



Designation: **E415 – 14 E415 – 15**

Standard Test Method for Analysis of Carbon and Low-Alloy Steel by Spark Atomic Emission Spectrometry¹

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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the simultaneous determination of ~~2021~~ 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 % ^A	Quantitative Range, Mass Fraction % ^B
Aluminum	0 to 0.075	0.02 to 0.075
Arsenic	0 to 0.1	0.05 to 0.1
Boron	0 to 0.007	0.002 to 0.007
Calcium	0 to 0.003	0.001 to 0.003
Carbon	0 to 1.1	0.08 to 1.1
Chromium	0 to 2.25	0.02 to 2.25
Cobalt	0 to 0.18	0.008 to 0.18
Copper	0 to 0.5	0.04 to 0.5
Manganese	0 to 2.0	0.10 to 2.0
Molybdenum	0 to 0.6	0.03 to 0.6
Nickel	0 to 5.0	0.02 to 5.0
Niobium	0 to 0.085	0.02 to 0.085
Nitrogen	0 to 0.015	0.004 to 0.015
Phosphorous	0 to 0.085	0.02 to 0.085
Silicon	0 to 1.15	0.07 to 1.15
Sulfur	0 to 0.055	0.01 to 0.055
Tin	0 to 0.045	0.01 to 0.045
Titanium	0 to 0.2	0.004 to 0.2
Vanadium	0 to 0.3	0.004 to 0.3
Zirconium	0 to 0.05	0.02 to 0.05

Element	Composition Range, %	
	Applicable Range, Mass Fraction % ^A	Quantitative Range, Mass Fraction % ^B
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
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

¹ 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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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

^A Applicable range in accordance with Guide E1763 for results reported in accordance with Practice E1950.

^B Quantitative range in accordance with Practice E1601.

NOTE 1—The mass fraction ranges of the elements listed have been established through cooperative testing² of reference materials. ~~Included, in addition to the original data of Test Method E415 – 71, are data from cooperative testing of a broader range of reference materials to expand the element mass fraction ranges.~~

1.2 This test method covers analysis of specimens having a diameter adequate to overlap and seal the bore of the spark 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:³

[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 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 Spectrochemical Analysis](#)

[E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques](#)

[E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis](#)

[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 \(Withdrawn 2015\)⁴](#)

[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 Documents

[ASTM MNL 7 Manual on Presentation of Data and Control Chart Analysis⁵](#)

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.

² Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report [RR-E2-1004RR:E01-1122](#). Contact ASTM Customer Service at service@astm.org.

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

⁴ The last approved version of this historical standard is referenced on www.astm.org.

⁵ ASTM Manual Series, ASTM International, 8th edition, 2010.

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 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 Spectrometer, Spectral Lines—having Table 1 a reciprocal linear dispersion of 0.60 nm/mm, or better, in the first order and a focal length of 0.75 m to 3 m. Its approximate range shall be from 145.0 nm to 400.0 nm. Masks shall be provided in the spectrometer to eliminate scattered radiation. The spectrometer shall be provided with an air inlet and a vacuum outlet. The spectrometer shall be operated at a vacuum of 3.33 Pa (25 lists spectral lines and internal standards usable for carbon and low alloy steel. The spectrometer must be able μm of mercury) or below. The primary slit width can range from 20 μm to 50 measure μm . Secondary slit widths—least one of the listed spectral lines for each of the listed elements. Spectral lines other than those listed in Table 1 can vary (normally between 37 may be used provided it can be shown experimentally that $\mu\text{equivalent}$ —precision and 200 accuracy μare) depending on the element wavelength and possible interfering wavelengths. obtained.

6.5 Measuring System, consisting of photomultipliers (spectrometer capable of converting light Note 2) 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. intensities to measurable electrical signals. The measuring system may consist of one of the following configurations:

NOTE 2—Spectrometers equipped with CCD and/or CID detectors may be used provided the user can verify that the data obtained are equivalent to the precision and bias statistics in this method.

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 Vacuum Pump, 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 O₂ (g) residual <500 ng/g and H₂O (g) residual <1 $\mu\text{g/g}$ 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 m-Hg) or less.

NOTE 2—A pump with a displacement of at least 0.23 m³/min (8 ft³/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 controlled. 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 thoriated tungsten rods, or other material, provided it can be shown experimentally that equivalent precision and bias is are obtained. The rods can vary in diameter from 1.5 mm to 6.5 mm (depending on the instrument manufacturer) design) and typically are machined to a 90° or 120° angled tip.

7.1.1 A black deposit will collect on the tip of the electrode. This deposit should be removed between specimens (typically with a wire brush). If not removed, it can reduce the overall intensity of the spectral radiation or transfer slight amounts of contamination between specimens, or both. The number of acceptable excitations burns on an electrode varies from one instrument to another, and should be established in each laboratory.

NOTE 3—It has been reported that thousands of excitations burns can be performed on a thoriated tungsten electrode before replacement is necessary.

7.2 Inert Gas, Argon, in accordance with Practice E406.

TABLE 1 Internal Standard and Analytical Lines

Element	Wavelength, nm	Line Classification ^A	Possible Interference ^B
Aluminum	394.40	I	V, Mn, Mo, Ni
	308.22	I	V, Mn
Arsenic	197.20	I	Mo, W
	193.76	I	Mn
Boron	182.64	I	S, Mn, Mo
	182.59	I	W, Mn, Cu
Calcium	396.85	II	Nb
Carbon	193.09	I	Al
Chromium	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
Copper	327.40	I	Nb
	213.60	II	Mo, Cr
Iron (IS)	271.44	II	
	273.07	II	Co
Manganese	293.31	II	Cr, Mo, Ni
	255.86	II	Zr
Molybdenum	379.83	II	Mn
	277.54	I	Cu, V, Co, Mn
Nickel	386.41	I	V, Cr
	231.60	II	Co, Ti
Niobium	227.02	II	Nb, W
	319.50	II	Mo, Al, V
Nitrogen	149.26	I	Fe, Ti, Si, Mn, Cu, Ni and nitride-forming elements such as Ti
			Mo
Phosphorus	178.29	I	Mo, Cr, W
Silicon	288.16	I	Fe, V
	251.61	I	Mn
Sulfur	180.73	I	Mn, Mo, Al
Tin	189.99	II	Nb
Titanium	337.28	II	Nb
	324.20	II	Nb
Vanadium	310.23	II	Fe, Mo, Nb, Ni
	311.07	II	Mn, Ti, Fe
Zirconium	343.82	II	W

TABLE 1 Internal Standard and Analytical Lines

Element	Wavelength, λ , nm	Line Classification ^A	Possible Interference ^B
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

TABLE 1 *Continued*

Element	Wavelength, λ , nm	Line Classification ^A	Possible Interference ^B
	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	
<u>Iron (IS)</u>	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	
	226.76	II	
	218.65	II	
	216.20	I	
	193.53	II	
	190.48	I	
	187.75	II	
	149.65	II	
	271.44	II	
	273.07	II	
	492.39	I	Co
<u>Lead</u>	405.75	I	Mn
<u>Manganese</u>	293.31	II	Cr, Mo, Ni
	255.86	II	Zr
	263.82	II	Al, W
<u>Molybdenum</u>	379.83	II	Mn
	202.03	II	
	277.54	I	Cu, V, Co, Mn
	281.61	II	Mn
	386.41	I	V, Cr
<u>Nickel</u>	471.44	I	
	227.73	II	
	341.48	I	
	352.45	I	
	231.60	II	Co, Ti
	227.02	II	Nb, W
	243.79	II	Co, Fe, Ni
<u>Niobium</u>	313.08	II	Ti, V

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TABLE 1 *Continued*

Element	Wavelength, λ , nm	Line Classification ^A	Possible Interference ^B
	<u>319.50</u>	II	Mo, Al, V
Nitrogen	<u>149.26</u>	I	Fe, Ti, Si, Mn, Cu, Ni and nitride forming elements such as Ti
Phosphorus	<u>178.29</u>	I	Mo
Silicon	<u>288.16</u>	I	Mo, Cr, W
	<u>251.61</u>	I	Fe, V
	<u>212.41</u>	I	Mo, Ni, V, Cu, Nb
	<u>390.55</u>	I	Cr, Cu, W, Ti
Sulfur	<u>180.73</u>	I	Mn
Tin	<u>147.52</u>	II	
	<u>189.99</u>	II	Mn, Mo, Al
Titanium	<u>308.80</u>	I	Cu, Co
	<u>337.28</u>	II	Nb
Tungsten	<u>324.20</u>	II	Nb
	<u>400.88</u>	I	
	<u>202.99</u>	II	Ti, V, Mn
	<u>220.50</u>	II	Co
Vanadium	<u>437.92</u>	I	
	<u>310.23</u>	II	Fe, Mo, Nb, Ni
Zirconium	<u>468.78</u>	I	
	<u>349.62</u>	II	
	<u>343.82</u>	II	W
	<u>206.19</u>	II	W

^A The numerals I or II in the line classification column indicate that the line has been classified in a term array and definitely assigned to the normal atom (I) or to the singly ionized atom (II).

^B Interferences are dependent upon instrument design, spectrum line choices, and excitation conditions, and those listed require confirmation based upon specimens selected especially to demonstrate suspected interferences.

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8. Reference Materials

8.1 *Certified Reference Materials (CRMs)*—These are available from the National Institute of Standards and Technology (NIST) and other sources and span all or part of the mass fraction ranges listed in 1.1. They are used to calibrate the spectrometer for the elements of interest or to validate the performance of the test method. It is not recommended to use CRMs as verifiers or to establish the repeatability of the chemical measurement process. Differences can occur between CRMs and production samples prepared by the sampling procedures recommended in this test method. Certain element's (for example, sulfur) calibrations may need to be corrected with values from reference materials made by normal production sampling techniques and analyzed by Test Methods E350 and E1019.

NOTE 4—Certified Reference Materials manufactured by NIST are trademarked with the name, “Standard Reference Materials, SRMs.” Material.”

8.2 *Reference Materials (RMs)*—These are available from multiple suppliers or can be developed in house. RMs—Reference Materials are typically used to in control procedures (verifiers) and drift correct (standardants) the spectrometer. in drift correction (standardization) of the spectrometer, and they may be useful in calibrations. These reference materials shall be homogenous and contain appropriate compositions—mass fractions of each element to be controlled for the intended purpose. Refer to Guide E2972 or drift corrected, or both for production of your own reference materials.

8.3 Several issues can impact the selection and use of CRMs and RMs:

8.3.1 Samples and reference materials may exhibit differences in metallurgical structure, in particular having different sizes, compositions, and distributions of inclusions. Inhomogeneous distribution of inclusions can worsen repeatability of individual measurements of elements found in the inclusions. Some inclusions may be removed during preburn steps prior to integration of intensities, causing low results. Typical samples can be used to determine repeatability of individual measurements to yield estimates consistent with performance for actual samples.

8.3.2 For certain elements, there may be no available reference materials with metallurgical structure similar to typical samples. Therefore, calibrations may be biased. It is recommended to validate results using typical samples analyzed using Test Methods E350 and E1019.