



Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques¹

This standard is issued under the fixed designation E1019; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

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

Element	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
Niobium	0.002 to 6.00
Copper	0.005 to 10.00
Hydrogen	0.0001 to 0.0030
Iron	0.01 to 100.0
Lead	0.001 to 0.50
Magnesium	0.001 to 0.05
Manganese	0.01 to 20.0
Molybdenum	0.002 to 30.00
Nickel	0.005 to 84.00
Nitrogen	0.0005 to 0.50
Oxygen	0.0005 to 0.03
Phosphorus	0.001 to 0.90
Selenium	0.001 to 0.50
Silicon	0.001 to 6.00
Sulfur (Metal Reference Materials)	0.002 to 0.35
Sulfur (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 Test Method	10-20
Nitrogen by the Inert Gas Fusion–Thermal Conductivity Test Method	32-42
Oxygen by the Inert Gas Fusion Test Method	43-54
Sulfur by the Combustion–Infrared Absorption Test Method (Calibration with Metal Reference Materials)	55-65
Sulfur by the Combustion–Infrared Absorption Test Method (Potassium Sulfate Calibration)	21-31

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

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 6.

2. Referenced Documents

2.1 *ASTM Standards*:²

- D1193 Specification for Reagent Water
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

¹ These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferroalloys.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

3. Terminology

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

4. Significance and Use

4.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts, capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

5. Apparatus and Reagents

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

6. Hazards

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

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

7. Sampling

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

8. Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places as directed in Practice E29.

9. Interlaboratory Studies

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

TOTAL CARBON BY THE COMBUSTION INSTRUMENTAL MEASUREMENT TEST 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 Test 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, Test 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, Test Method B*—The detector consists of an IR energy source, a separate measure chamber and reference chamber, and a diaphragm acting as one plate of a parallel plate capacitor. During specimen combustion, the flow of CO_2 with its oxygen gas carrier 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, Test 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 at its maximum. During combustion, the IR absorption properties of CO and CO_2 gases in the chamber cause a loss of energy; therefore a loss in signal results which is proportional to concentrations 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 perform the above operations automatically and calibrated using steels of known carbon content.

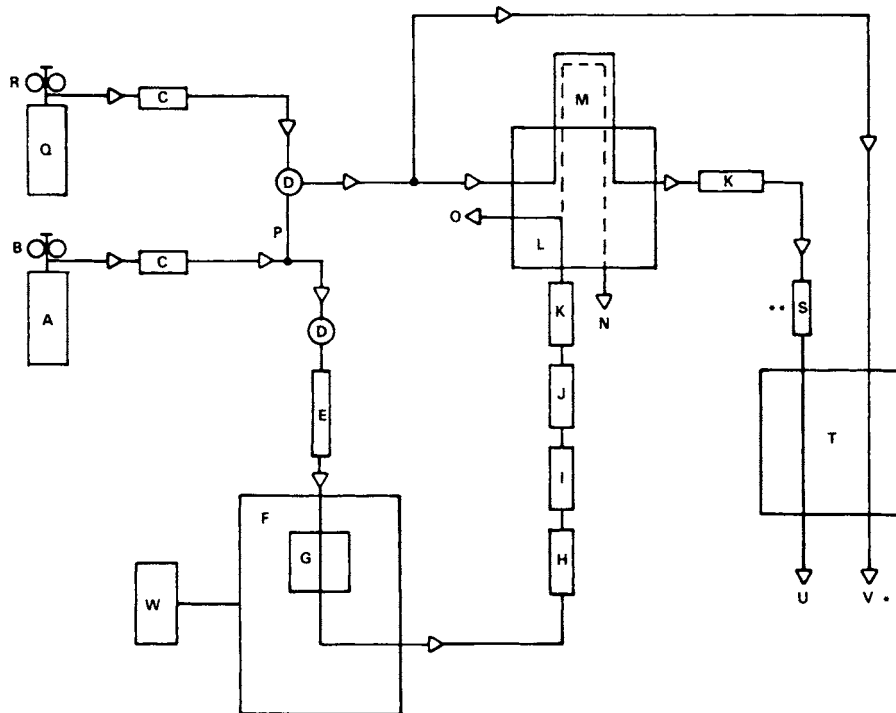
12. Interferences

12.1 For the scope of elements typically found in materials to be tested by this method refer to 1.1.

13. Apparatus

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

13.2 *Crucibles*—Use crucibles that meet or exceed the specifications of the instrument manufacturer and prepare the crucibles by heating in a suitable furnace for not less than 40 min at approximately 1000 °C. Remove from the furnace and cool before use. Crucibles may be stored in a desiccator prior to use. Heating of crucibles is particularly important when analyzing for low levels of carbon and may not be required if the material to be analyzed has higher levels of carbon such as



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| <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> |
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* 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 Test Method

that found in pig iron. Above certain concentrations, as determined by the testing laboratory, the nontreatment of crucibles will have no adverse effect. The analytical ranges for the use of untreated crucibles shall be determined by the testing laboratory and supporting data shall be maintained on file to validate these ranges.

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

14. Reagents

14.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society,

where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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

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

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K. (<http://uk.vwr.com>), and the *United States Pharmacopeia—National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD (<http://www.usp.org/USPNF>).