Designation: E1999 – 99 (Reapproved 2004)

Standard Test Method for Analysis of Cast Iron Using Optical 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 optical emission spectrometric analysis of cast iron by use of the point-to-plane technique for the following elements in the concentration ranges shown (Note 1):

Elements	Concentration 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 in accordance with Practice E1601.

NOTE 1—The concentration ranges of the elements listed have been established through cooperative testing of reference materials. These concentration 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

E158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis[3]

E172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis[3]

E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves

E351 Test Methods for Chemical Analysis of Cast Iron—All Types

E406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry

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

E1059 Practice for Designating Shapes and Sizes of Nongraphite Counter Electrodes

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

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

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] Withdrawn.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

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.

4. Summary of Test Method

4.1 The most sensitive lines for carbon, phosphorus, sulfur and tin lie in the ultraviolet region. The absorption of the radiation by air in this region is overcome by flushing the spark chamber with argon or argon-hydrogen gas mixture and either evaluating all or portions of the spectrometer or filling all or portions of the spectrometer with an inert gas. 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 of a selected iron line, or at a predetermined time, and the relative radiant energies of the analytical lines are recorded and converted to concentration.

5. Significance and Use

5.1 The chemical composition of cast iron alloys must be determined accurately in order to insure 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 a method. Frequently, these interferences must be determined and proper corrections made by the use of various reference materials. 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 calibration curve which is employed. Alternatively, mathematical corrections may be used to solve for interelement effects (refer to Practice E158). Various mathematical correction procedures are commonly utilized. Any of these is acceptable, which will achieve analytical accuracy equivalent to that provided by this test method.

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 to 12-mm (⅛ 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. See Practice E172. Any other excitation source whose performance has been proven to be equivalent may be used.

7.3 *Excitation Chamber*, automatically flushed with argon or other inert support gas. Gases and electrodes are described in 8.1 and 8.2.

NOTE 2—Clean the excitation chamber when the counter electrode is replaced. Clean the lens or protective window after approximately 200 to 300 excitations, or at a statistically determined time based on intensity loss, to minimize transmission losses.

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 to 500.0 nm. The spectrometers used to test this method had a dispersion of 0.3 to 0.6 nm/mm and a focal length of 0.5 to 0.75 m. Spectral lines are listed in Table 1. The primary slit width is 15

TABLE 1 Analytical and Internal Standard Lines, Possible Interference

Element	Wavelength, nm	Reported Possible Interfering Elements
Carbon	193.093	A1, Mo, Cu, S
Chromium	267.716	Mo, S, Mn
	265.859	
Copper	211.209	Ni
	221.81	
	327.4	Mo, P
	510.5	V
Manganese	293.306	Cr, Mo, W
Molybdenum	202.03	Ni
	281.61	Mn
Nickel	243.789	Mn
	231.604	Mn
	341.4	
	352.45	Mo
Phosphorus	178.287	Cr, Mn, Mo, Cu
Silicon	212.411	Mo, Cu, Ni
	251.612	
	288.16	Mo, Cr
Sulfur	180.731	Mn, Cu, Cr
Tin	189.989	Mn, Mo, Fe
Titanium	334.904	Cr
	337.2	Fe
	334.2	
Vanadium	310.23	Ni
	311.07	
Iron[A]	273.074	
	271.4	
	281.33	
	360.89	

[A] Internal standard.

[4] ASTM Manual Series, ASTM, 6th Edition, 1990.