Designation: E 1086 – 94 (Reapproved 2000)

Standard Test Method for Optical Emission Vacuum Spectrometric Analysis of Stainless Steel by the Point-to-Plane Excitation Technique[1]

This standard is issued under the fixed designation E 1086; 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[2] provides for the optical emission vacuum spectrometric analysis of stainless steel in solid form by the point-to-plane excitation technique for the following elements in the concentration ranges shown:

Element	Concentration Range, %
Chromium	17.0 to 23.0
Nickel	7.5 to 13.0
Molybdenum	0.01 to 3.0
Manganese	0.01 to 2.0
Silicon	0.01 to 0.90
Copper	0.01 to 0.30
Carbon	0.005 to 0.25
Phosphorus	0.003 to 0.15
Sulfur	0.003 to 0.065

1.2 This test method is designed for the routine analysis of chill-cast disks or inspection testing of stainless steel samples that have a flat surface of at least 13 mm (0.5 in.) in diameter. The samples must be sufficiently massive to prevent overheating during the discharge and of a similar metallurgical condition and composition as the reference materials.

1.3 Analytical curves are plotted using the concentration ratio method as shown in Practice E 158. One or more of the reference materials must closely approximate the composition of the specimen. The technique of analyzing reference materials along with unknowns and performing the indicated mathematical corrections may also be used to correct for interference effects and to compensate for errors resulting from instrument drift. A variety of such systems are commonly used. Any of these that will achieve analytical accuracy equivalent to that reported for this test method are acceptable.

1.4 *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 Materials[3]

E 158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis[3]

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis[3]

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves[3]

E 353 Test Methods for Chemical Analysis of Stainless, Heat-Resisting, Maraging, and Other Similar Chromium-Nickel-Iron Alloys[3]

E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis[4]

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data[4]

E 1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis[4]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E 135.

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 energy of selected analytical lines are converted into electrical energies by photomultiplier tubes and 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

[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.

Current edition approved Jan. 15, 1994. Published March 1994. Originally published as E 1086 – 85. Last previous edition E 1086 – 85 (1989).

[2] Supporting data for this test method are on file at ASTM Headquarters. Request Research Report RR: E02-1023.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

charge on each capacitor is measured, and displayed or recorded as a relative energy or concentration.

5. Significance and Use

5.1 The chemical composition of stainless steels must be determined accurately in order to ensure the desired metallurgical properties. This procedure is suitable for manufacturing control and inspection testing.

6. Apparatus

6.1 *Sample Preparation Equipment*:

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

6.1.1.1 *Refractory Mold Ring*, having a minimum inside diameter of 32 mm (1.25 in.) and a minimum height of 25 mm (1.0 in.). The ring is placed on a flat surface of a copper plate approximately 50 mm (2.0 in.) thick.

6.1.1.2 *Book-Type Steel Mold*, to produce a chill-cast disk 64 mm (2.5 in.) in diameter and 13 mm (0.5 in.) thick. The wall thickness of the mold should be 32 mm (1.25 in.) to aid chill casting.

6.1.2 *Abrasive Grinder*, a suitable belt grinder, horizontal disk grinder, or similar grinding apparatus. The resulting surface should be uniformly plane and free of defects. These may be either wet or dry grinding devices. Grinding materials with grit sizes ranging from 60 to 180 have been found satisfactory.

6.2 *Excitation Source*, with parameters capable of producing a usable spectrum as described in 10.2 and conforming to Practice E 172.

6.3 *Excitation Stand*, suitable for mounting in optical alignment, a flat surface of the specimen in opposition to a counter electrode. The stand shall provide an atmosphere of argon and may be water cooled. Electrode and argon are described in 7.1 and 7.2.

6.4 *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 to 500.0 nm. Spectrometer characteristics for two of the instruments used in this test method are described as having dispersion of 0.697 nm/mm (first order), the focal length of 1 m. Spectral lines are listed in Table 1.

6.5 *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.

6.6 *Readout Console*, capable of indicating the ratio of the analytical lines to the internal standard with sufficient precision to produce the accuracy of analysis desired.

6.7 *Vacuum Pump*, capable of maintaining a vacuum of 25 µm Hg.

6.8 *Flushing System*, consisting of argon tanks, a pressure regulator, and a gas flowmeter. 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 flushing system shall be in accordance with Practice E 406.

TABLE 1 Analytical and Internal Standard Lines

Element	Wavelength, nm	Concentration Switch Over Points
Chromium	298.919	
Nickel	243.789	
	227.021	
	218.549	
	216.910	
Molybdenum	202.030	<1 %
	281.615	
	308.561	>1 %
	369.265	
Manganese	293.306	
Silicon	251.612[A]	
	288.158	
Copper	327.396	<0.10 %
	224.699	>0.10 %
Carbon	193.092	
Phosphorus	178.287[A]	
Sulfur	180.731	
Iron[B]	271.441	
	322.706	

[A] Silicon 251.612 can have a small but significant interference from molybdenum 251.611. Phosphorus 178.287 may show small but significant interferences from unlisted lines or background due to molybdenum, chromium, and manganese. Interference corrections will not be necessary if: separate silicon and phosphorus curves are used for 316 and 317 alloys; the manganese content varies only between 0.7 and 1.5 %; and the chromium concentration is held between 17 and 20 %.

[B] Either iron line 271.441 or 322.775 with narrow entrance and exit slits to avoid interference from manganese 322.809 can be used as internal standard with any of the listed analytical lines. Iron 271.441 is not appropriate for tungsten tool steels or super alloys with high cobalt because of interference from cobalt 271.442.

NOTE 1—It is not within the scope of this test method to prescribe all details of equipment to be used. Equipment varies among laboratories.

7. Reagents and Materials

7.1 *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 E 406.

7.2 *Electrodes*, may be 3 to 6 mm (0.125 to 0.25 in.) in diameter ground to a 90° tip or whatever the instrument manufacturer recommends for the particular source. Hard-drawn, fine, silver rods, thoriated-tungsten rods, or other material may be used provided it can be shown experimentally that equivalent precision and accuracy are obtained.

8. Reference Materials

8.1 *Certified Reference Materials* are available from the National Institute of Standards and Technology[5] and other international certification agencies.

8.2 *Reference Materials* with matrices similar to that of the test specimen and containing varying amounts of the elements to be determined may be used provided they have been chemically analyzed in accordance with ASTM standard test methods. These reference materials shall be homogeneous, and free of voids or porosity.

[5] National Institute of Standards and Technology, U.S. Department of Commerce, Gaithersburg, MD 20899.