



Designation: E 327 – 94

Standard Test Method for Optical Emission Spectrometric Analysis of Stainless Type 18-8 Steels by the Point-To-Plane Technique¹

This standard is issued under the fixed designation E 327; 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 stainless Type 18-8 steels for the following elements:

Element	Concentration Range, %
Chromium	17.0 to 20.0
Nickel	8.0 to 15.0
Manganese	0.8 to 2.0
Silicon	0.4 to 0.8
Copper	0.1 to 0.2

1.2 This test method is designed for routine analysis of chill-cast disks or inspection testing of stainless Type 18-8 steel flats upon which a surface of at least 13-mm ($\frac{1}{2}$ -in.) diameter may be prepared. The samples must be sufficiently massive to prevent overheating during the discharge and of similar metallurgical condition and composition as the standards used.

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:

E 130 Practice for Designation of Shapes and Sizes of Graphite Electrodes²

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials²

E 158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis²

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis²

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.

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves²

E 876 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 E 135 **E 135**.

4. Summary of Test Method

4.1 A controlled discharge is produced between the flat ground surface of the sample and a specially shaped graphite electrode. The radiant energy of selected analytical lines is converted into electrical energy by multiplier photo tubes and is stored on capacitors. The discharge is terminated at a predetermined level of accumulated radiant energy from the internal standard iron line or after a fixed exposure time. At the end of the exposure period, the charge on each condenser is evaluated electronically and displayed or recorded as relative energies or concentrations.

5. Apparatus

5.1 Sample Preparation Equipment:

5.1.1 *Sample Mold*, capable of producing castings that are homogeneous and free from voids and porosity. The following mold types have been found to produce acceptable samples:

5.1.1.1 *Cast Iron Mold*—A mold 70 mm ($2\frac{3}{4}$ in.) deep, 64 mm ($2\frac{1}{2}$ in.) in diameter at the top of the mold, and 57 mm ($2\frac{1}{2}$ in.) in diameter at the bottom of the mold. The wall thickness of the mold is approximately 32 mm ($1\frac{1}{4}$ in.).

5.1.1.2 *Refractory Mold Ring*—A mold that has a minimum inside diameter of 32 mm ($1\frac{1}{2}$ in.) and a minimum of 25 mm (1 in.) in height. The ring is placed on a flat surface of a copper plate approximately 50 mm (2 in.) thick.

5.1.1.3 *Book-Type Steel Mold*, to produce a chill-cast disk 64 mm ($2\frac{1}{2}$ in.) in diameter and 13 mm ($\frac{1}{2}$ in.) thick.

5.1.2 *Abrasive Grinder*—A suitable belt grinder, horizontal disk grinder, or similar grinding apparatus may be used. The

³ *Annual Book of ASTM Standards*, Vol 03.06.



resulting surface should be perfectly plane and free of defects. These may be either wet or dry grinding devices. Grit size of grinding material in the range from 60 to 180 has been found satisfactory.

5.2 *Electrode Cutter*, for shaping electrodes to the configuration selected in 6.1.

5.3 *Excitation Source*, with parameters capable of producing a usable spectrum as described in 9.2. (See Practice E 172E 172.)

5.4 *Excitation Stand*, suitable for mounting in optical alignment a flat disk sample in opposition to a graphite counter electrode.

5.5 *Spectrometer*, having sufficient resolving power and linear dispersion to separate clearly the analytical lines from other lines in the spectrum of the sample in the spectral region 2200 to 4600 Å. Suitable instruments can be purchased commercially. Spectrometer characteristics for two of the instruments used in the evaluation of this test method are given in Table 1.

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

6. Reagents and Materials

6.1 *Electrodes*—The counter electrode shall be Type C-2 or C-5 as given in Practice E 130E 130.

7. Standards

7.1 Primary standards are available from the National Institute of Standards and Technology.⁴ These cover all or part of the concentration ranges listed in 1.1. They are valuable in establishing preliminary working curves and determining the precision of the instrument. However, due to metallurgical differences between these standards and the production samples prepared by the sampling procedures recommended for this test method, curves based on primary standards must be corrected with values from secondary standards.

7.2 *Secondary Standards*—These shall be chemically analyzed test samples taken from production heats. They shall cover the concentration ranges of the elements determined and shall include all of the specific types of alloys being analyzed. These standards shall be homogeneous and free of voids or porosity. They shall be prepared in the manner described in 8.1.

⁴ National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD 20899.

TABLE 1 Spectrometer Characteristics

	Instrument A	Instrument B
Focal length, m	1.5	3
Concave grating, grooves/mm	1200	1200
Reciprocal linear dispersion, Å/mm (first order)	7	2.75
Primary slit width, µm	20	75
Secondary slit width, µm	20 to 100	50 to 200
Wavelength coverage, Å	2200 to 4600	2200 to 4600

The metallurgical history of secondary standards should be compatible with that of the samples being analyzed.

8. Preparation of Samples and Secondary Standards

8.1 *Cast Samples*—Cast the sample from molten metal in the sample mold, remove from the mold, and let air cool. Obtain a smooth flat surface by grinding. Make certain that the cast samples are homogeneous and free of voids or pits in the region to be sparked. To speed up the surface preparation, preliminary surfacing with a relatively coarse grinder (30 to 50 grit) will be found helpful. For the final polishing, a grit between 60 and 180 shall be selected and used for all samples. Standards and test pieces must have the same surface treatment. Standards and samples shall be refinished before being resparked on the same area. The grinding material or finish, or both, must be such that a minimum of contamination will occur from this source.

8.2 *Rolled or Forged Samples*—If necessary, saw or machine to the appropriate size and prepare the surface as described in 7.1.

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 test method to prescribe 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 and further details of operation of a particular spectrometer, refer to the manufacturer's handbook.

9.1 Program the spectrometer to accommodate the internal standard lines and analytical lines listed in Table 2 (Note 2). Connect the photomultipliers, capacitors, and related measuring system. Position or test the position of the spectrometer exit slits to ensure that peak radiation passes through each slit and is incident upon the photomultiplier. This shall be done initially and as often as necessary thereafter to maintain proper alignment.

TABLE 2 Analytical Lines

Analytical Line, Å	Internal Standard Line, Å	Concentration Range, %
Cr 2989.19	Fe 2714.41	5.0 to 20.0
Cr 2860.93	Fe 2714.41	
Cr 2862.57	Fe 2714.41	
Mn 2593.73	Fe 2714.41	0.1 to 5.0
Mn 2949.21	Fe 2714.41	
Mn 4034.49	Fe 3719.94	
Mn 2933.06	Fe 2714.41	
Ni 2316.04	Fe 2714.41	2.0 to 15.0
Ni 3414.76	Fe 3719.94	
Ni 3012.00	Fe 3719.94	
Si 2516.12	Fe 2714.41	0.1 to 2.0
Si 2516.12	Fe 3719.94	
Si 2881.58	Fe 2714.41	
Si 2881.58	Fe 3719.94	
Cu 3273.96	Fe 2714.41	0.05 to 0.50
Cu 3273.96	Fe 4404.75	
Cu 3273.96	Fe 3719.94	