Designation: E1999 – 23

Standard Test Method for Analysis of Cast Iron by Spark Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation E1999; 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 analysis of cast iron by spark atomic emission spectrometry for the following elements in the ranges shown (Note 1):

Elements	Ranges, % Applicable Range, %	Quantitative Range, %[A]
Carbon	1.9 to 3.8	1.90 to 3.8
Chromium	0 to 2.0	0.025 to 2.0
Copper	0 to 0.75	0.015 to 0.75
Manganese	0 to 1.8	0.03 to 1.8
Molybdenum	0 to 1.2	0.01 to 1.2
Nickel	0 to 2.0	0.02 to 2.0
Phosphorus	0 to 0.4	0.005 to 0.4
Silicon	0 to 2.5	0.15 to 2.5
Sulfur	0 to 0.08	0.01 to 0.08
Tin	0 to 0.14	0.004 to 0.14
Titanium	0 to 0.12	0.003 to 0.12
Vanadium	0 to 0.22	0.008 to 0.22

[A]Quantitative range as directed in Practice E1601.

NOTE 1—The ranges of the elements listed have been established through cooperative testing of reference materials. These ranges can be extended by the use of suitable reference materials.

1.2 This test method covers analysis of specimens having a diameter adequate to overlap the bore of the spark stand opening (to effect an argon seal). The specimen thickness should be sufficient to prevent overheating during excitation. A heat sink backing may be used. The maximum thickness is limited only by the height that the stand will permit.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

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

E406 Practice for Using Controlled Atmospheres in Atomic Emission Spectrometry

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry (Withdrawn 2023)[3]

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis (Withdrawn 2019)[3]

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)[3]

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

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

2.2 *Other Documents:*

MNL 7 Manual on Presentation of Data and Control Chart Analysis[4]

3. Terminology

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

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

[4] ASTM Manual Series, ASTM International, 8th Edition, 2010.

4. Summary of Test Method

4.1 A capacitor discharge is produced between the flat, prepared surface of the disk specimen and a conically shaped electrode. The discharge is terminated at a predetermined intensity of a selected iron line, or at a predetermined time, and the relative radiant energies of the analytical lines are recorded and converted to mass fractions.

4.2 Carbon, phosphorus, sulfur and tin emit in the vacuum ultraviolet region. The absorption of the radiation by air in this region is overcome by flushing the spark chamber with argon or an argon-hydrogen gas mixture and either evacuating the spectrometer or filling the spectrometer with an inert gas such as nitrogen or argon.

NOTE 2—It is not within the scope of this test method to prescribe specific details of every instrument that could be used for the analysis of cast iron by spark atomic emission spectrometry. The parameters listed in this test method represent the parameters of the specific instruments used during the interlaboratory study to produce the precision and bias listed in this test method. Other spark atomic emission spectrometers with different parameters may be used provided that they produce equivalent or better precision and bias data.

5. Significance and Use

5.1 The chemical composition of cast iron alloys shall be determined accurately in order to ensure the desired metallurgical properties. This procedure is suitable for manufacturing control and inspection testing.

6. Interferences

6.1 Interferences may vary with spectrometer design and excitation characteristics. Direct spectral interferences may be present on one or more of the wavelengths listed in this test method. Frequently, these interferences shall be determined and proper corrections made using various reference materials. Refer to Table 1 for possible interferences. The composition of the sample being analyzed should match closely the composition of one or more of the reference materials used to prepare and control the calibrations. Alternatively, mathematical corrections may be used to solve for interelement effects. Various mathematical correction procedures are commonly utilized. Any of these correction procedures are acceptable that produce precision and accuracy results equal to or better than the results in the interlaboratory study for this test method.

TABLE 1 Analytical and Internal Standard Lines, Possible Interferences

Element	Wavelength, nm	Reported Possible Interfering Elements
Carbon	193.09	Al, Mo, Cu, S
Chromium	267.72	Mo, S, Mn
	265.86	
Copper	211.21	Ni
	221.81	
	327.40	Mo, P
	510.55	V
Manganese	293.31	Cr, Mo, W
Molybdenum	202.03	Ni
	281.61	Mn
Nickel	243.79	Mn
	231.60	Mn
	341.48	
	352.45	Mo
Phosphorus	178.29	Cr, Mn, Mo, Cu
Silicon	212.41	Mo, Cu, Ni
	251.61	
	288.16	Mo, Cr
Sulfur	180.73	Mn, Cu, Cr
Tin	189.99	Mn, Mo, Fe
Titanium	334.90	Cr
	337.28	Fe
	334.19	
Vanadium	310.23	Ni
	311.07	
Iron[A]	273.07	
	271.44	
	281.33	
	360.89	

[A] Internal standard.

7. Apparatus

7.1 When required, use sample preparation equipment as follows:

7.1.1 *Sample Mold,* to produce graphite-free white chilled iron samples that are homogeneous, free of voids or porosity in the region to be excited, and representative of the material to be analyzed. A chill-cast disk approximately 40 mm (1 ½ in.) in diameter and 3-mm to 12-mm (⅛-in. to ½-in.) thick is satisfactory. A sample mold made from copper with a low oxygen content has proven to be optimum for this purpose. Refer to Practice E1806 for iron sampling procedures.

7.1.2 *Surface Grinder or Sander with Abrasive Belts or Disks,* capable of providing a flat, clean, uniform surface on the reference materials and specimens.

7.2 *Excitation Source,* capable of providing sufficient energy to sample the specimen and excite the analytes of interest. Excitation sources whose performance has been proven to be equivalent may be used.

7.3 *Excitation Chamber,* automatically flushed with argon or other inert gas. Clean the excitation chamber when the counter electrode is replaced.

7.3.1 Clean the lens or protective window as recommended by the instrument manufacturer.

7.4 *Spectrometer,* having sufficient resolving power and linear dispersion to separate clearly the analytical lines from other lines in the spectrum in the spectral region 170.0 nm to 520.0 nm. The spectrometers used to test this method had a dispersion of 0.3 nm/mm to 0.6 nm/mm and a focal length of 0.5 m to 0.75 m. Spectral lines are listed in Table 1. The primary slit width is 15 µm to 50 µm. Secondary slit width is 15 µm to 200 µm. The spectrometer shall be provided with one or more of the following: