



Designation: ~~E2209~~—13 E2209 – 21

Standard Test Method for Analysis of High Manganese Steel by Spark Atomic Emission Spectrometry¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This test method ~~provides for~~ covers the analysis of high manganese steel by spark atomic emission spectrometry for the following elements in the ranges shown:

Elements	Composition Range, %
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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. Validation of these extensions may be conducted by following Practice E2587. Sulfur is not included because differences in results between laboratories exceeded acceptable limits at all ~~analyte~~ sulfur 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.~~

1.3 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:³

- ~~A128/A128M~~ E29 Specification for Steel Castings, Austenitic Manganese
- 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

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.

~~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 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 Inert Gas 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 (Withdrawn 2019)⁴

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

E2587 Practice for Use of Control Charts in Statistical Process Control

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 exposureintegration 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.

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.~~ this method (Table 1). 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.~~ utilized. 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~~ cannot be used. The ~~sample~~ sampler 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):

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

TABLE 1 Wavelengths

Element	Wavelength (nm)	Line Classification	Possible Interferences ⁴
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	
Nickel	281.61	II	Al, Mn
	386.41	I	V, Cr
	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

⁴ 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 all the interferences that these lines may have. ~~Care should be taken~~ Take care to address the interferences when calibrating the instrument.

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Type of Grinding Preparation
Type of Grinding Preparation
Grinding Medium
Grit of Grinding Medium

Document Preview
ASTM E2209-21
Belt and/or-Disk
Belt or Disk, or Both
Aluminum Oxide, Zirconium Oxide
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 argon and argon electrode 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 450.0 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.

NOTE 3—Many current spectrometer manufacturers utilize CMOS/CCD array technology and this enables equivalent resolution with a shorter focal length than specified in 7.4.

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

7.5.1 A photomultiplier (PMT) array having individual voltage adjustments, capacitors in which the output of each PMT 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.

7.5.2 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.

7.5.3 A hybrid design using both PMT's and semiconductor arrays.

7.6 *Vacuum Pump*, if required, capable of maintaining a vacuum of approximately 3 Pa. There are some equipment ~~manufacturers~~manufacturers 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~~Argon (supplied from gas cylinders or liquid tanks), ~~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 tungsten~~ rod ground to a 15°, 30°, 45° or 90° angle conical tip, which conforms to Practice **E1059**, was found satisfactory. The instrument manufacturer will define the material and geometry. 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~~Calibration Reference Materials (RMs) shall be certified reference materials from recognized certification agencies. They shall cover the composition ranges of the elements to be determined and shall include all of the specific types of alloys being analyzed. ~~The calibrants~~They shall be homogeneous and free of voids and porosity. ~~The~~Their metallurgical history of the ~~calibrants~~ should be similar to that of the specimens being analyzed. ~~Refer to Test Methods E353 and E1019 for chemical analysis of high manganese steel alloys.~~

9.2.1 ~~In~~When selecting ~~calibrants~~calibration RMs, use caution with compositions that are unusual. One element may influence the radiant energy of another element. Tests should be made to determine if interrelations exist between elements in the ~~calibrants~~calibration RMs.

10. Preparation of ~~Calibrants~~Calibration RMs and Specimens

10.1 Rough grind, either wet or dry, with a coarse grinding belt or disk. ~~The final grind of the specimen must be the same grit as the calibrants.~~ Dry the specimens, if wet, for proper excitation in the argon atmosphere. Ensure that the specimens are homogeneous and free from voids and pits in the region to be excited. ~~Cast specimens from~~Refer to 7.1.1 molten metal into a suitable mold and 7.1.2 cool. ~~Immersion and stream samplers are also suitable for use.~~ Prepare the surface of the specimens and reference materials in a similar manner.

11. Excitation and Instrument Parameters

11.1 *Electrical Parameters*—Electrical parameters within the following ranges were found acceptable. Burn parameters are normally established by the spectrometer manufacturer. The following ranges are historical guidelines and newer instruments may vary from these.

Triggered Capacitor Discharge

Capacitance, --F	2.5 to 15
Capacitance, μF	2.5 to 15
Inductance, --H	50 to 70
Inductance, μH	50 to 70
Resistance, Σ	residual to 5
Resistance, Ω	residual to 5
Potential, V	940 to 1000
Peak Current, A	100 to 275
Current pulse duration, --s	130 to 250
Current pulse duration, μs	130 to 250
Number of discharges/s	60 to 120

11.2 Spectrometer Configurations:
Spectrometer Parameters

Focal Length	0.5 m to 1.2 m
Dispersion	0.5 nm/mm to 2.16 nm/mm
Vacuum	1 Pa to 25 Pa

11.3 Excitation Conditions:

Flush Time	2 s to 5 s
Preburn	10 s to 30 s
Exposure	5 s to 20 s
Integration	5 s to 20 s

11.4 *Initiation Circuit*—The initiator circuit parameters shall be adequate to uniformly trigger the capacitor discharge. The values for these parameters will vary with the instrument. ~~Normal values~~ Values found to be adequate are listed as follows:

Capacitance (d-c charged) --F	1.2
Capacitance (d-c charged) μF	1.2
Inductance, --H	residual
Inductance, μH	residual
Resistance, Σ	residual
Resistance, Ω	residual
Potential, V	425

<https://standards.iteh.ai/catalog/standards/sist/cf8914ee-37c3-4bcb-adca-09825efa3a80/astm-e2209-21>

11.4.1 *Other Electrical Parameters*—Excitation units, on which the precise parameters given in 11.1 and 11.4 are not available, may be used, provided that it can be shown experimentally that equivalent precision and accuracy are obtained.

11.5 *Electrode System*—Insert the counter electrode in the lower electrode position. Adjust the analytical gap to 3 mm, 4 mm, 5 mm, or 7 mm depending on the manufacturer's recommendations for that particular instrument.

11.6 *Discharge Source*—Most capacitor discharge sources in today's current spectrometers are of either the two types: directional self-initiating capacitor discharge type discharge, or a triggered capacitor discharge type discharge.

12. Preparation of Instrumentation

12.1 Prepare the spectrometer in accordance with as directed in the manufacturer's instructions.

NOTE 4—It is not within the scope of this method to prescribe all details of equipment to be used. Equipment varies between laboratories.

13. Calibration, Standardization, Drift Correction (Standardization), and Verification

13.1 *Calibration*—Using the conditions given in Section 11.3.11, excite the ~~calibrants~~ calibration RMs and potential drift correction samples in a random sequence, bracketing these burns with excitations of any materials intended for use as verifiers. (A verifier may be used as a ~~calibrant~~ calibration RM even though it is burned only as a verifier.) There should be at least five ~~calibrants~~ calibration RMs for each element, spanning the required composition range. Make replicate excitations in accordance with as directed in 14.2. Using the averages of the data for each point, determine analytical curves as described in Practice E305 or the instrument manufacturer's ~~instructions~~ instructions.