



Standard Test Method for X-Ray Emission Spectrometric Analysis of Low-Alloy Steels and Cast Irons¹

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^{ε1} NOTE—Editorial changes were made in October 2000.

1. Scope

1.1 This test method covers the X-ray emission spectrometric analysis of low-alloy steels and cast irons for the following elements in the ranges indicated:

Elements	Concentration Range, %
Manganese	0.20 to 1.50
Nickel	0.10 to 1.00
Chromium	0.10 to 1.00
Molybdenum	0.04 to 0.40
Copper	0.05 to 0.30
Vanadium	0.03 to 0.25

NOTE 1—These concentration ranges can be extended by the use of suitable standards. The detection limit for the elements is lower than the listed value. The ranges represent the actual levels at which this test method was tested.

1.2 *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:

- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²
- E 350 Test Methods for Chemical Analysis of Carbon Steel, Low Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron²
- E 351 Test Methods for Chemical Analysis of Cast Iron—All Types²
- E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory³

3. Terminology

3.1 All terms used in this test method are in conformance with Terminology E 135.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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 Annual Book of ASTM Standards, Vol 03.05.

³ Annual Book of ASTM Standards, Vol 03.06.

4. Summary of Test Method

4.1 The sample is finished to a clean, uniform surface, and then irradiated by an X-ray beam of high energy. The secondary X rays produced are dispersed by means of a crystal and the intensities are measured by a detector at selected wavelengths. The results are obtained by relating measured intensities to an appropriate calibration curve.

5. Significance and Use

5.1 This test method is a comparative method intended for use as a routine method to test materials for compliance with compositional specifications. It is assumed that all who use this test method will be trained analysts capable of performing skillfully and safely. It is expected that work will be performed in a properly equipped laboratory under appropriate quality control practices, such as those described in Guide E 882.

6. Apparatus

6.1 Specimen Preparation Equipment:

6.1.1 *Disk or Belt Sander*, capable of providing test specimens with a uniform, clean surface finish, or other equivalent finishing device.

6.2 Excitation Source:

6.2.1 *X-ray Generator*, with a full-wave rectified power supply, or constant potential power supply.

6.2.2 *X-ray Tube*, with a high-purity tungsten target.

6.3 Spectrometer:

6.3.1 *Analyzing Crystal*, lithium fluoride, flat or curved having a 2d spacing of 0.40276 nm.

6.3.2 *Collimation*—Provide appropriate collimation to achieve the required resolution. For curved optics, no collimation is required.

6.3.3 *Goniometer*—A goniometer that provides an angular scan from 10 to 148°.

6.3.4 *Detectors*, proportional or scintillation counter.

6.4 *Measuring Unit*—An electronic circuit panel capable of amplifying, counting, or integrating pulses received from the detector tube. In addition, a pulse height analyzer should be available for pulse energy discrimination if needed. Good precision and accuracy have been obtained without the use of a pulse height analyzer.

7. Reference Materials

7.1 *Certified Reference Materials*— Low-alloy steel and cast iron certified reference materials are commercially available from a number of sources.

7.2 *Low-Alloy Steel Reference Materials*—Low-alloy steel and cast iron reference materials can be used. They should be analyzed and thoroughly evaluated in accordance with Test Methods E 350 and E 351.

8. Safety Precautions

8.1 Normal safety precautions for X-ray spectrographic installations are to be observed. It is recommended that suitable monitoring devices (film badges and dosimeters) be available and worn by all participating personnel. Accurate control and regulation of monitoring devices at all times is recommended.

9. Preparation of Reference Materials and Specimens

9.1 Using the belt sander equipped with aluminum oxide abrasive paper, prepare a smooth, clean surface on the test specimen having a minimum area of 25 mm². Use 240 grit or finer, but after a grit has been selected, the same grit number should always be used. Other equivalent finish-machining methods can be used.

NOTE 2—Any facets or imperfections in the flatness of the finished surface have been found to give marked changes in response.

9.2 Repolish the reference materials as needed to eliminate oxidized surfaces.

10. Preparation of Apparatus

10.1 Prepare the apparatus as follows:

Voltage, kV	50 (Note 3)
Current, mA	40 (Note 3)
Detector	scintillation or proportional

NOTE 3—Lower values for voltage and current have been found satisfactory.

11. Excitation and Radiation Measurement

11.1 *Excitation*—Place the specimen into the instrument, taking care not to contaminate the specimen surface. Generate the secondary fluorescence using the settings listed in Section 9.

11.2 *Radiation Measurements*—Make radiation measurement of the analytical lines listed in Table 1 using the settings listed in Section 9.

NOTE 4—The listed degree 2 θ peaks represent the theoretical values for a lithium fluoride crystal. The actual goniometer setting for these peaks must be determined experimentally within each laboratory. Periodic checks to verify this setting are advisable.

TABLE 1 Analytical Peaks

Element	Shell	Order	° 2 θ (Note 5)	Wave-length, nm	Crystal
Nickel	K	1	48.61	0.1659	LiF
Manganese	K	1	62.91	0.2103	LiF
Chromium	K	1	69.29	0.2291	LiF
Molybdenum	K	1	20.28	0.0710	LiF
Copper	K	1	44.96	0.1542	LiF
Vanadium	K	1	76.84	0.2505	LiF

11.2.1 Obtain the radiation measurement for each element. Collect sufficient counts to produce the required statistical precision. Use the following to determine the minimum number of counts required:

Element	Total Counts
Nickel	64 000
Manganese	64 000
Chromium	64 000
Molybdenum	32 000
Copper	32 000
Vanadium	16 000

NOTE 5—Larger numbers than the above listed total counts have been accumulated within a reasonable counting time by various instruments.

12. Calibration and Standardization

12.1 *Calibration*—Make measurements on a sufficient number of reference materials to establish the calibration curve. Prepare the calibration curves for each element by plotting the intensity versus concentration values.

12.2 *Standardization*—Whenever samples are to be analyzed, run at least a high- and low-concentration material to ascertain that the calibration is current.

13. Calculation

13.1 The concentrations are determined from the prepared calibration curves.

14. Precision and Bias

14.1 *Precision*—Precision data are shown in Table 2.

14.2 *Bias*—Representative bias data are shown in Table 3.

14.2.1 It is unlikely that any results using this test method will deviate more than the maximum deviation shown in Table 3, provided the operating parameters are as specified.

NOTE 6—The bias approaches the precision when the reference materials and the unknown are similar in composition. When reference materials and unknowns are not alike, one must verify the absence of interferences such as zirconium on molybdenum K_α, the absence of inter-element effects such as molybdenum and nickel on chromium, and the absence of differences due to metallurgical history or condition of the sample.

15. Keywords

15.1 cast iron; low-alloy steel; spectrometric analysis; X-ray emission

TABLE 2 Precision Data^A

Element	Concentration Range, %	Relative Standard Deviation, RSD % ^B
Manganese	0.52 to 1.41	0.60
Nickel	0.23 to 0.79	0.83
Chromium	0.21 to 0.86	0.88
Molybdenum	0.05 to 0.35	1.02
Copper	0.06 to 0.27	1.04
Vanadium	0.03 to 0.22	1.16

^A The values were obtained by pooling data from five laboratories.

^B Relative standard deviation, RSD, in this method, is calculated as follows:

$$RSD = (100/\bar{X}) \sqrt{\sum d^2 / (n - 1)}$$

where:

\bar{X} = average concentration, %,

d = difference of the determination from the mean, and

n = number of determinations.

TABLE 3 Bias Data

Element	Established Value, ^A %	Deviation of X-ray Value ^B from Established Value, relative %	Maximum Observed Deviation from Established Value, %
Manganese	0.640	1.14	0.03
	0.910	0.55	0.02
	1.120	0.96	0.025
Nickel	0.325	1.42	0.01
	0.520	0.23	0.005
	0.695	0.07	0.007
Chromium	0.350	0.49	0.01
	0.515	2.35	0.015
	0.700	0.74	0.03
Molybdenum	0.100	1.90	0.005
	0.195	3.33	0.008
	0.295	0.58	0.005
Copper	0.060	3.83	0.005
	0.080	0.63	0.004
	0.175	0.97	0.007
Vanadium	0.045	12.44	0.01
	0.145	0.68	0.008

^A The samples used for this program are secondary standards whose values are averages from chemical analyses performed by eight laboratories.

^B These values were calculated from the total results reported from three separate days of study, and five participating laboratories.

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