



Designation: E415 – 21

# Standard Test Method for Analysis of Carbon and Low-Alloy Steel by Spark Atomic Emission Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E415; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the simultaneous determination of 21 alloying and residual elements in carbon and low-alloy steels by spark atomic emission vacuum spectrometry in the mass fraction ranges shown **Note 1**.

Element	Composition Range, %	
	Applicable Range, Mass Fraction % <sup>A</sup>	Quantitative Range, Mass Fraction % <sup>B</sup>
Aluminum	0 to 0.093	0.006 to 0.093
Antimony	0 to 0.027	0.006 to 0.027
Arsenic	0 to 0.1	0.003 to 0.1
Boron	0 to 0.007	0.0004 to 0.007
Calcium	0 to 0.003	0.002 to 0.003
Carbon	0 to 1.1	0.02 to 1.1
Chromium	0 to 8.2	0.007 to 8.14
Cobalt	0 to 0.20	0.006 to 0.20
Copper	0 to 0.5	0.006 to 0.5
Lead <sup>C</sup>	0 to 0.2	0.002 to 0.2
Manganese	0 to 2.0	0.03 to 2.0
Molybdenum	0 to 1.3	0.007 to 1.3
Nickel	0 to 5.0	0.006 to 5.0
Niobium	0 to 0.12	0.003 to 0.12
Nitrogen	0 to 0.015	0.01 to 0.055
Phosphorous	0 to 0.085	0.006 to 0.085
Silicon	0 to 1.54	0.02 to 1.54
Sulfur	0 to 0.055	0.001 to 0.055
Tin	0 to 0.061	0.005 to 0.061
Titanium	0 to 0.2	0.001 to 0.2
Vanadium	0 to 0.3	0.003 to 0.3
Zirconium	0 to 0.05	0.01 to 0.05

<sup>A</sup> Applicable range in accordance with Guide E1763 for results reported in accordance with Practice E1950.

<sup>B</sup> Quantitative range in accordance with Practice E1601.

<sup>C</sup> Newly added element, refer to 15.4 and Table 3.

NOTE 1—The mass fraction ranges of the elements listed have been established through cooperative testing<sup>2</sup> of reference materials.

1.2 This test method covers analysis of specimens having a diameter adequate to overlap and seal the bore of the spark

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.01 on Iron, Steel, and Ferroalloys.

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<sup>2</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1122. Contact ASTM Customer Service at service@astm.org.

stand opening. The specimen thickness can vary significantly according to the design of the spectrometer stand, but a thickness between 10 mm and 38 mm has been found to be most practical.

1.3 This test method covers the routine control analysis in iron and steelmaking operations and the analysis of processed material. It is designed for chill-cast, rolled, and forged specimens. Better performance is expected when reference materials and specimens are of similar metallurgical condition and composition. However, it is not required for all applications of 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.*

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:<sup>3</sup>

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E305 Practice for Establishing and Controlling Spark Atomic Emission Spectrochemical Analytical Curves

E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron

E406 Practice for Using Controlled Atmospheres in Atomic Emission Spectrometry

E691 Practice for Conducting an Interlaboratory Study to

<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.

Determine the Precision of a Test Method

**E1019** 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

**E1329** Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)<sup>4</sup>

**E1601** Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

**E1763** Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods (Withdrawn 2015)<sup>4</sup>

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

**E1950** Practice for Reporting Results from Methods of Chemical Analysis

**E2972** Guide for Production, Testing, and Value Assignment of In-House Reference Materials for Metals, Ores, and Other Related Materials

2.2 *Other ASTM Document:*<sup>5</sup>

**ASTM MNL 7** Manual on Presentation of Data and Control Chart Analysis

### 3. Terminology

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

### 4. Summary of Test Method

4.1 A capacitor discharge is produced between the flat, ground surface of the disk specimen and a conically shaped electrode. The discharge is terminated at a predetermined intensity time integral of a selected iron line, or at a predetermined time, and the relative radiant energies of the analytical lines are recorded. The most sensitive lines of arsenic, boron, carbon, nitrogen, phosphorus, sulfur, and tin lie in the vacuum ultraviolet region. The absorption of the radiation by air in this region is overcome by evacuating the spectrometer or by use of a vacuum ultraviolet (VUV) transparent gas and flushing the spark chamber with argon.

### 5. Significance and Use

5.1 This test method for the spectrometric analysis of metals and alloys is primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use this test method will be analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

### 6. Apparatus

6.1 *Sampling Devices:*

6.1.1 Refer to Practice **E1806** for devices and practices to sample liquid and solid iron and steel.

6.2 *Excitation Source*, capable of providing electrical parameters to spark a sample. See **11.1** for details.

6.3 *Spark Chamber*, automatically flushed with argon. The spark chamber shall be mounted directly on the spectrometer and shall be provided with a spark stand to hold a flat specimen and a lower counter electrode of rod form.

6.3.1 Follow the manufacturer's recommendations for cleaning the spark chamber. During continuous operation, this typically should be done every 24 h. Follow the manufacturer's recommendations for cleaning the entrance lens or window (verifier data or other reference sample intensity data can typically indicate when this is necessary).

6.4 *Spectral Lines*—**Table 1** lists spectral lines and internal standards usable for carbon and low alloy steel. The spectrometer must be able to measure at least one of the listed spectral lines for each of the listed elements. Spectral lines other than those listed in **Table 1** may be used provided it can be shown experimentally that equivalent precision and accuracy are obtained.

6.5 *Measuring System*, spectrometer capable of converting light intensities to measurable electrical signals. The measuring system may consist of one of the following configurations:

6.5.1 A photomultiplier (PMT) array having individual voltage adjustments, capacitors in which the output of each photomultiplier is stored, a voltage measuring system to register the voltages on the capacitors either directly or indirectly, and the necessary switching arrangements to provide the desired sequence of operation.

6.5.2 A semiconductor detector array (CCD or CMOS), pixel selection electronics to reset the pixels and to transport the voltage of an individual pixel to one or more output ports of the detector arrays, and a voltage measuring system to register the voltage of said output ports.

6.5.3 A hybrid design using both photomultipliers and semiconductor arrays.

6.6 *Optical Path*—If the instrument is operated using a VUV transparent gas, check the manufacturer's suggested gas purity. It may be necessary to have a gas purification system consisting of a circulation pump and a cleaning cartridge to keep the O<sub>2</sub> (g) residual <500 ng/g and H<sub>2</sub>O (g) residual <1 µg/g and remove impurities of nitrogen and hydrocarbons. If the instrument is using a vacuum pump, it should be capable of maintaining a vacuum of 3.33 Pa (25 µm Hg) or less.

NOTE 2—A pump with a displacement of at least 0.23 m<sup>3</sup>/min (8 ft<sup>3</sup>/min) is usually adequate.

6.7 *Gas System*, consisting of an argon supply with pressure and flow regulation. Automatic sequencing shall be provided to actuate the flow at a given rate for a specific time interval. The flow rate may be manually or automatically set. The argon system shall be in accordance with Practice **E406**.

### 7. Reagents and Materials

7.1 *Counter Electrodes*—The counter electrodes can be silver or tungsten rods, or other material, provided it can be shown experimentally that equivalent precision and bias are obtained. The rods can vary in diameter from 1.5 mm to 6.5

<sup>4</sup>The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>5</sup>ASTM Manual Series, ASTM International, 8th edition, 2010.

**TABLE 1 Internal Standard and Analytical Lines**

Element	Wavelength, $\lambda$ , nm	Line Classification <sup>A</sup>	Possible Interference <sup>B</sup>
Aluminum	396.15	I	Mo
	394.40	I	V, Mn, Mo, Ni
	308.22	I	V, Mn
Antimony	217.6	I	Ni, Nb, Mn, W
Arsenic	189.04	I	V, Cr
	197.20	I	Mo, W
	193.76	I	Mn
Boron	345.13	II	
	182.64	I	S, Mn, Mo
	182.59	I	W, Mn, Cu
Calcium	393.37	II	
	396.85	II	Nb
Carbon	165.81	I	Cr
	193.09	I	Al
Chromium	312.26	II	V
	313.21	II	
	425.44	I	
	298.92	II	Mn, V, Ni, Nb, Mo
	267.72	II	Mn, Mo, W
Cobalt	345.35	I	Cr, Mo
	228.62	II	Ni, Cr
	258.03	II	Fe, Mn, W
Copper	212.3	II	Si
	324.75	I	Mn, Nb
	327.40	I	Nb
	224.26	II	W, Ni
	213.60	II	Mo, Cr
	510.55	I	W
	136.14	II	
	157.40	II	
	172.24	II	
	174.28	II	
	179.34	I	
	182.88	II	
	205.13	I	
	216.20	I	
	217.81	I	
	218.65	II	
	226.76	II	
	235.12	II	
	239.15	I	
	277.21	I	
	281.33	I	
	285.18	I	
	296.69	II	
297.05	I		
299.95	I		
300.81	I		
303.74	I		
304.76	I		
Iron (IS)	305.91	I	
	316.79	I	
	517.16	I	
	321.33	II	
	487.21	I	
	458.38	II	
	413.70	I	
	410.75	I	
	383.63	I	
	363.83	I	
	339.93	I	
	328.68	I	
	308.37	I	
	282.33	I	
249.59	I		