Designation: E1019 – 18

Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Inert Gas Fusion Techniques[1]

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.

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

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	Mass Fraction 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	0.002 to 0.35
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 and Infrared Absorption or Thermal Conductivity Detection Test Method	10 – 20
Nitrogen by the Inert Gas Fusion and Thermal Conductivity Detection Test Method	32 – 42
Oxygen by the Inert Gas Fusion and Infrared Absorption or Thermal Conductivity Detection Test Method	43 – 54
Sulfur by the Combustion-Infrared Absorption Detection Test Method	55 – 65
Sulfur by the Combustion–Infrared Absorption Test Method (Potassium Sulfate Calibration) – *Discontinued 2018*	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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazards statements are given in Section 6.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

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

Current edition approved April 15, 2018. Published June 2018. Originally approved in 1984. Last previous edition approved in 2011 as E1019 – 11. DOI: 10.1520/E1019-18.

[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.

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 Metals (Withdrawn 1998)[3]
E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method
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.

5.2 These methods were originally developed for older technology manual instrumentation with the flow schematics indicated. Current commercially available instruments are more automated and may have slightly different flow schematics and should be capable of producing data meeting or exceeding the precision and bias requirements.

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 to sample the materials, refer to those parts of Practice E1806.

8. Rounding Calculated Values

8.1 Rounding of test results obtained using these test methods shall be performed as directed in Practice E29, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

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

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 AND INFRARED ABSORPTION OR THERMAL CONDUCTIVITY DETECTION TEST METHOD

10. Scope

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

11. Summary of Test Method

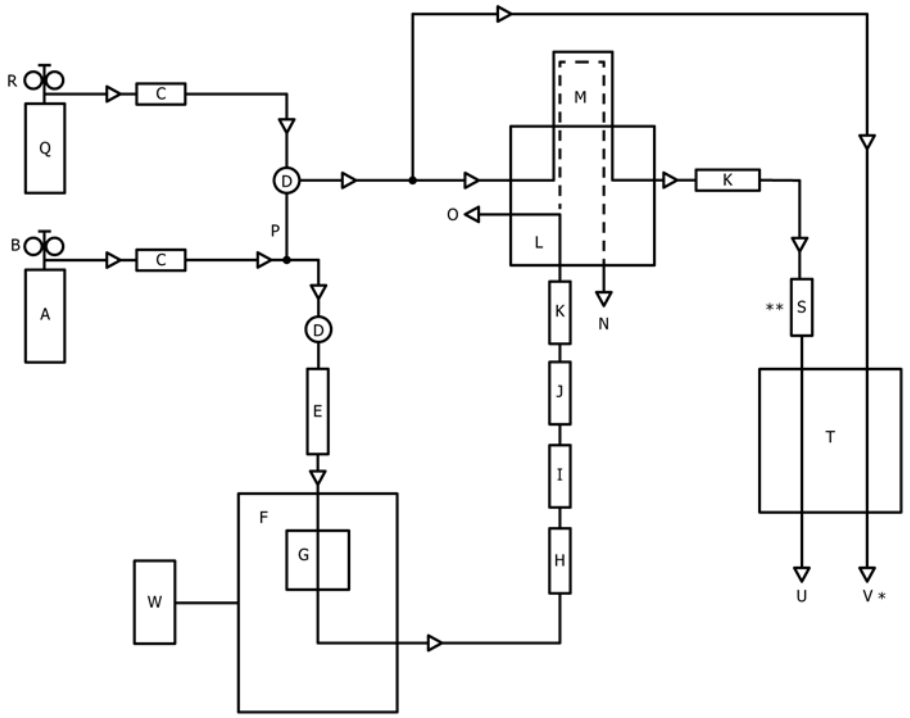
11.1 The carbon is converted to carbon dioxide (CO_2) by combustion in a stream of oxygen.

11.1.1 *Thermal Conductivity Test Method*—The CO_2 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 CO_2 is measured in a thermistor-type conductivity cell. Refer to Fig. 1 for example.

11.1.2 *Infrared (IR) Absorption, Test Method A*—The amount of CO_2 is measured by infrared (IR) absorption. 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 amount 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 measured over a period of time. Refer to Fig. 2 for example.

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 carrier gas 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 capacitance creating an electric signal change that is amplified for measurement as CO_2. Total carbon, as CO_2, is measured over a period of time. Refer to Fig. 3 for example.

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



A—High Purity Oxygen
B—Oxygen Regulator (2 Stage)
C—Sodium Hydroxide Impregnated Clay and 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_2 Converter (suitable catalyst)
K—Magnesium Perchlorate (Note 1 in 14.4)
L—Valve Manifold
M—CO_2 Absorber – Zeolite
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 Supply

* 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 Detection Test Method

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 amounts of each gas in the closed loop. Total carbon, as CO_2 plus CO, is measured over a period of time. Refer to Fig. 4 for example.

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

12. Interferences

12.1 The elements ordinarily present in iron, steel, nickel, and cobalt alloys do not interfere.

13. Apparatus

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

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.

13.2.1 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. Heating of crucibles is particularly important when