



# Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys<sup>1</sup>

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.

*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 These test methods<sup>2</sup> cover the determination of carbon, sulfur, nitrogen, and oxygen, in steel and in iron, nickel, and cobalt alloys having chemical compositions within the following limits:

Element	Concentration Range, %
Aluminum	0.001 to 18.00
Antimony	0.002 to 0.03
Arsenic	0.0005 to 0.10
Beryllium	0.001 to 0.05
Bismuth	0.001 to 0.50
Boron	0.0005 to 1.00
Cadmium	0.001 to 0.005
Calcium	0.001 to 0.05
Carbon	0.001 to 4.50
Cerium	0.005 to 0.05
Chromium	0.005 to 35.00
Cobalt	0.01 to 75.0
Columbium	0.002 to 6.00
Copper	0.005 to 10.00
Hydrogen	0.0001 to 0.0030
Iron	0.01 to 100.0
Lead	0.001 to 0.50
Magnesium	0.001 to 0.05
Manganese	0.01 to 20.0
Molybdenum	0.002 to 30.00
Nickel	0.005 to 84.00
Nitrogen	0.0005 to 0.50
Oxygen	0.0005 to 0.03
Phosphorus	0.001 to 0.90
Selenium	0.001 to 0.50
Silicon	0.001 to 6.00
Sulfur (Metal Reference Materials)	0.002 to 0.35
Sulfur (Potassium Sulfate)	0.001 to 0.600
Tantalum	0.001 to 10.00
Tellurium	0.001 to 0.35
Tin	0.002 to 0.35
Titanium	0.002 to 5.00
Tungsten	0.005 to 21.00
Vanadium	0.005 to 5.50

Zinc	0.005 to 0.20
Zirconium	0.005 to 2.500

1.2 The test methods appear in the following order:

Method	Sections
Carbon, Total, by the Combustion–Instrumental Measurement Method	10-20
Nitrogen by the Inert Gas Fusion–Thermal Conductivity Method	32-42
Oxygen by the Inert Gas Fusion Method	43-54
Sulfur by the Combustion–Infrared Absorption Method (Calibration with Metal Reference Materials)	55-65
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 6.

## 2. Referenced Documents<sup>3</sup>

### 2.1 ASTM Standards:

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

## 3. Terminology

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

<sup>1</sup> 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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<sup>2</sup> 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.

<sup>3</sup> 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.

#### 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 E 50.

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

#### 7. Sampling

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

#### 8. Rounding Calculated Values

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

#### 9. Interlaboratory Studies

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

### TOTAL CARBON BY THE COMBUSTION INSTRUMENTAL MEASUREMENT METHOD

#### 10. Scope

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

#### 11. Summary of Test Method

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

11.1.1 *Thermal Conductivity Method*—The carbon dioxide is absorbed on a suitable grade of zeolite, released by heating the zeolite, and swept by helium or oxygen into a chromatographic column. Upon elution, the amount of carbon dioxide is measured in a thermistor-type conductivity cell. Refer to Fig. 1.

11.1.2 *Infrared (IR) Absorption, Method A*—The amount of carbon dioxide is measured by infrared (IR) absorption. Carbon dioxide ( $\text{CO}_2$ ) 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  $\text{CO}_2$  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  $\text{CO}_2$ , is monitored and measured over a period of time. Refer to Fig. 2.

11.1.3 *Infrared (IR) Absorption, Method B*—The detector consists of an IR energy source, a separate measure chamber and reference chamber, and a diaphragm acting as one plate of a parallel plate capacitor. During specimen combustion, the flow of  $\text{CO}_2$  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  $\text{CO}_2$  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  $\text{CO}_2$ . Total carbon, as  $\text{CO}_2$ , is monitored and measured over a period of time. Refer to Fig. 3.

11.1.4 *Infrared (IR) Absorption, Method C, Closed Loop*—The combustion is performed in a closed loop, where CO and  $\text{CO}_2$  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  $\text{CO}_2$ , the energy received by each detector is maximum. During combustion, the IR absorption properties of CO and  $\text{CO}_2$  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  $\text{CO}_2$  plus CO, is monitored and measured over a period of time. Refer to Fig. 4.

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

#### 12. Interferences

12.1 The elements ordinarily present do not interfere.

#### 13. Apparatus

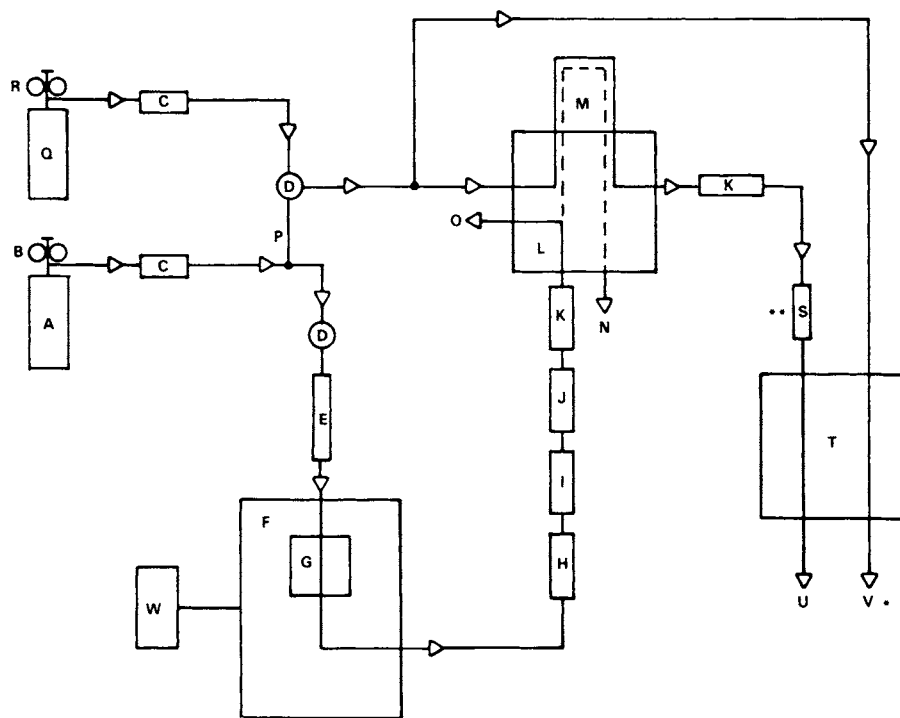
13.1 *Combustion and Measurement Apparatus*—See Figs. 1-4.

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

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

#### 14. Reagents

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



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 CO<sub>2</sub> Converter  
 K—Magnesium Perchlorate  
 L—Valve Manifold

M—CO<sub>2</sub> Collection Trap  
 N—Furnace Combustion Exhaust  
 O—Furnace Purge Exhaust  
 P—Metal Connector To Use Oxygen As Carrier Gas  
 Q—High Purity Helium  
 R—Helium Regulator (2 Stage)  
 S—Chromatographic Column  
 T—TC Cell/Readout  
 U—Measure Flowmeter  
 V—Reference Flowmeter  
 W—Furnace Power Stat

\* May be sealed chamber if oxygen is carrier gas.

\*\* Not required if oxygen is carrier gas.

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

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

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

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

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

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

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

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

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

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

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

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