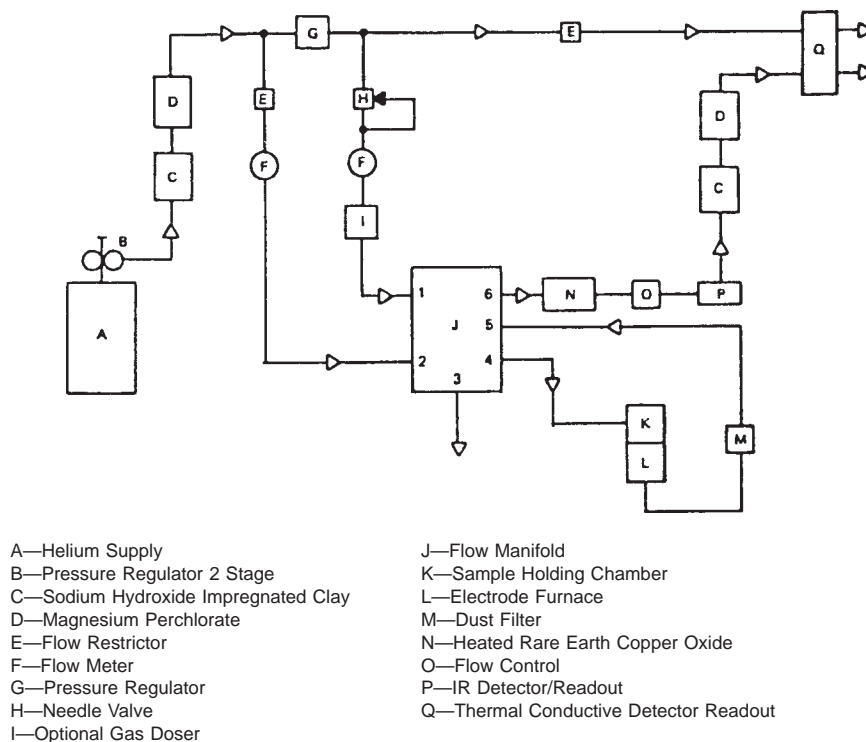
40.2.1 Manual Operation:

40.2.1.1 Set the analyzer to operate in the manual mode.

40.2.1.2 Place an empty crucible on the furnace lower electrode assembly and close the furnace.

40.2.1.3 Weigh a 1.0 g specimen to the nearest 1 mg and refer to the manufacturer's recommended procedure, including entry of sample weight.

40.2.1.4 Start the analysis cycle, referring to the manufacturer's recommended procedure (Note 17).



Manifold Porting

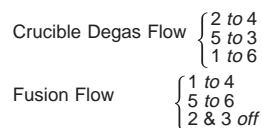


FIG. 11 Nitrogen/Oxygen Method B Flow Diagram

40.2.2 Automatic Operation:

40.2.2.1 Set the analyzer to operate in the automatic mode.

40.2.2.2 Place a 1.0 g specimen weighed to the nearest 1 mg in the loading device. Refer to the manufacturer's recommended procedure, including entry of sample weight.

40.2.2.3 Place an empty crucible on the furnace lower electrode assembly and close the furnace.

40.2.2.4 Start the analysis cycle, referring to the manufacturer's recommended procedure (Notes 17 and 18).

NOTE 17—If the samples are to be analyzed in the automatic mode, blank determination and calibration should also be performed in the automatic mode. If samples are to be analyzed in the manual mode, blank determination and calibration should be performed in the manual mode. Solid samples may be analyzed in either automatic or manual mode, but drillings or chips should be analyzed in the manual mode.

NOTE 18—The presence of nitrogen as refractory nitrides in the matrix may affect sample fusion and nitrogen evolution under standard operating conditions of the analyzer. It is therefore recommended that calibrants be of the same or similar composition as the samples to be analyzed.

41. Calculation

41.1 With most commercially available instruments the readings will be given directly in concentration (Note 19).

NOTE 19—If the analyzer does not compensate for blank and sample

weight values, then the equation is:

$$N \text{ nitrogen, \%} = \frac{(A - B) \times C}{D} \quad (3)$$

where:

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

42. Precision and Bias ⁷

42.1 Precision—Nine laboratories cooperated in testing this test method and obtained the precision data summarized in Tables 5-7.

42.2 Bias—The accuracy of this test method can be inferred from the data in Tables 5-7 by comparing the certified values for nitrogen with the average values obtained (Note 20).

NOTE 20—Although this test method was tested to only 0.22 %, most commercial instruments are believed to be capable of analyzing samples containing nitrogen up to 0.5 %.

⁷ Supporting data are available from ASTM International Headquarters. Request RR: E03-1051.

TABLE 5 Statistical Information—Nitrogen, Range I (chip)

Test Specimen	Nitrogen Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
No. 1 High purity iron (Euro CRM 088.1, 0.0009 % N)	0.0010	0.0005	0.0008
No. 2 High purity iron (JSS-001, 0.0017 % N)	0.0016	0.0004	0.0010
No. 3 Carbon steel (BAM 028/1, 0.0029 % N)	0.0033	0.0007	0.0012
No. 4 Carbon steel (JK NR3B, 0.0054 % N)	0.0051	0.0005	0.0010
No. 5 Carbon steel (JK 2D, 0.0085 % N)	0.0099	0.0013	0.0015
No. 6 Tool steel (SRM 50 C, 0.012 % N)	0.012	0.0008	0.0010
No. 7 Tool steel (NBS 153a, 0.024 % N)	0.026	0.001	0.002
No. 8 Stainless steel (SRM 73c, 0.037 % N)	0.037	0.003	0.003
No. 9 Cast iron (SRM 890, 0.089 % N)	0.088	0.004	0.008

TABLE 6 Statistical Information—Nitrogen, Range II (chip)

Test Specimen	Nitrogen Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
No. 1 High-temperature alloy A661 (Car Tech, 0.1598 % N)	0.115	0.005	0.015
No. 2 Stainless steel (SRM 367, 0.168 % N)	0.158	0.005	0.017
No. 3 Valve steel (Leco 502-016, 0.218 % N)	0.203	0.009	0.020

TABLE 7 Statistical Information—Nitrogen, Range I (solid)

Test Specimen	Nitrogen Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
No. 1 Low alloy steel (NBS 1098, 0.0032 % N)	0.0041	0.0006	0.0014
No. 2 Low alloy steel (NBS 1096, 0.0040 % N)	0.0043	0.0006	0.0011
No. 3 Cast iron (ACIPCO ~0.01 % N)	0.0076	0.0011	0.0014
No. 4 Low alloy steel 4320 (TC, 0.0080 % N)	0.0083	0.0011	0.0015
No. 5 Tool steel (LSC, 0.064 % N)	0.067	0.004	0.006
No. 6 High temperature alloy Inco 600 (LTV, 0.1 % N)	0.088	0.008	0.015
No. 7 Stainless steel, Type 316 (LTV, 0.09 % N)	0.096	0.004	0.007

OXYGEN BY THE INERT GAS FUSION METHOD

43. Scope

43.1 This test method covers the determination of oxygen in the range 0.001 % to 0.005 % in steel.

44. Summary of Test Method

44.1 This test method is written for use with automated, commercially available analyzers that are based on the inert gas fusion principle and use a variety of gas conditioning and measuring techniques. All use calibration methods traceable to certified reference materials.

44.2 The sample, contained in a small, single-use graphite crucible, is fused under a flowing inert gas stream at a temperature sufficient to release oxygen, nitrogen, and hydrogen. The oxygen combines with carbon from the crucible to form carbon monoxide (CO) and is carried by the flowing inert gas stream to either a thermal conductivity or an infrared detector. In some instruments the carbon monoxide (CO) is converted to carbon dioxide (CO₂) and detect the gas in an infrared cell as CO₂. The detector output is compared to that obtained from similar certified reference materials and is displayed as oxygen content of the sample.

44.3 In a typical instrument based on thermal conductivity detection (Fig. 12), the sample is fused in a stream of helium which carries the released gases over copper oxide which oxidizes CO to CO₂ and hydrogen to water. The water is then absorbed by magnesium perchlorate and the remaining nitrogen and carbon dioxide are separated chromatographically. The

nitrogen is eluted from the chromatographic column within seconds and is either measured or disregarded. The oxygen (as CO₂) then elutes from the column and enters the measuring cell of the thermistor bridge. Bridge output is integrated and the data are processed and displayed directly as percent oxygen.

44.4 In a typical instrument based on infrared detection (Fig. 13), the sample is fused in a stream of inert gas and the evolved gases are passed directly into a cell through which infrared energy is transmitted. The absorption of infrared energy by the CO in the gas stream causes a reduction in the energy received at an infrared detector. The detector response data are processed and displayed directly as percent oxygen.

45. Interferences

45.1 The elements usually present in these alloys do not interfere.

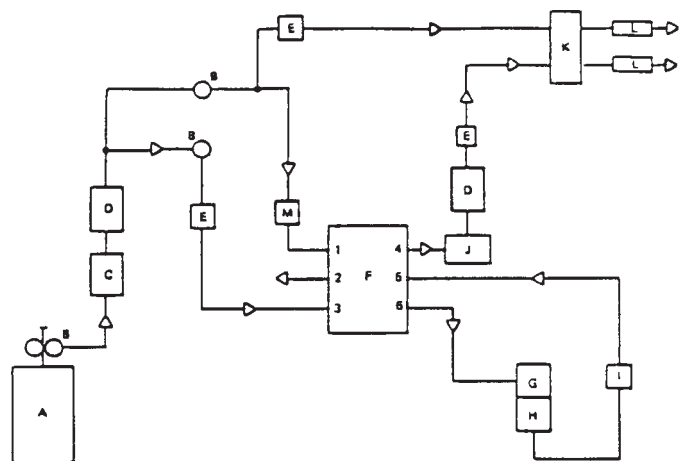
46. Safety Precautions

46.1 For precautions to be observed in the use of certain reagents in this test method, refer to Practices E 50.

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

47. Apparatus

47.1 *Instrument*—The general features of the instruments used in developing the test data accompanying this method are shown schematically in Figs. 12 and 13. These units are commercially available.



- A—Helium Supply
- B—Pressure Regulator
- C—Sodium Hydroxide Impregnated Clay
- D—Magnesium Perchlorate
- E—Flow Control
- F—Flow Manifold
- G—Sample Holding Chamber
- H—Electrode Furnace
- I—Dust Filter
- J—Heated Rare Earth Copper Oxide
- K—Thermal Conductive Detector/Readout
- L—Flow Rotameter
- M—Optional Gas Doser

Manifold Porting

Crucible Degas Flow $\left\{ \begin{array}{l} 2 \text{ to } 4 \\ 5 \text{ to } 3 \\ 1 \text{ to } 6 \end{array} \right.$

Fusion Flow $\left\{ \begin{array}{l} 1 \text{ to } 4 \\ 5 \text{ to } 6 \\ 2 \text{ \& } 3 \text{ off} \end{array} \right.$

FIG. 12 Apparatus for Determination of Oxygen by the Inert Gas Fusion-Thermal Conductivity Method

47.2 Graphite Crucibles—The crucibles must be made of high-purity graphite and should be those recommended by the manufacturer or their equivalent.

48. Reagents

48.1 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.

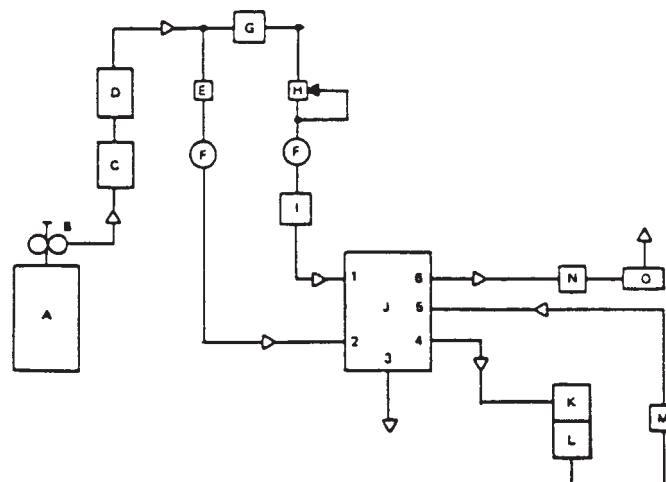
48.2 Acetone—Residue after evaporation must be <0.0005 %.

48.3 Ascarite II (Sodium Hydroxide on Clay)—Used in some instruments to absorb residual CO₂ in the inert gas stream.

48.4 Inert Gas (Helium, Argon, or Nitrogen as Specified for the Instrument being Used)—Use the purity specified by the manufacturer.

48.5 Magnesium Perchlorate (Mg(ClO₄)₂)—Used in most instruments as a moisture trap. Use the purity specified by the manufacturer.

48.6 Rare Earth Copper Oxide, as recommended by the instrument manufacturer.



- A—Inert Gas
- B—Pressure Regulator 2 Stage
- C—Sodium Hydroxide Impregnated Clay
- D—Magnesium Perchlorate
- E—Flow Restrictor
- F—Flow Meter
- G—Pressure Regulator
- H—Needle Valve
- I—Optional Gas Doser
- J—Flow Manifold
- K—Sample Holding Chamber
- L—Electrode Furnace
- M—Dust Filter
- N—Flow Control
- O—IR Detector/Readout

Manifold Porting

Crucible Degas Flow $\left\{ \begin{array}{l} 1 \text{ to } 4 \\ 5 \text{ to } 2 \\ 3 \text{ to } 6 \end{array} \right.$

Fusion Flow $\left\{ \begin{array}{l} 1 \text{ to } 6 \\ 5 \text{ to } 4 \\ 3 \text{ \& } 2 \text{ off} \end{array} \right.$

FIG. 13 Apparatus for Determination of Oxygen by the Inert Gas Fusion-Infrared Absorption Method

49. Preparation of Apparatus

49.1 Assemble the apparatus as recommended by the manufacturer. Make the required power, gas, and water connections. Turn on the instrument and allow sufficient warm up time to stabilize the system.

49.2 Change the chemical traps and filters as required. Test the furnace and the analyzer to ensure the absence of leaks. Make a minimum of two determinations using a sample as directed in Section 52 to condition the instrument before attempting to calibrate the system or to determine the value of the blank.

50. Sample Preparation

50.1 Use only solid samples to minimize the potential for errors due to surface oxidation. Samples must be of the proper size to permit free introduction into the sample loading device, if required, and to fit in the graphite crucible.

50.2 Cut the sample to an appropriate size using a silicon carbide, water-cooled cut-off wheel or by other means that will avoid overheating. Avoid oxide cutting or oxide abrading materials. Thin, flat samples may be stamped using a punch and die. Using a clean file or water cooled silicon carbide grinding media, abrade all surfaces of the sample to remove all traces of surface oxidation or other impurities, rinse in acetone, air-dry, and weigh. Be sure that all pores in the surface of the sample are free of acetone.

50.3 Do not touch the sample with fingers during and following the final stages of cleaning. Store the prepared samples in a desiccator. If they are not analyzed within four hours of preparation, repeat the filing step or grinding step prior to analysis. For some material, such as plain carbon sheet steel, a rimmed or semi-killed deoxidation practice is used and the oxygen may not be uniformly distributed throughout the thickness of the sample. In these cases, the removal of too much surface material may cause erroneous results.

NOTE 21—Careful adherence to the sample preparation procedures described above is critical to obtaining accurate and precise results. The use of small and irregular shaped samples requires a diligent effort to ensure that all surface contamination has been removed.

51. Calibration

51.1 *Calibration Reference Materials*—Select three reference materials containing approximately 0.0005 %, 0.002 %, and 0.005 % oxygen and designate them as Calibrants A, B, and C, respectively.

NOTE 22—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 must be considered as well, if it was not included in the derivation of the published uncertainty values.

51.2 *Gas Dosing*—Automated and manual gas dosing (recommended by some instrument manufacturers) can be used to set up the instrument, but instrument response must be confirmed as described below using the reference materials described in 51.1.

51.3 *Adjustment of Response of Measurement System*—Using Calibrant B as the sample, proceed as directed in 52.2-52.4. Repeat 52.2-52.4 until the absence of drift is indicated; continue running a series of samples until the last four readings have a maximum variation range of 0.0005 % oxygen, making appropriate adjustments to the calibration potentiometer, if applicable. Record potentiometer settings. Adjust the instrument calibration to provide a reading within ± 0.0003 % absolute of the certified value for the calibrant using the average of the last four readings. Refer to the manufacturer's instructions.

51.4 *Determination of Blank Reading*—Using Calibrant A as the sample, proceed as directed in 52.2-52.4. Repeat a sufficient number of times to establish that a low average blank value (0.0002 % oxygen or less) and consistent individual blank values (± 0.0001 % oxygen) are obtained. Blank values are equal to the total result from the crucible and Calibrant A minus the certified oxygen content of Calibrant A. Record the average value of four successive blank determinations which meet the above requirements for maximum and consistent values. If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 52.2-52.4. Enter the average blank value in the appropriate mechanism of the analyzer; refer to manufacturer's instructions. This mechanism will electronically compensate for the blank value (Note 23).

NOTE 23—If the unit does not have this function, the blank value must be subtracted from the total result prior to any calculation.

51.5 Calibration:

51.5.1 Weigh an appropriate sample size of Calibrant C (refer to manufacturer's instructions) to the nearest 1 mg and place it in the instrument sample loading device or directly in the outgassed crucible, following the manufacturer's direction.

51.5.2 Follow the calibration procedure recommended by the manufacturer using Calibrant C to determine the calibration slope. Run samples until the maximum variation of four successive samples does not exceed 0.0005 % oxygen. Treat each sample as directed in 52.2-52.4 before proceeding to the next one.

51.5.3 Confirm the calibration by analyzing an additional sample of Calibrant C after the calibration procedure is completed. The value should agree with the assigned value for Calibrant C within a range of $\pm s$, the standard deviation of a single determination of Calibrant C using this method. If not, repeat the calibration procedure.

51.5.4 Next, weigh at least two samples of appropriate size of Calibrant B to the nearest 1 mg and transfer to the instrument sample loading device. Treat each sample as directed in 52.2-52.4 before proceeding to the next one.

51.5.5 Record the results and compare them to the assigned value for oxygen in Calibrant B. The results should agree with the assigned value for Calibrant B within a range of $\pm s$, the standard deviation of a single determination of Calibrant B using this method. If not, refer to the manufacturer's instructions for checking the linearity of the instrument.

NOTE 24—Repeat the calibration when (1) a different lot of crucibles is used, (2) the system has not been in use for 1 h, or (3) the carrier gas supply has been changed.

52. Procedure

52.1 Assemble the apparatus, calibrate, set the blank, and test the performance as directed in 49 and 51.

52.2 Transfer an appropriate sized sample, weighed to the nearest 1 mg to the instrument sample loading device. (Alternatively, the weighed sample may be manually introduced into the outgassed crucible in Section 52.5. Refer to the manufacturer's directions.)

52.3 Place a crucible on the furnace pedestal and raise the pedestal into position.

52.4 Start the crucible degassing cycle (Note 25); refer to the manufacturer's recommended procedure regarding entry of sample weight.

NOTE 25—For some instruments this procedure automatically precedes the analysis cycle.

52.5 Transfer the sample to the crucible and start the analysis cycle.

52.6 Repeat 52.1-52.5 two additional times and report the average of the three determinations.

53. Calculation

53.1 Follow the manufacturer's directions to ensure that all essential variables in the calculation of analysis results have been accounted for, including the blank, weight of sample, and calibration data. Since the output of most modern instruments is given directly in percent concentration, post-analysis calculations may not be required.

54. Precision and Bias

54.1 *Precision*—Sixteen laboratories cooperated in testing this method and obtained the data summarized in Table 8. Note that the values for R_1 and R_2 are based on $M = 3$ (see Practice E 173), this means that similar values for R_1 and R_2 may be expected from the averages of triplicate determinations.

54.2 *Bias*—Information on the accuracy of this test method is incomplete at this time. The user is cautioned to verify by the use of certified reference materials, if available, that the accuracy of this test method is adequate for the contemplated use.

SULFUR BY THE COMBUSTION-IR INFRARED ABSORPTION METHOD (CALIBRATION WITH METAL REFERENCE MATERIALS)

55. Scope

55.1 These test methods cover the determination of sulfur in the range of 0.002 % to 0.35 %.

56. Summary of Test Method

56.1 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.

56.1.1 *Infrared Absorption: Test Method A*—Sulfur dioxide (SO_2) absorbs infrared (IR) energy at a precise wavelength within the IR spectrum. The 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. The absorption of IR energy can be therefore 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.

56.1.2 *Infrared Absorption: Test Method B*—The combustion is performed in a closed loop in which 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. The IR absorption properties of the SO_2 gas in the chamber cause a loss of energy during combustion; a loss in signal, therefore, results that is proportional to the concentration of the gas in the closed loop. Total sulfur, as SO_2 , is monitored and measured over a period of time. Refer to Fig. 6.

56.1.3 *Infrared Absorption: Test 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 SO_2 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 SO_2 present in the measure chamber, while none is absorbed passing through the reference chamber. This creates an IR energy imbalance reaching the diaphragm and thus distorting it. This distortion alters the fixed capacitance, creating an electric signal change that is amplified for measurement as SO_2 . Total SO_2 is monitored and measured over a period of time. Refer to Fig. 7.

57. Interferences

57.1 The elements normally present do not interfere.

58. Apparatus

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

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

58.3 *Crucible Tongs*, capable of handling recommended crucibles.

59. Reagents

59.1 *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.

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

59.3 *Magnesium Perchlorate*, (known commercially as *Anhydron*).

TABLE 8 Statistical Information—Oxygen (wt., %)

Test Specimen	Oxygen Found	Repeatability ^A (R_1 , Practice E 173)	Reproducibility ^A (R_2 , Practice E 173)
Ni-Cr-Mo Alloy (AISI 4330)	0.00048	0.00018	0.00040
Tool Steel (M-50)	0.00056	0.00015	0.00061
Cr-Alloy Steel (52100)	0.00080	0.00022	0.00088
Carbon Steel (1070)	0.00118	0.00026	0.00065
Carbon Steel (1570)	0.00126	0.00024	0.00071
Alloy Steel (33100)	0.00139	0.00023	0.00048
Cr-Alloy Steel (52100)	0.00218	0.00034	0.00091
Ni-Cr-Mo Alloy (8622)	0.00237	0.00034	0.00076
Carbon Steel (1026)	0.00357	0.00023	0.00092
Cr-Stainless (440C)	0.00437	0.00032	0.00108
Ingot Iron (NBS SRM-466, 0.0050 wt. % oxygen)	0.00538	0.00031	0.00081

^A $M = 3$, see Practice E 173, paragraphs 4.11 and 4.12 and Note 1.

59.4 *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.

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

59.6 *Tungsten Accelerator (Low Sulfur)*, 12 to 20-mesh (Note 26).

NOTE 26—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.

60. Sample Preparation

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

60.2 If necessary, wash in acetone and dry at 70 to 100°C.

61. Preparation of Apparatus

61.1 Assemble the apparatus as recommended by the manufacturer.

61.2 Test the furnace and analyzer to ensure the absence of leaks, and make the required electrical power connections. Prepare the analyzer for operation in accordance with the manufacturer's instructions. Make a minimum of two determinations using a specimen and accelerator, as directed in 63.2 and 63.3, before attempting to calibrate the system or determine the blank.

62. Calibration

62.1 *Calibrants* (Note 27):

62.1.1 For Range I, 0.002 % to 0.010 % sulfur, select three certified reference materials containing approximately 0.001 %, 0.005 %, and 0.010 % sulfur and designate them as Calibrants A, B, and C, respectively.

62.1.2 For Range II, 0.010 % to 0.10 % sulfur, select two certified reference materials containing approximately 0.02 % and 0.08 % sulfur and designate them as Calibrants BB and CC, respectively.

62.1.3 For Range III, 0.10 % to 0.35 % sulfur, select two certified reference materials containing approximately 0.10 % and 0.40 % sulfur and designate them as Calibrants BBB and CCC, respectively.

NOTE 27—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 must be considered as well, if it was not included in the derivation of the published uncertainty values.

62.2 *Adjustment of Response of Measurement System:*

62.2.1 Transfer 1.0 g of Calibrant B, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible (Note 28).

62.2.2 Proceed as directed in 63.1.2 and 63.1.3.

62.2.3 Repeat the steps given in 62.2.1 and 62.2.2 until the absence of drift is indicated. Adjust the signal to provide a reading within ± 0.0003 of the certified percent sulfur value for the certified reference material.

62.3 *Determination of Blank Readings—Range I:*

62.3.1 Transfer 1.0 g of Calibrant A, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, into a preburned crucible (Note 28).

NOTE 28—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, weighed to the nearest 5 mg, using the same amount throughout the entire test method.

62.3.2 Proceed as directed in 63.1.2 and 63.1.3.

62.3.3 Repeat the steps given in 62.3.1 and 62.3.2 a sufficient number of times to establish that low (less than 0.05 mg of sulfur) and consistent (± 0.01 mg of sulfur) readings are obtained. Blank values are equal to the total result of the accelerator and Calibrant A minus the certified value for the calibrant.

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

62.3.5 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 62.3.1-62.3.4.

62.3.6 Enter the average blank value in the analyzer (Note 29); refer to the manufacturer's instructions. This mechanism will compensate electronically for the blank value.

NOTE 29—If the unit does not have this function, the blank value must be subtracted from the total result prior to any calculation.

62.4 *Determination of Blank Reading—Range II*—Proceed as directed in section 62.3.

62.5 *Determination of Blank Reading—Range III:*

62.5.1 Transfer 0.5 g of Calibrant A, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible (Note 28).

62.5.2 Proceed as directed in 62.3.2-62.3.6.

62.6 *Calibration—Range I (0.002 % to 0.010 % Sulfur):*

62.6.1 Weigh four 1.0-g test samples of Calibrant C to the nearest 1 mg, and then place them in preburned crucibles. Add 1.5 g of accelerator, weighed to the nearest 5 mg (Note 28), to each.

62.6.2 Follow the calibration procedure recommended by the manufacturer. Use Calibrant C as the calibrant and analyze at least three test samples to determine the calibration slope. Treat each test sample as directed in 63.1.2 and 63.1.3 before proceeding to the next one.

62.6.3 Confirm the calibration by analyzing Calibrant C following the calibration procedure. The value should be within the allowable limits of the certified value for the certified reference material. If not, repeat the steps given in 62.6.1 and 62.6.2.

62.6.4 Weigh at least two 1.0-g test samples of Calibrant B, weighed to the nearest 1 mg, and transfer them to preburned crucibles. Add 1.5 g of accelerator, weighed to the nearest 5 mg (Note 28), to each.

62.6.5 Treat each test sample as directed in 63.1.2 and 63.1.3 before proceeding to the next one.

62.6.6 Record the results of 62.6.4 and 62.6.5 and compare them to the certified sulfur value of Calibrant B. The results should be within the allowable limits of the certified reference

material. If not, refer to the manufacturer's instructions for determining the linearity of the system (Note 30).

NOTE 30—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.

62.7 Calibration—Range II (0.010 % to 0.10 % Sulfur):

62.7.1 Proceed as directed in 62.6.1-62.6.3, using Calibrant CC.

62.7.2 Proceed as directed in 62.6.4-62.6.6, using Calibrant BB.

62.8 Calibration—Range III (0.10 % to 0.35 % Sulfur):

62.8.1 Weigh four 0.5-g test samples of Calibrant CCC, to the nearest 1 mg, and place them in preburned crucibles. Add 1.5 g of accelerator, weighed to the nearest 5 mg (Note 28), to each.

62.8.2 Follow the calibration procedure recommended by the manufacturer. Use Calibrant CCC as the primary calibrant and analyze at least three test samples to determine the calibration slope. Treat each test sample as directed in 63.1.2 and 63.1.3 before proceeding to the next one.

62.8.3 Confirm the calibration by analyzing Calibrant CCC following the calibration procedure. The value should be within the allowable limits of the certified value for the certified reference material. If not, repeat the steps given in 62.8.1 and 62.8.2.

62.8.4 Weigh at least two 0.5-g test samples of Calibrant BBB, weighed to the nearest 1 mg, and transfer them to preburned crucibles. Add 1.5 g of accelerator, weighed to the nearest 5 mg (Note 28), to each.

62.8.5 Treat each test sample as described in 63.1.2 and 63.1.3 before proceeding to the next one.

62.8.6 Record the results of 62.8.4 and 62.8.5 and compare them to the certified sulfur value of the Calibrant BBB. The results should be within the allowable limits of the certified reference material. If not, refer to the manufacturer's instructions for determining the linearity of the analyzer (Note 30).

63. Procedure

63.1 Procedure—Range I:

63.1.1 Stabilize the furnace and analyzer as directed in Section 59. Transfer 1.0 g of test sample, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible (see 58.2) (Note 28).

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

TABLE 9 Statistical Information, Sulfur, Range I (0.002 % to 0.010 % S)

Test Material	Certified Value, % Sulfur	Sulfur Found, %	Repeatability (R_1 , Practice E 173)	Reproducibility (R_2 , Practice E 173)
JK NR24	0.0010	0.00103	0.00025	0.00078
NIST 132b	0.0030	0.0027	0.00033	0.00094
High Temperature Alloy	...	0.0043	0.00051	0.00118
NIST 50c	0.0064	0.0065	0.0005	0.0024

TABLE 10 Statistical Information, Sulfur, Range II (0.010 % to 0.10 % S)

Test Material	Certified Value, % Sulfur	Sulfur Found, %	Repeatability (R_1 , Practice E 173)	Reproducibility (R_2 , Practice E 173)
NIST 890	0.015	0.0149	0.0015	0.0039
NIST 163	0.027	0.0264	0.0015	0.0092
NIST 73c	0.036	0.0356	0.0032	0.0078

TABLE 11 Statistical Information, Sulfur, Range III (0.10 % to 0.35 % S)

Test Material	Certified Value, % Sulfur	Sulfur Found, %	Repeatability (R_1 , Practice E 173)	Reproducibility (R_2 , Practice E 173)
NIST 6g	0.124	0.1200	0.0076	0.0239
NIST 129c	0.245	0.2451	0.0074	0.0243
NIST 133	0.356	0.3683	0.0174	0.0373

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

63.2 Procedure—Range II—Proceed as directed in 63.1.

63.3 Procedure—Range III—Proceed as directed in 63.1, using a 0.5-g test sample.

64. Calculation

64.1 Since most commercially available instruments calculate percent concentrations directly, including corrections for blank and sample weight, calculations by the analyst are not required (Note 31).

NOTE 31—Use the following formula if the analyzer does not compensate for blank and sample weight values:

$$\text{sulfur, \%} = [(A - B) \times C/D] \quad (4)$$

where:

- A = DVM reading for test sample,
- B = DVM reading for blank,
- C = weight compensator setting, and
- D = test sample weight in grams.

65. Precision and Bias

65.1 Precision—Nine laboratories cooperated in testing these test methods and obtained the precision data summarized in Tables 9-11 (see Note 32).

NOTE 32—In Tables 9-11, all results in Ranges I and II are from nine laboratories. The results in Range III are from eight laboratories.

65.2 Bias—The accuracy of these test methods can be inferred from the data given in Tables 9-11 by comparing the certified values for sulfur with the average values obtained (see Note 33).

NOTE 33—Although these test methods were tested to only 0.35 %, most commercial analyzers are believed to be capable of analyzing samples containing sulfur up to 0.5 %.

66. Keywords

66.1 carbon content; cobalt alloys; combustion; determination; fusion; gas dosing; inert gas fusion; infrared absorption; iron alloys; nickel alloys; oxygen content; sulfur content; thermal conductivity

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