



Standard Test Method for Optical Emission Vacuum Spectrometric Analysis of Blast Furnace Iron by the Point-to-Plane Technique¹

This standard is issued under the fixed designation E485; 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 describes the spectrochemical procedure for the analysis of blast furnace iron (hot metal) containing 4.2 to 5.0 % carbon for the following elements in the indicated ranges:

Elements	Concentration Range, %
Silicon	0.50 to 2.00
Manganese	0.20 to 1.50
Phosphorus	0.020 to 0.15
Titanium	0.02 to 0.10
Sulfur	0.010 to 0.050

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:²

E30 Test Methods for Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron³

E130 Practice for Designation of Shapes and Sizes of Graphite Electrodes

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 Analysis³

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 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³ Withdrawn.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.

E172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis^{3 3}

E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves³

E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data³

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 sample is excited in an inert gas atmosphere by a controlled triggered capacitor discharge using the point-to-plane technique. Using a vacuum spectrometer, the radiant energies of selected analytical lines and an internal standard line are measured by photomultipliers. The output current of each photomultiplier is accumulated and stored during the exposure period as a charge on an associated capacitor, where it appears as a measurable voltage. At the end of the exposure period the voltages corresponding to the analytical lines relative to the voltage for the internal standard line are measured. The measuring system may be calibrated in terms of percent concentration.

5. Apparatus

5.1 Sample Preparation Equipment:

5.1.1 *Sample Mold*, to produce gray 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 cast disk approximately 28 mm (1½ in.) in diameter and 12 to 25 mm (½ to 1 in.) thick is satisfactory.

5.1.2 *Grinder*, to prepare a flat clean surface of the sample for analysis.

5.2 *Supporting Electrode*, to hold the sample in optical alignment and provide good electrical contact.

5.3 *Excitation Source*, providing a controlled triggered capacitor discharge with parameters described in 9.2. (See Practice **E172**.)

5.4 *Spectrometer*, having the following representative characteristics:

	Vacuum	Air
Focal length, m	1	1½
Concave grating, grooves/mm	2160	960
Reciprocal linear dispersion, Å/mm	4.6	6.9
Primary slit width, µm	25 to 75	25 to 75
Secondary slit width, µm	see Table 1	see Table 1
Wavelength coverage, nm	175.0 to 215.0	190.0 to 811.5
Pressure, torr	30 × 10 ⁻³ or less	

5.5 *Measuring System*, consisting of photomultipliers having individual voltage adjustment, capacitors on which the output of each photomultiplier is stored, an amplifier, a recorder system to register the voltages on the capacitors either directly or indirectly (as a ratio), and the necessary switching arrangements to provide the desired sequence of operation.

6. Materials

6.1 *Counter Electrode*—A rod of carbon, 6.15 mm (0.242 in.) in diameter. The shape of the rod tip shall be conical with the tip, 120 deg included angle, (ASTM Type C-2 described in Practice E130) or flat (ASTM Type C-3 also described in E130). The analytical gap shall be 3 or 4 mm.

6.2 *Argon*, high-purity, 99.998 % pure.

7. Reference Materials

7.1 Primary calibration reference materials for the analysis of blast furnace iron are available from the National Institute of Standards and Technology. Other reference materials may be used which have been tested for homogeneity and which have been standardized in accordance with Test Methods E30 to establish analytical curves. Other reference materials can be obtained by casting material from production.

7.2 In selecting other reference materials, observe caution with compositions that are unusual. Avoid using as reference materials cast iron that contains residual amounts of alloying elements in concentrations greater than normally found as residuals in blast furnace iron (greater than 0.20 %). One element may adversely influence the radiation of another element. If cast iron reference materials are used, tests shall be made to determine if interrelations exist between elements in the reference materials. To avoid interelemental effects, it is suggested that the reference materials closely match the composition of the blast furnace iron samples.

8. Preparation of Samples

8.1 *Sampling*—Observe the usual precautions when sampling blast furnace iron to assure that the sample represents as

closely as possible the entire lot of material under test. Samples shall be free of pipe, blowholes, seams, occluded slag, flakes of graphite on the surface, and other physical imperfections. The samples shall be adequate to overlap the bore of the spark stand opening to affect an argon seal of sufficient bulk to prevent overheating during excitation. They shall weigh at least 40 g and be at least 12 mm (½ in.) thick. Samples and reference materials shall be similar in metallurgical condition.

8.2 *Surface Preparation*—Prepare samples and reference materials by grinding or machining to provide uniform flat surface areas. For speed and convenience, it is recommended that a belt or surface grinder with aluminum oxide abrasive be employed, and that a coarse grit, such as 40 or 50, be used.

9. Preparation of Apparatus

NOTE 1—The instructions given herein apply to most spectrometers; however, some settings and adjustments may need to be varied and, depending on the particular equipment, additional preparation of the equipment may be required. It is not within the scope of an ASTM method to prescribe the minute details of the apparatus preparation which differ not only for each manufacturer, but also often for different equipment from the same manufacturer. For a description of a spectrometer and further details of its operation, refer to the manufacturer's handbook.

9.1 Table 1 lists the recommended analytical and internal standard lines. Also given are the recommended exit slit widths. Align or test the position of the exit slits (Note 2) to ensure that peak radiation passes through each slit and is upon the photomultipliers. This shall be done initially, and as often as necessary thereafter to maintain proper optical alignment.

NOTE 2—The manner and frequency of positioning or checking the alignment of the exit slits will depend on factors such as: type of spectrometer, manufacturer, the variety of analytical problems encountered, and frequency of use. For details of setting and operating a particular spectrometer, refer to the manufacturer's handbook. Each laboratory should establish a suitable check procedure.

9.2 Electrical Parameters (Note 3):

Voltage, kV	1
Capacitance, µF	2
Inductance, µH	50
Damping resistance, Ω	0
Discharges/s	60
Current, r-f A	0.2
Type of discharge	triggered capacitor discharge

NOTE 3—The power supply voltage should be controlled within ±2 %.

9.3 Exposure Conditions:

Flush period, s	5 to 20
Preburn period, s	10 to 50 (Note 4)
Exposure period, s	20 to 25 (Note 4)
Argon flow,	
flush period, litres/s	0.20 to 0.40
preburn period, litres/s	0.16 to 0.40
exposure period, litres/s	0.16 to 0.40

NOTE 4—The exact preburn and exposure times required for the intensity ratios to reach reproducible values shall be established in each laboratory.

9.4 *Electrode System*—Insert the carbon counter electrode in the lower electrode position. Insert a calibrant sample in the upper electrode position and condition the counter electrode by one or two burns. Adjust the analytical gap to between 3 and 4 mm.

TABLE 1 Internal Standards and Analytical Lines

Element	Wavelength, nm	Exit slit Width, ^A µm
Iron ^B	271.441 (or 195.16)	75 to 300
Manganese	293.306	25 to 75
Phosphorus	178.287	25 to 75
Sulfur	180.731	25 to 75
Silicon	288.158 (or 251.61)	25 to 75
Titanium	324.20	150

^A Exit slit width is dependent on the optical characteristics of the spectrometer, and thus may be different from one instrument to another.

^B Internal standard.