



Designation: E 1019 – 00₃

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 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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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 (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:

	Sections
Carbon, Total, by the Combustion-Instrumental Measurement—Method	—
Carbon, Total, by the Combustion-Instrumental Measurement Method	9-19
Nitrogen by the Inert Gas Fusion-Thermal Conductivity Method	10-20
Nitrogen by the Inert Gas Fusion-Thermal Conductivity Method	31-41
Oxygen by the Inert Gas Fusion Method	32-42
Oxygen by the Inert Gas Fusion Method	42-53
Oxygen by the Inert Gas Fusion Method	43-54
Sulfur by the Combustion-Infrared Absorption Method (Calibration with Metal Standards)	—
Sulfur by the Combustion-Infrared Absorption Method (Calibration with Metal Reference Materials)	54-64
Sulfur by the Combustion-Infrared Absorption Method (Potassium Sulfate Calibration)	55-65
Sulfur by the Combustion-Infrared Absorption Method (Potassium Sulfate Calibration)	—
Sulfur by the Combustion-Infrared Absorption Method (Potassium Sulfate Calibration)	20-30
Sulfur by the Combustion-Infrared Absorption Method (Potassium Sulfate Calibration)	21-31

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. 6.~~

2. Referenced Documents ³

2.1 ASTM Standards:

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.

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*, Vol 14.02, volume information, refer to the standard's Document Summary page on the ASTM website.

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 1806 Practice 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. Significance and Use

~~3.1 These test methods for the chemical analysis~~ Terminology

~~3.1 For definition 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~~ terms used in a properly equipped laboratory. this test method, refer to Terminology E 135.

4. Apparatus ~~Significance and Reagents~~

~~4.1 Apparatus and reagents required~~ Use

~~4.1 These test methods for each determination 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 separate sections preceding the procedure.~~ a properly equipped laboratory.

5. Hazards

~~5.1 For hazards to be observed in the use of certain~~ Apparatus and Reagents

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

6. Sampling ~~Hazards~~

~~6.1 For procedures for sampling hazards to be observed in the materials, use of certain reagents in this test method, refer to those parts of Practice E 1806.~~ 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. Rounding ~~Calculated Values~~

~~7.1 Calculated values shall be rounded to~~ Sampling

~~7.1 For procedures for sampling the desired number materials, refer to those parts of places as directed in Practice E 29.~~ 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

9. Scope

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

10. Scope

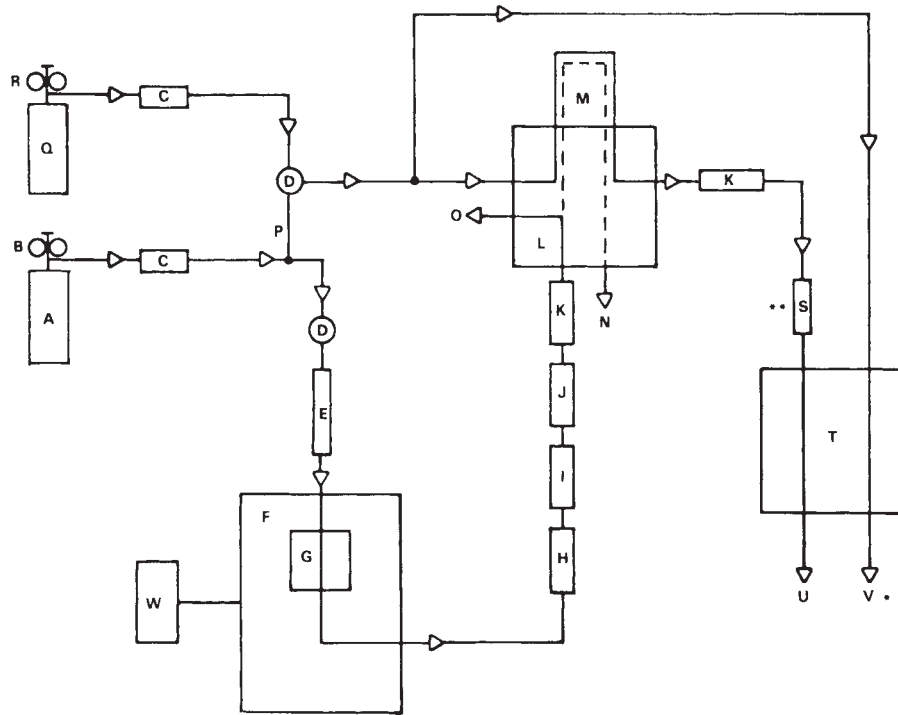
10.1 This test method covers the determination of carbon in concentrations from 0.005 % to 4.5 %.

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 (CO₂) absorbs IR energy at a precise wavelength within the IR spectrum. Energy of this wavelength is absorbed as the



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—Chromographic 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.

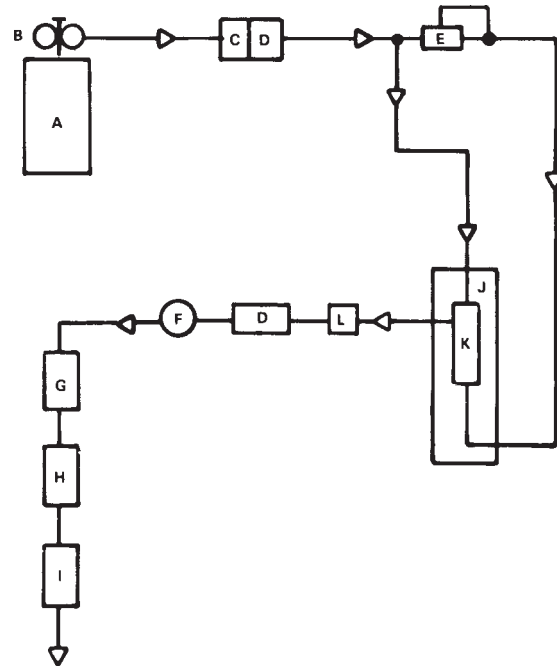
FIG. 1 Apparatus for Determination of Carbon by the Combustion Thermal Conductivity Method

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.

101.1.3 *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.

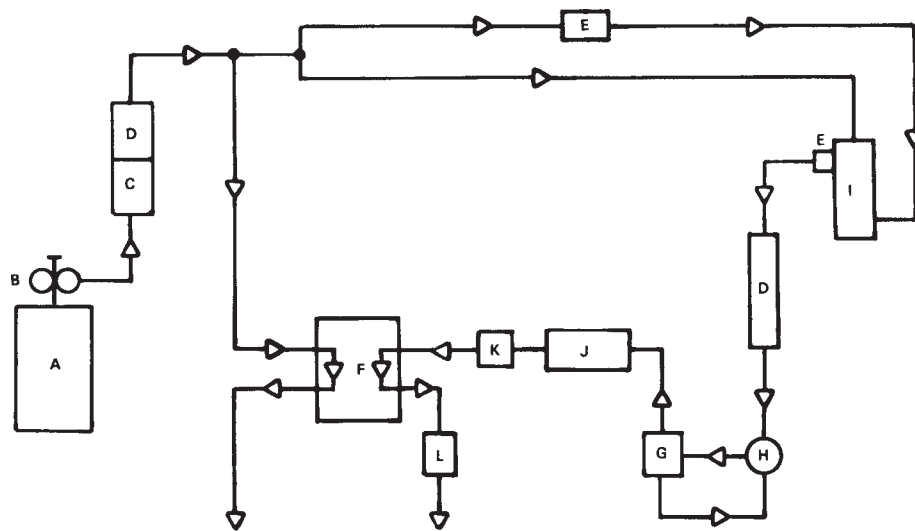
101.1.4 *Infrared (IR) Absorption, Method C, Closed Loop*—The combustion is performed in a closed loop, where CO and CO₂ are detected in the same infrared cell. Each gas is measured with a solid state energy detector. Filters are used to pass the appropriate IR wavelength to each detector. In the absence 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.

101.2 This test method is written for use with commercial analyzers, equipped to carry out the above operations automatically



- | | |
|-------------------------------------|-----------------------------------|
| 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



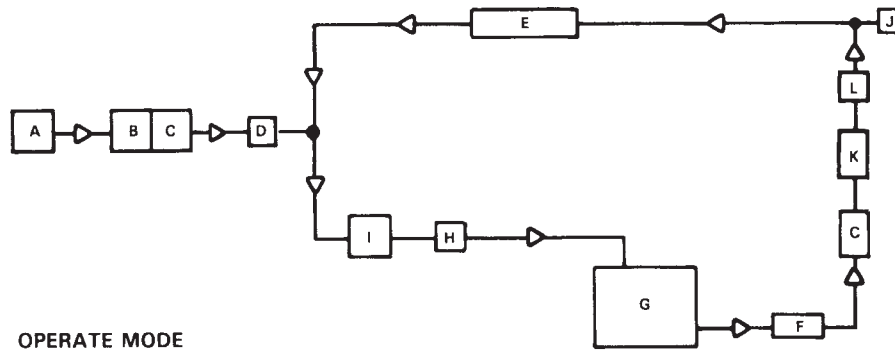
- | | |
|-------------------------------------|-----------------------------------|
| A—Oxygen Cylinder | G—Orifice |
| B—Two Stage Regulator | H—Pressure Regulator |
| C—Sodium Hydroxide Impregnated Clay | I—Combustion Chamber |
| D—Magnesium Perchlorate | J—CO to CO ₂ Converter |
| E—Dust Trap | K—SO ₃ Trap |
| F—IR Cell/Readout | L—Measure Flow Rotameter |

FIG. 3 Infrared Absorption Method B

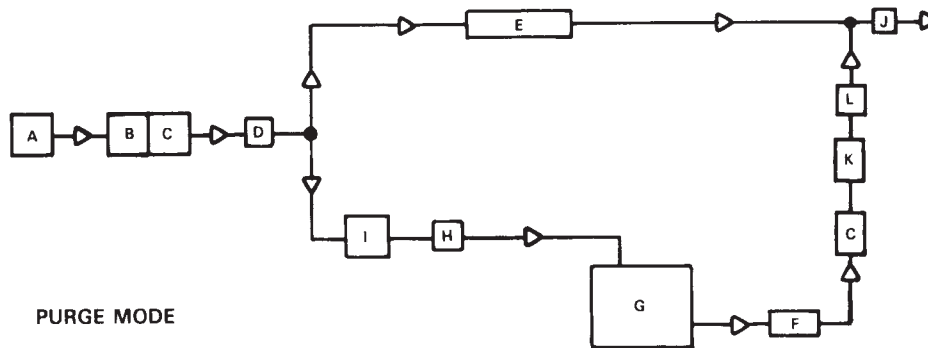
and calibrated using steels of known carbon content.

11. Interferences

11.1 The elements ordinarily present do not interfere.



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

12. Interferences

12.1 The elements ordinarily present do not interfere.

13. Apparatus

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

123.2 *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.

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

134. Reagents

134.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

13.2—

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

13.3—

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

⁴ *Annual Book of Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of ASTM reagents not listed by the American Chemical Society, see *Analar Standards—Vol 03-05: 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.

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.

~~13.4~~

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

~~13.5~~

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

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

~~13.6~~

14.9 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 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 size, but not finer than 40 mesh.~~

~~15.2 Wash in acetone 18.1.2 and dry at 70 18.1.3 before attempting to 100°C. 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

~~167.1 Calibration Standards Reference Materials~~ (Note 2):

~~167.1.1 For Range I, 0.005 % to 0.10 % carbon, select three primary standard certified reference materials containing approximately 0.005 %, 0.05 %, and 0.10 % carbon and designate them as Standards Calibrants A, B, and C, respectively.~~

~~167.1.2 For Range II, 0.10 % to 1.25 % carbon, select two primary standard certified reference materials containing approximately 0.12 % and 1.00 % carbon and designate them as Standards Calibrants BB and CC, respectively.~~

~~167.1.3 For Range III, 1.25 % to 4.50 % carbon, select two primary standard certified reference materials containing approximately 1.25 % and 4.00 % carbon and designate them as Standards Calibrants BBB and CCC, respectively.~~

NOTE 2—The accuracy uncertainty of results obtained using this test method is dependent upon on the accuracy uncertainty of the test methods used values assigned to certify the carbon concentration in the calibration primary standard reference materials. The homogeneity of the reference materials must be considered as well, if it was up not included in their derivation of the published uncertainty values.

~~167.2 Adjustment of Response of Measurement System:~~

~~167.2.1 Transfer 1.0 g of Standard Calibrant B, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, to a preburned crucible.~~

~~167.2.2 Proceed as directed in 178.1.2 and 178.1.3.~~

~~167.2.3 Repeat 167.2.1 and 167.2.2 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 certified reference material.~~

~~167.3 Determination of Blank Reading—Range I:~~

~~167.3.1 Transfer 1.0 g of Standard Calibrant A, weighed to the nearest 1 mg, and 1.5 g of accelerator, weighed to the nearest 5 mg, into a preburned crucible.~~

~~167.3.2 Proceed as directed in 178.1.2 and 178.1.3.~~

~~167.3.3 Repeat 167.3.1 and 167.3.2 a sufficient number of times to establish that low (less than 0.020 mg 0.002% of carbon) and consistent ($\pm 0.0002 \text{ mg}$ $\%$ of carbon) readings are obtained. Blank values are equal to the total result of the accelerator and Standard Calibrant A minus the certified value for the primary standard certified reference material.~~

~~167.3.4 Record the average value of at least three blank determinations.~~

~~167.3.5 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 167.3.1-167.3.4.~~

~~167.3.6 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.

167.4 *Determination of Blank Reading—Range II*—Proceed as directed in 167.3.

167.5 *Determination of Blank Reading—Range III*:

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

167.5.2 Proceed as directed in 167.3.2-167.3.6.

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

167.6.1 Weigh four 1.0 g specimens of Standard 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.

167.6.2 Follow the calibration procedure recommended by the manufacturer. Use Standard Calibrant C as the primary ~~calibration standard~~ calibrant and analyze at least three specimens to determine the calibration slope. Treat each specimen, as directed in 178.1.2 and 178.1.3, before proceeding to the next one.

167.6.3 Confirm the calibration by analyzing Standard Calibrant C following the calibration procedure. The value should be within the allowable limits of the certified value for the ~~primary standard~~ certified reference material. If not, repeat 167.6.1 and 167.6.2.

167.6.4 Weigh at least two 1.0 g specimens of Standard 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.

167.6.5 Treat each specimen as directed in 178.1.2 and 178.1.3 before proceeding to the next one.

167.6.6 Record the results of 167.6.4 and 167.6.5 and compare them to the certified carbon value of Standard Calibrant B. The results should be within the allowable limits of the ~~primary standard~~ 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.

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

167.7.1 Proceed as directed in 167.6.1-167.6.3, using Standard Calibrant CC.

167.7.2 Proceed as directed in 167.6.4-167.6.6, using Standard Calibrant BB.

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

167.8.1 Weigh four 0.5 g specimens of Standard 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.

167.8.2 Follow the calibration procedure recommended by the manufacturer. Use Standard Calibrant CCC as the ~~primary calibration standard~~ certified reference material and analyze at least three specimens to determine the calibration slope. Treat each specimen, as directed in 178.1.2 and 178.1.3, before proceeding to the next one.

167.8.3 Confirm the calibration by analyzing Standard Calibrant CCC following the calibration procedure. The value should be within the allowable limits of the certified value for the ~~primary standard~~ certified reference material. If not, repeat 167.8.1 and 167.8.2.

167.8.4 Weigh at least two 0.5 g specimens of Standard 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.

167.8.5 Treat each specimen as described in 178.1.2 and 178.1.3 before proceeding to the next one.

167.8.6 Record the results of 167.8.4 and 167.8.5 and compare to the certified carbon value of the Standard Calibrant BBB. The results should be within the allowable limits of the ~~primary standard~~ certified reference material. If not, refer to manufacturer's instructions for checking the linearity of the analyzer (Note 4).

178. Procedure

178.1 *Procedure—Range I*:

178.1.1 Stabilize the furnace and analyzer as directed in Section 14. 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 123.2.)

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

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

178.2 *Procedure—Range II*—Proceed as directed in 178.1.

178.3 *Procedure—Range III*—Proceed as directed in 178.1, using a 0.5 g specimen.

189. Calculation

189.1 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:

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

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
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 (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
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 (NBS 133b, 0.128 C)	0.126	0.005	0.013
6. Type 416 stainless steel (SRM 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
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

where:

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

~~19.~~

20. Precision and Bias

~~19.1~~⁵

~~20.1~~ *Precision*—Nine laboratories cooperated in testing this method and obtained the data summarized in Tables ~~1-3~~, 1-3. Testing was performed in compliance with Practice E 173.

~~19.2~~

~~20.2~~ *Bias*—~~No information on the accuracy of this test method is known. 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 ~~may be judged, however, by comparing accepted reference values with~~ is adequate for the corresponding arithmetic average obtained by interlaboratory testing, contemplated use.

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

20. Scope

~~20.1~~ 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.

Annual Book of

⁵ Supporting data are available from ASTM Standards, Vol 03.06, International Headquarters. Request RR:E03-1038.

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

242.1 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.

242.1.1 *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.

242.1.2 *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.

242.1.3 *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. Interferences

23.1 The elements ordinarily present do not interfere.

24. Apparatus

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

234.2 *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.

234.3 *Micropipet*, (50 µL).

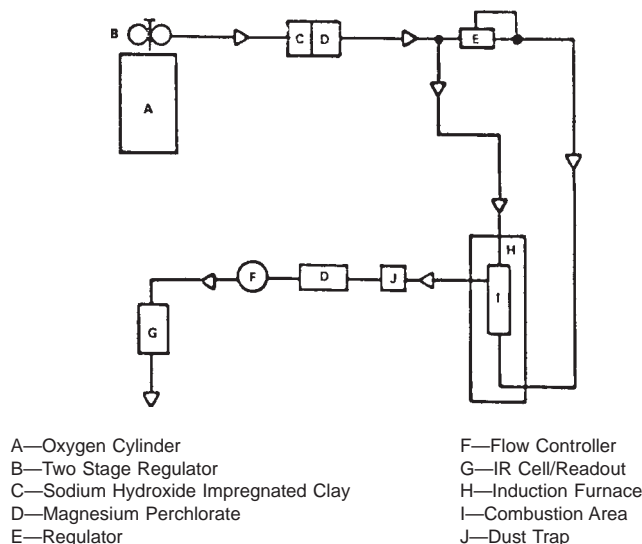
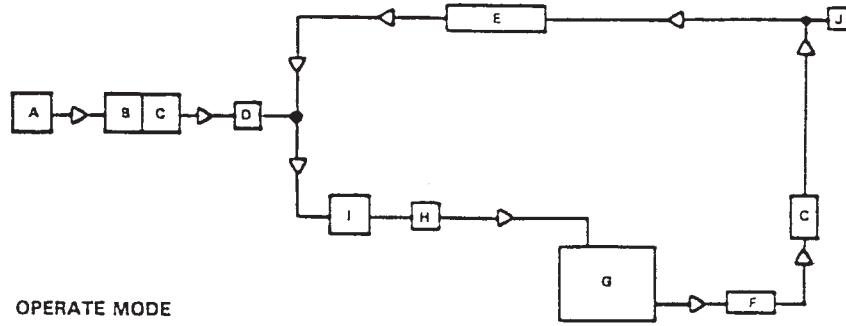
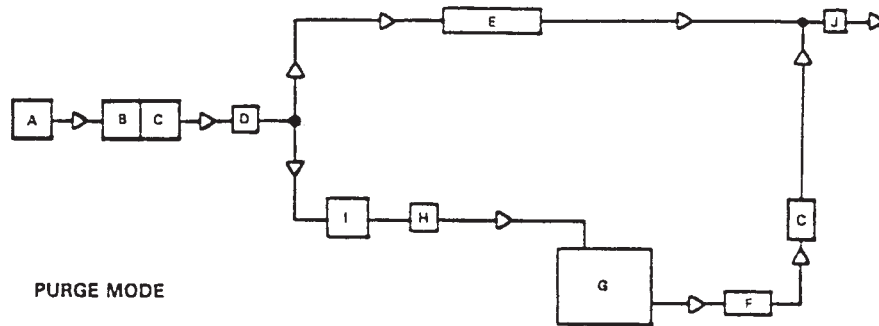


FIG. 5 Infrared Absorption Method A



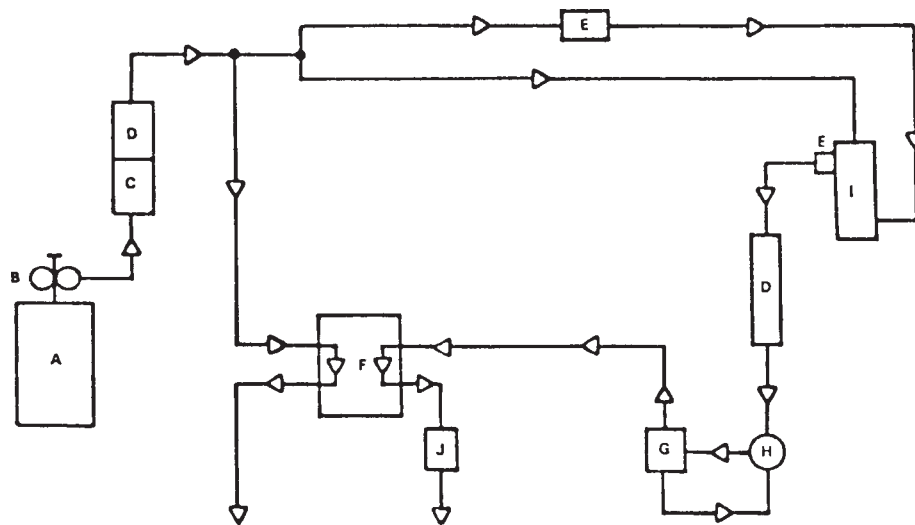
OPERATE MODE



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

234.4 Crucible Tongs—Capable of handling recommended crucibles.

234.5 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.

245. Reagents

245.1 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 %.

~~24.2~~

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 *Anhydrone*).³

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.

~~24.4~~

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.

245.7 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.

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 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-size but not finer than 40 mesh.~~

~~26.2 Wash in acetone 29.2 and dry at 70 29.3 before attempting to 100°C.~~

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

278.1 *Calibration—Standards Reference Materials* :

278.1.1 Weigh to the nearest 0.0001 g the following masses of K₂SO₄:

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

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

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

278.1.4 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 278.1.5.

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

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

278.2 *Adjustment of Response of Measurement System:*

278.2.1 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 289.2 and 289.3.

278.2.2 Repeat 278.2.1 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.

278.3 *Determination of Blank Reading* :

278.3.1 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 289.2 and 289.3.

278.3.2 Repeat 278.3.1 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 H.

28.3.3 ~~Record the sulfur contained in 1.0 g of iron.~~

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

278.3.4 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 278.3.1-278.3.3. Prepare more capsules of sulfur solution H if necessary.

278.3.5 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.

278.4 *Calibration:*

278.4.1 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.

278.4.2 Follow calibration procedure recommended by the manufacturer using dried capsules of sulfur solution D as the primary standard, calibrant, analyzing at least three specimens to determine the calibration slope. Treat each capsule as directed in 289.2 and 289.3 before proceeding to the next one.

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

278.4.3 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 278.4.1 and 278.4.2.

278.4.4 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.

278.4.5 Treat each capsule as directed in 289.2 and 289.3 before proceeding to the next one.

278.4.6 Record the results of 278.4.5 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. 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

2930.1 Since most commercially available instruments calculate percent concentrations directly, including corrections for blank and sample weight, calculations by the analyst are not required.

NOTE 910—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.

301. Precision and Bias

30.1⁶

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.

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

NITROGEN BY THE INERT GAS FUSION THERMAL CONDUCTIVITY METHOD

312. Scope

312.1 This test method covers the determination of nitrogen (N) in concentrations from 0.0010 % to 0.2 % (Note 101).

⁶ Discontinued 1998; see 1997 Annual Book of

⁶ Supporting data are available from ASTM Standards, Vol 03.06. International Headquarters. Request RR: E03-1041.

NOTE 101—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.

323. Summary of Test Method

323.1 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.

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

33. Interferences

33.1 The elements ordinarily present do not interfere.

34. Interferences

34.1 The elements ordinarily present do not interfere.

35. Apparatus

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

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

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

356. Reagents

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

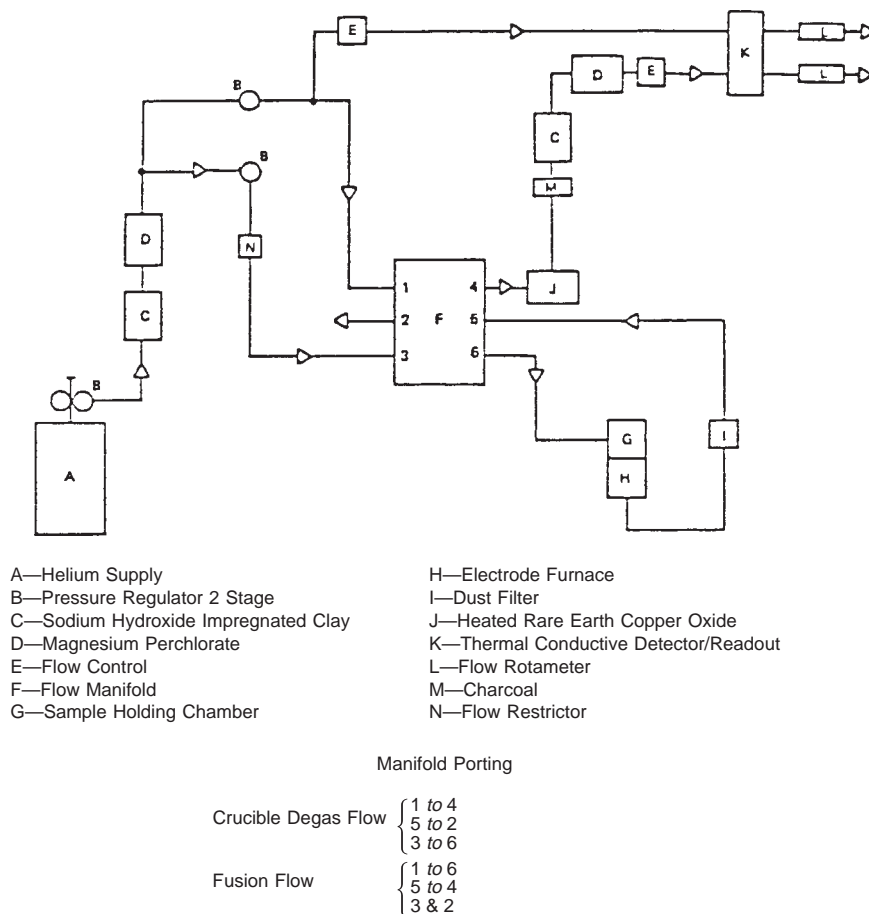
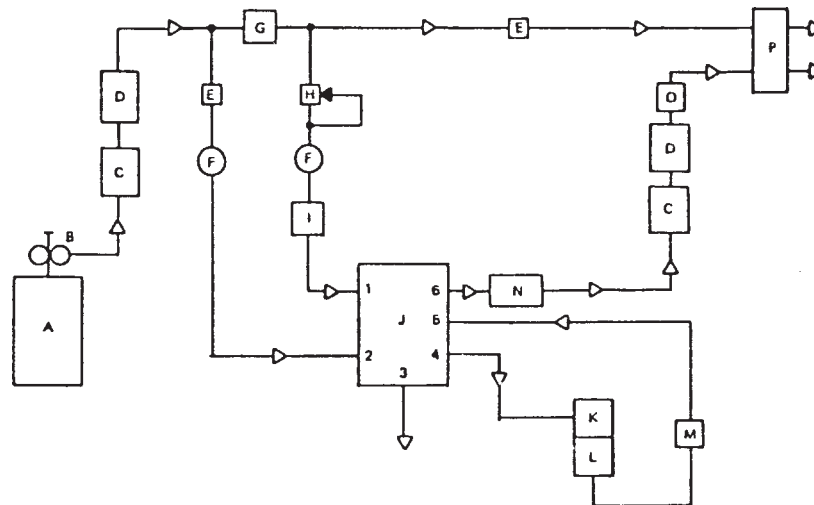


FIG. 8 Nitrogen Method A Flow Diagram



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

Manifold Porting

- | | |
|---------------------|---------------------------------|
| Crucible Degas Flow | { 2 to 4
5 to 3
1 to 6 |
| Fusion Flow | { 1 to 4
5 to 6
2 & 3 off |

FIG. 9 Nitrogen Method B Flow Diagram

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.

35.2—

36.3 Copper.

36.4 Helium, high-purity (99.99 %).

35.3—

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

35.4—

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*).

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.

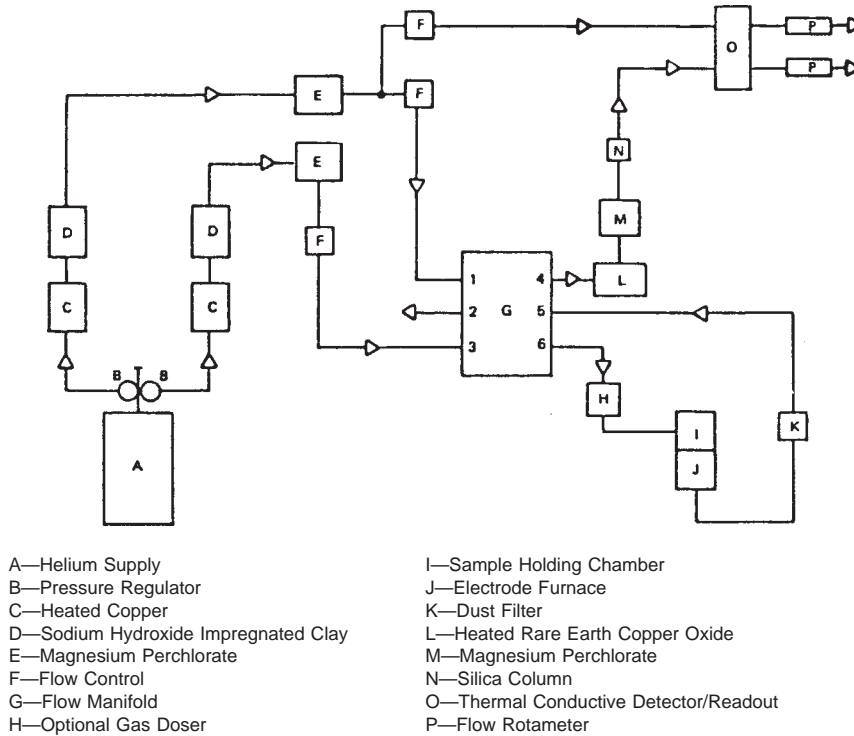
37. Preparation of Apparatus

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.

38. Sample Preparation

378.1 Use chips, drillings, or solid form specimens prepared as directed in 378.2 or 378.3 (Note 142).



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

Manifold Porting

- Crucible Degas Flow { 1 to 4
5 to 2
3 to 6
- Fusion Flow { 1 to 6
5 to 4
3 & 2 off

FIG. 10 Nitrogen/Oxygen Method A Flow Diagram

NOTE 142—Size all specimens to permit free introduction through the loading device of the equipment or directly into the graphite crucible.

378.2 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.

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

389. Calibration

38.1 Calibration Standards

39.1 Reference Materials:

389.1.1 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~~, Calibrants A, B, C, D, and E, respectively (Note 123).

389.1.2 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~~ Calibrants AA, BB, and CC, respectively (Note 123).

~~NOTE 12—The accuracy 13—The uncertainty of the results obtained using this test method is largely dependent upon on the accuracy uncertainty of the methods used values assigned to certify the nitrogen concentration in the calibration reference materials. The homogeneity of certified the reference materials, as well as upon their homogeneity. Thus, calibration specimens should materials must be NBS Standard Reference Materials (SRM) or other certified reference materials.~~

~~38.2— considered as well, if it was not included in the derivation of the published uncertainty values.~~

39.2 Adjustment of Response of Measurement System:

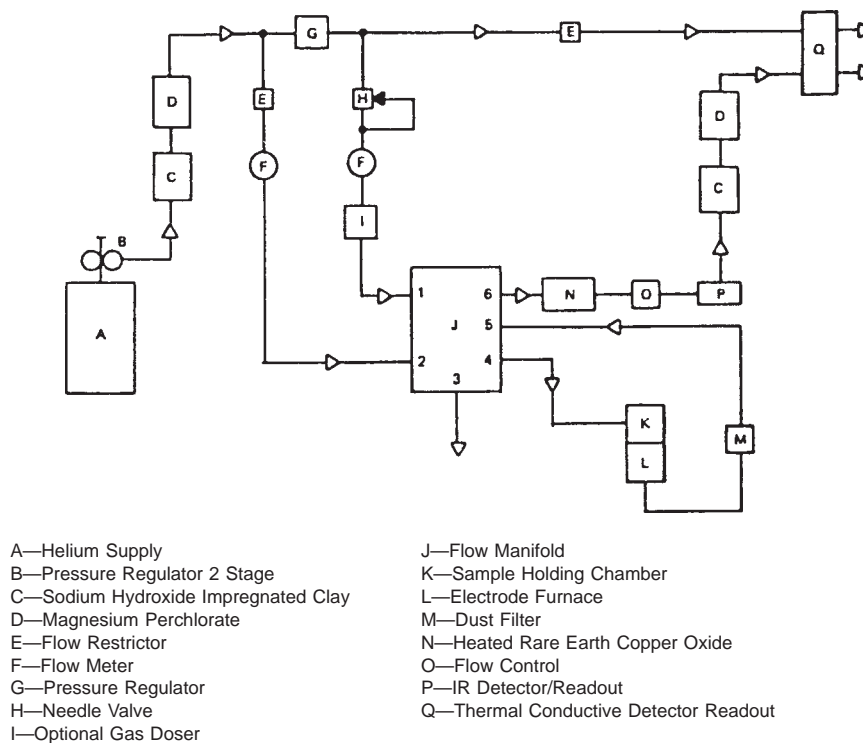
389.2.1 Prepare a 1.0 g specimen of ~~standard~~ Calibrant C weighed to the nearest 1 mg as directed in 378.2 or 378.3.

389.2.2 Proceed as directed in ~~39.2.1~~ 40.2.1 or ~~39~~ 40.2.2.

389.2.3 Repeat 389.2.1 and 389.2.2 and adjust as recommended by the manufacturer until the absence of drift is indicated.

389.3 Determination of Blank Reading—Ranges I and II:

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



Manifold Porting

Crucible Degas Flow { 2 to 4
 5 to 3
 1 to 6
 Fusion Flow { 1 to 4
 5 to 6
 2 & 3 off

FIG. 11 Nitrogen/Oxygen Method B Flow Diagram

389.3.2 Make three blank determinations as directed in 39.2.1 40.2.1 or 39.2.2 40.2.2 with the sample omitted. Use a fresh crucible each time (Note 134).

NOTE 134—If the loading device is used to analyze the unknown or standard calibration sample, see Note 142.

389.3.3 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 389.3.1 and 389.3.2.

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

389.3.5 Enter the average blank value in the appropriate mechanism of the analyzer (Note 145); refer to manufacturer’s instructions. This mechanism will electronically compensate for the blank value.

NOTE 145—If the unit does not have this function, the average blank value must be subtracted from the DVM readings for standards reference materials and specimens (Note 189).

389.4 Calibration Procedure—Range I:

389.4.1 Prepare four 1.0 g specimens of Calibration Standard Calibrant D weighed to the nearest 1 mg, as directed in 378.2 or 378.3.

389.4.2 Follow the calibration procedure recommended by the manufacturer using standard Calibrant D as the primary standard-certified reference material. Analyze at least three standard reference material specimens to determine calibration slope. Treat each specimen as directed in 39.2.1 40.2.1 or 39.2.2 40.2.2 before proceeding to the next one.

389.4.3 Confirm the calibration by analyzing standard Calibrant D after calibration. The value should be within the allowable limits of the certified value. If not, repeat 389.4.1 and 389.4.2.

389.4.4 Prepare two 1.0 g specimens each of standards Calibrants A, B, C, and E weighed to the nearest 1 mg, as directed in 378.2 or 378.3.

389.4.5 Treat each specimen as directed in 39.2.1 40.2.1 or 39.2.2 40.2.2 before proceeding to the next one.

389.4.6 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 156—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.

389.5 Calibration Procedure—Range II:

389.5.1 Proceed as directed in 389.4.1-389.4.3 using standard Calibrant CC.

389.5.2 Proceed as directed in 389.4.4-389.4.6 using standards Calibrants AA and BB (Note 15).

~~39. 16).~~

40. Procedure

3940.1 Assemble the apparatus and condition it as directed in Section ~~36.~~

~~39.2- 37.~~

40.2 Procedure—Ranges I and II:

3940.2.1 Manual Operation:

3940.2.1.1 Set the analyzer to operate in the manual mode.

3940.2.1.2 Place an empty crucible on the furnace lower electrode assembly and close the furnace.

3940.2.1.3 Weigh a 1.0 g specimen to the nearest 1 mg and refer to the manufacturer's recommended procedure, including entry of sample weight.

3940.2.1.4 Start the analysis cycle, referring to the manufacturer's recommended procedure (Note 167).

3940.2.2 Automatic Operation:

3940.2.2.1 Set the analyzer to operate in the automatic mode.

3940.2.2.2 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.

3940.2.2.3 Place an empty crucible on the furnace lower electrode assembly and close the furnace.

3940.2.2.4 Start the analysis cycle, referring to the manufacturer's recommended procedure (Notes ~~16 17~~ and 178).

NOTE 167—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 178—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 ~~calibration standards~~ calibrants be of the same or similar composition as the samples to be analyzed.

401. Calculation

401.1 With most commercially available instruments the readings will be given directly in concentration (Note 189).

NOTE 189—If the analyzer does not compensate for blank and sample weight values, then the equation is:

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

where:

A = DVM reading for specimen,

B = DVM reading for blank,

C = weight compensator setting, and

D = specimen weight, g.

412. Precision and Bias

~~41.1-7~~

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 ~~19~~-20).

NOTE 1920—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 %.

OXYGEN BY THE INERT GAS FUSION METHOD

42. Scope

42.1 This test method covers the determination of oxygen in the range 0.001 to 0.005 % in steel and in iron, nickel, and cobalt alloys having chemical compositions within the limits shown in 1.1.

⁷ Supporting data are available from ASTM International Headquarters. Request RR: E03-103851.

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 (Eurostandard 088.1, 0.0009 % N)	0.0040	0.0005	0.0008
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 (NBS 50 C, 0.012 % N)	0.012	0.0008	0.0010
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 (NBS 73c, 0.037 % N)	0.037	0.003	0.003
No. 8 Stainless steel (SRM 73c, 0.037 % N)	0.037	0.003	0.003
No. 9 Cast iron (NBS 890, 0.089 % N)	0.088	0.004	0.008
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 (NBS 367, 0.168 % N)	0.158	0.005	0.017
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

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

434.1 This test method is written for use with automated, commercially available analyzers ~~which 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 primary Standard Reference Materials (SRM).

43.2 ~~The certified reference materials.~~

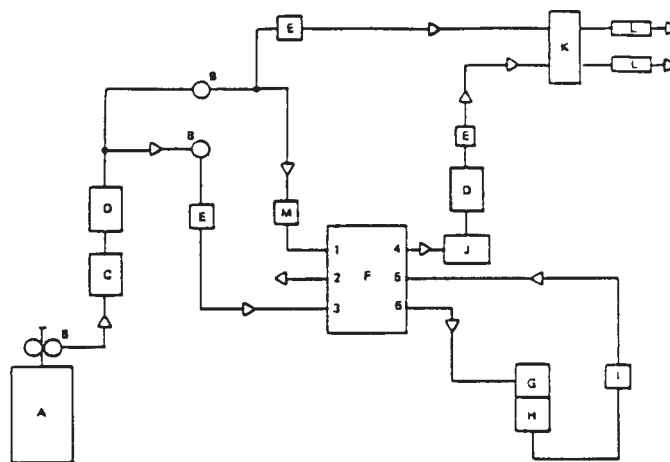
44.2 The sample, contained in a small, single-use graphite crucible, is fused under a flowing inert gas stream at a temperature sufficient to release oxygen, nitrogen, and hydrogen. The oxygen combines with carbon from the crucible to form carbon monoxide (CO) and is carried by the flowing inert gas 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 infared cell as CO₂. The detector output is compared to that obtained from similar ~~Standard Reference Materials~~ certified reference materials and is displayed as oxygen content of the sample.

434.3 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.

434.4 In a typical instrument based on infrared detection (Fig. 13), the sample is fused in a stream of ~~nitrogen or argon 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.

44. Interferences

44.1 ~~The elements usually present in these alloys do not interfere.~~



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

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

45. Safety Precautions

45.1 For precautions to be observed ~~Interferences~~

45.1 The elements usually present in the use of certain reagents in this test method, refer to Practices E 50.

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

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

478. Reagents

478.1 *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.

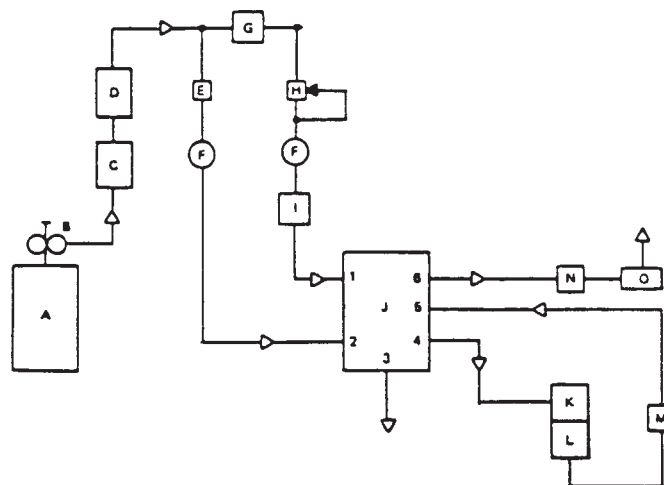
48.2 *Acetone*—Residue after evaporation must be <0.0005 %.

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

47.3 *stream.*

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

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



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

Manifold Porting

- | | |
|----------------|-------------|
| Crucible Degas | { 1 to 4 |
| Flow | { 5 to 2 |
| | { 3 to 6 |
| Fusion Flow | { 1 to 6 |
| | { 5 to 4 |
| | { 3 & 2 off |

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

48. Preparation of Apparatus

48.1 Assemble the apparatus

48.6 *Rare Earth Copper Oxide*, 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.

48.2 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 16 to condition the instrument before attempting to calibrate the system or to determine the value of the blank. manufacturer.

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

4950.1 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.

4950.2 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 oxides 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.

4950.3 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 20~~1~~—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.

501. Calibration

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

NOTE 21~~—The accuracy~~ 22~~—The uncertainty of the results obtained using this test method is dependent to a large extent upon on the accuracy~~ uncertainty of the methods used values assigned to certify the oxygen concentration in the calibration reference materials. The homogeneity of the reference materials must be considered as well, if it was up not included in their derivation of the published uncertainty values.

501.2 ~~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 ~~standards~~ reference materials described in 501.1.

501.3 ~~Adjustment of Response of Measurement System~~ —Using ~~Standard Calibrant B~~ as the sample, proceed as directed in 512.2-512.4. Repeat 512.2-512.4 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 ~~standard calibrant~~ using the average of the last four readings. Refer to the manufacturer's instructions.

501.4 ~~Determination of Blank Reading~~— Using ~~Standard Calibrant A~~ as the sample, proceed as directed in 512.2-512.4. 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 ~~Standard Calibrant A~~ minus the certified oxygen content of ~~Standard 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 512.2-512.4. 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 223).

NOTE 223—If the unit does not have this function, the blank value must be subtracted from the total result prior to any calculation.

501.5 ~~Calibration~~—:

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

51.5.2 Follow the calibration procedure recommended by the manufacturer using ~~standard Calibrant C as the primary standard,~~ analyzing samples of Standard C to determine the calibration slope. Run samples until the maximum variation range of four successive samples does not exceed 0.0005 % oxygen. Treat each sample ~~of the standard~~ as directed in 512.2-512.4 before proceeding to the next one.

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

51.5.4 Next, weigh at least two ~~appropriate-sized~~ samples of ~~Standard B,~~ weighed appropriate size of Calibrant B to the nearest 1 ~~mg,~~ mg and transfer to the instrument sample loading device. Treat each sample ~~of the standard~~ as directed in 512.2-512.4 before proceeding to the next one.

51.5.5 Record the results and compare them to the ~~actual certified oxygen assigned value of Standard B;~~ they for oxygen in Calibrant B. The results should ~~be within~~ agree with the allowable limit 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 system. ~~instrument.~~

NOTE 234—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.

512. Procedure

512.1 Assemble the apparatus, calibrate, set the blank, and test the performance as directed in ~~48~~ 49 and 501.

512.2 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 512.5. Refer to the manufacturer's directions.)

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

512.4 Start the crucible degassing cycle (Note 245); refer to the manufacturer’s recommended procedure regarding entry of sample weight.

NOTE 245—For some instruments this procedure automatically precedes the analysis cycle.

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

512.6 Repeat 512.1-512.5 two additional times and report the average of the three determinations.

52. Calculation

52.1 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.

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

534.1 *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.

534.2 *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-IRREDARED ABSORPTION METHOD (CALIBRATION WITH METAL STANDARDS)

54. Scope

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

556.1 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.

556.1.1 *Infrared Absorption: Test Method A*— Sulfur dioxide (SO₂) 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 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.

556.1.2 *Infrared Absorption: Test Method B*— The combustion is performed in a closed loop in which 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. The IR absorption properties of the SO₂ gas in the chamber cause a loss of energy during combustion; a loss in

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.

signal, therefore, results that 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.

556.1.3 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 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 and 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.

56. Interferences

~~56.1 The elements normally present do not interfere.~~

57. Interferences

~~57.1 The elements normally present do not interfere.~~

58. Apparatus

~~578.1 Combustion and Measurement Apparatus~~—See Figs. 5-7.

~~578.2 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.

~~578.3 Crucible Tongs~~, capable of handling recommended crucibles.

589. Reagents

~~589.1 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.

~~589.2 Acetone~~—The residue after evaporation must be <0.0005 %.

~~589.3 Magnesium Perchlorate~~, (known commercially as *Anhydrone*).

~~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.

~~58.4~~

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

~~59.6 Tungsten Accelerator (Low Sulfur)~~, 12 to 20-mesh (Note 256).

~~NOTE 256~~—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.

59. Sample Preparation

~~59.1 The sample should be uniform in size but not finer than 40 mesh.~~

~~59.2 If necessary, wash in acetone and dry at 70 to 100°C.~~

60. Preparation of Apparatus

~~60.1 Assemble the apparatus as recommended by the manufacturer.~~

~~60.2 Test the furnace and analyzer to ensure the absence of leaks, and make the required electrical power connections. Prepare the analyzer for operation.~~

~~60.1 The sample should be uniform in accordance with the manufacturer's instructions. Make a minimum of two determinations using a specimen and accelerator, as directed size but not finer than 40 mesh.~~

~~60.2 If necessary, wash in 62.2 acetone and 62.3, before attempting dry at 70 to calibrate the system or determine the blank. 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

~~61.1 Calibration Standards~~

62.1 *Calibrants* (Note 267):

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

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

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

NOTE 26—~~The accuracy 27—The uncertainty of these results obtained using this test methods is dependent on the accuracy uncertainty of the methods used values assigned to certify the sulfur concentration in the calibration primary standard reference materials. The homogeneity of the reference materials must be considered as well, if it was not included in their derivation of the published uncertainty values.~~

612.2 *Adjustment of Response of Measurement System:*

612.2.1 Transfer 1.0 g of Standard 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 278).

612.2.2 Proceed as directed in 623.1.2 and 623.1.3.

612.2.3 Repeat the steps given in 612.2.1 and 612.2.2 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 ~~primary standard certified~~ reference material.

612.3 *Determination of Blank Readings—Range I:*

612.3.1 Transfer 1.0 g of Standard 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 278).

NOTE 278—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.

612.3.2 Proceed as directed in 623.1.2 and 623.1.3.

612.3.3 Repeat the steps given in 612.3.1 and 612.3.2 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 Standard Calibrant A minus the certified value for the ~~primary standard reference material~~.

612.3.4 *Record calibrant.*

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

612.3.5 If the blank readings are too high or inconsistent, determine the cause, correct it, and repeat the steps as directed in 612.3.1-612.3.4.

612.3.6 Enter the average blank value in the analyzer (Note 289); refer to the manufacturer's instructions. This mechanism will compensate electronically for the blank value.

NOTE 289—If the unit does not have this function, the blank value must be subtracted from the total result prior to any calculation.

612.4 *Determination of Blank Reading—Range II*—Proceed as directed in section 612.3.

612.5 *Determination of Blank Reading—Range III:*

612.5.1 Transfer 0.5 g of Standard 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 278).

612.5.2 Proceed as directed in 612.3.2-612.3.6.

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

612.6.1 Weigh four 1.0-g test samples of Standard 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 278), to each.

612.6.2 Follow the calibration procedure recommended by the manufacturer. Use Standard Calibrant C as the ~~primary calibration standard calibrant~~ and analyze at least three test samples to determine the calibration slope. Treat each test sample as directed in 623.1.2 and 623.1.3 before proceeding to the next one.

612.6.3 Confirm the calibration by analyzing Standard Calibrant C following the calibration procedure. The value should be within the allowable limits of the certified value for the ~~primary standard certified~~ reference material. If not, repeat the steps given in 612.6.1 and 612.6.2.

612.6.4 Weigh at least two 1.0-g test samples of Standard 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 278), to each.

612.6.5 Treat each test sample as directed in 623.1.2 and 623.1.3 before proceeding to the next one.

612.6.6 Record the results of 612.6.4 and 612.6.5 and compare them to the certified sulfur value of Standard Calibrant B. The results should be within the allowable limits of the ~~primary standard certified~~ reference material. If not, refer to the manufacturer's instructions for determining the linearity of the system (Note 29)–30).

NOTE 2930—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.

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

612.7.1 Proceed as directed in 612.6.1-612.6.3, using Standard Calibrant CC.

612.7.2 Proceed as directed in 612.6.4-612.6.6, using Standard Calibrant BB.

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

612.8.1 Weigh four 0.5-g test samples of Standard 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 278), to each.

612.8.2 Follow the calibration procedure recommended by the manufacturer. Use Standard Calibrant CCC as the primary ~~calibration standard~~ calibrant and analyze at least three test samples to determine the calibration slope. Treat each test sample as directed in 623.1.2 and 623.1.3 before proceeding to the next one.

612.8.3 Confirm the calibration by analyzing Standard Calibrant CCC following the calibration procedure. The value should be within the allowable limits of the certified value for the ~~primary standard~~ certified reference material. If not, repeat the steps given in 612.8.1 and 612.8.2.

612.8.4 Weigh at least two 0.5-g test samples of Standard 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 278), to each.

612.8.5 Treat each test sample as described in 623.1.2 and 623.1.3 before proceeding to the next one.

612.8.6 Record the results of 612.8.4 and 612.8.5 and compare them to the certified sulfur value of the Standard Calibrant BBB. The results should be within the allowable limits of the ~~primary standard~~ certified reference material. If not, refer to the manufacturer’s instructions for determining the linearity of the analyzer (Note 29).

62. 30).

63. Procedure

623.1 *Procedure—Range I:*

623.1.1 Stabilize the furnace and analyzer as directed in Section 58. 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 578.2) (Note 278).

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

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

623.2 *Procedure—Range II—*Proceed as directed in 623.1.

623.3 *Procedure—Range III—*Proceed as directed in 623.1, using a 0.5-g test sample.

634. Calculation

634.1 Since most commercially available instruments calculate percent concentrations directly, including corrections for blank and sample weight, calculations by the analyst are not required (Note 301).

NOTE 301—Use the following formula if the analyzer does not compensate for blank and sample weight values:

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

where:

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

645. Precision and Bias

645.1 *Precision*—Nine laboratories cooperated in testing these test methods and obtained the precision data summarized in Tables 9-11 9-11 (see Note 312).

NOTE 312—In Tables 9-11, all results in Ranges I and II are from nine laboratories. The results in Range III are from eight laboratories.

645.2 *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 323).

**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

NOTE 323—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 %.

656. Keywords

656.1 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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