Designation: E2209 – 13

Standard Test Method for
Analysis of High Manganese Steel by Spark Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation E2209; 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 provides for the analysis of high manganese steel by spark atomic emission spectrometry for the following elements in the ranges shown:

Elements	Conposition Range, %
Aluminum (Al)	0.02 to 0.15
Carbon (C)	0.3 to 1.4
Chromium (Cr)	0.25 to 2.00
Manganese (Mn)	8.0 to 16.2
Molybdenum (Mo)	0.03 to 2.0
Nickel (Ni)	0.05 to 4.0
Phosphorus (P)	0.025 to 0.06
Silicon (Si)	0.25 to 1.5

NOTE 1—The ranges represent the actual levels at which this method was tested.[2] These composition ranges can be extended to higher compositions by the use of suitable reference materials. Sulfur is not included because differences in results between laboratories exceeded acceptable limits at all analyte levels.

1.2 *This test method may involve hazardous materials, operations, and equipment. 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]

A128/A128M Specification for Steel Castings, Austenitic Manganese
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves
E353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys
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
E1059 Practice for Designating Shapes and Sizes of Nongraphite Counter Electrodes (Withdrawn 2013)[4]
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
E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

2.2 *Other Document:*

ASTM MNL 7 ASTM Manual on Presentation of Data and Control Chart Analysis, 8th Edition, 2010.

3. Terminology

3.1 For definition of terms used in this method, refer to Terminology E135.

4. Summary of Test Method

4.1 A controlled discharge is produced between the flat surface of the specimen and the counter electrode. The radiant energies of selected analytical lines are converted into electrical energies by photomultiplier tubes and stored on capacitors. This discharge is terminated after a fixed integration time. At the end of the exposure period, the charge on each capacitor is measured and converted to mass fraction percent.

5. Significance and Use

5.1 The chemical composition of high manganese steel alloys must be determined accurately to ensure the desired metallurgical properties. This procedure is suitable for manufacturing control and inspection testing.

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[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] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1035.

[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.

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 may 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 that is employed. Alternatively, mathematical corrections may be used to solve for interelement effects. Various mathematical correction procedures are commonly utilized. Any of these are acceptable that will achieve analytical accuracy equivalent to that provided by this method.

7. Apparatus

7.1 *Sample Preparation Equipment:*

7.1.1 *Sample Mold,* to produce chilled cast samples approximately 38 mm (1½ in.) in diameter that are homogeneous, free of voids or porosity in the region to be excited, and representative of the material to be analyzed. Refer to Practice E1806 for steel sampling procedures.

7.1.2 *Immersion Sampler,* to take a sample from the bath or from the metal stream when pouring can be used. The sample should produce a sample of the same dimensions as listed in 7.1.1.

7.1.3 *Surface Grinder or Sander With Abrasive Belts or Disk,* capable of providing a flat uniform surface on the reference materials and specimens. The following table shows the various methods of sample preparation used in the Inter-Laboratory Study (ILS):

Type of Grinding Preparation	Belt and/or Disk
Grinding Medium	Aluminum Oxide, Zirconium Oxide
Grit of Grinding Medium	36 to 180

NOTE 2—Silicon carbide grinding medium may be used but it was not utilized by the laboratories in the Inter-Laboratory Study (ILS).

7.2 *Excitation Source,* capable of providing a triggered capacitor discharge having the source parameters meeting the requirements of 11.1.

7.3 *Excitation Stand,* suitable for mounting in optical emission alignment, a flat surface for the specimen in opposition to a counter electrode. This stand shall provide an atmosphere of argon. The electrode and argon are described in 8.1 and 8.2.

7.4 *Spectrometer,* having sufficient resolving power and linear dispersion to separate clearly the analytical lines from other lines in the spectrum of a specimen in the spectral region 170.0 nm to 450 nm. The spectrometer shall have a dispersion of at least 2 nm/mm and a focal length of at least 0.5 m. Gas purged spectrometers are an alternative to vacuum systems.

7.5 *Measuring System,* consisting of photomultiplier tubes having individual voltage adjustment, capacitors on which the output of each photomultiplier tube is stored and an electronic system to measure voltages on the capacitors either directly or indirectly, and the necessary switching arrangements to provide the desired sequence of operation.

7.6 *Vacuum Pump,* if required, capable of maintaining a vacuum of approximately 3 Pa. There are some equipment manufactures that will purge the optical portion of the spectrometer with argon or other inert gas rather than pull a vacuum on the optics. Either vacuum optics or purged optics are required to determine carbon and phosphorus in this method.

7.7 *Gas System,* consisting of an argon supply, a pressure regulator, and a gas flow meter. Automatic sequencing shall be provided to actuate the flow of argon at a given flow rate for a given time interval and to start the excitation at the end of the required flush period. The gas system shall be in accordance with Practice E406.

8. Reagents and Materials

8.1 *Argon,* either gaseous or liquid, must be of sufficient purity to permit proper excitation of the analytical lines of interest. Argon of 99.998 % purity has been found satisfactory. Refer to Practice E406.

8.2 *Counter Electrode*—A Tungsten or Thoriated Tungsten rod ground to a 15°, 30°, 45° or 90° angle conical tip, which conforms to Practice E1059, was found satisfactory. Other material may be used provided it can be shown experimentally that equivalent precision and bias are obtained.

9. Reference Materials

9.1 *Certified Reference Materials,* for high manganese steel are commercially available.

9.2 Calibrants shall be certified reference materials from recognized certification agencies. They shall cover the composition ranges of the elements to be determined and shall include

TABLE 1 Wavelengths

Element	Wavelength (nm)	Line Classification	Possible Interferences[A]
Aluminum	394.4	I	V, Mn, Mo
	396.152	I	Mo
Carbon	193.09	I	Al
Chromium	298.92	II	Mn, V, Ni, Nb, Mo
	267.72	II	Mn, Mo, V
	425.435	I	
Iron (Internal Standard)	273.07	I	
	271.44	II	
Manganese	263.81	II	
	290.02	II	
	293.31	II	Cr
Molybdenum	202.03	II	
	263.876	II	
	281.61	II	Al, Mn
	386.41	I	V, Cr
Nickel	231.60	II	Co, Ti
	218.54	II	
	352.45	I	
	341.476	I	
Phosphorus	178.29	I	Mo
Silicon	212.41	I	
	288.16	I	Mo, Cr, W
	251.61	I	Fe, V
Sulfur	180.73	I	Mn

[A] Interferences are dependent upon instrument design, and excitation conditions, and those listed require confirmation based upon specimens designed to demonstrate interferences. This standard method does not purport to address the interferences that these lines may have. Care should be taken to address the interferences when calibrating the instrument.