Designation: E1086 − 14

Standard Test Method for
Analysis of Austenitic Stainless Steel by Spark Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation E1086; 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] covers the analysis of austenitic stainless steel by spark atomic emission vacuum spectrometry for the following elements in the ranges shown

Element	Composition 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 One or more of the reference materials must closely approximate the composition of the specimen. The technique of analyzing reference materials 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:*[3]

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves

E406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis

E1060 Practice for Interlaboratory Testing of Spectrochemical Methods of Analysis[4]

E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis

E1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition

2.2 *Other ASTM Documents:*

ASTM MNL 7 Manual on Presentation of Data and Control Chart Analysis[5]

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 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 integration time. At the end of the integration period, the charge on each capacitor is measured, and displayed or recorded as a relative energy or mass fraction %.

5. Significance and Use

5.1 The chemical composition of stainless steels must be determined accurately to ensure the desired metallurgical

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[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:E02-1023.

[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.

[4] Withdrawn 1997.

[5] ASTM Manual Series, ASTM International, 8th edition, 2010.

properties. This procedure is suitable for manufacturing control and inspection testing.

6. Apparatus

6.1 *Sampling and Sample Preparation Equipment:*

6.1.1 Refer to Practice E1806 for devices and practices to sample liquid and solid steel.

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 in accordance with 11.1.

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. Counter electrodes 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 nm 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), and a 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.

TABLE 1 Analytical and Internal Standard Lines

Element	Wavelength, nm	Mass Fraction % 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	
	322.775	

[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.

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 or less.

6.8 *Gas System,* consisting of an argon supply with pressure and flow regulation. Automatic sequencing shall be provided to actuate the flow at a given rate for a specific time interval. The flow rate may be manually or automatically controlled. The argon system shall be in accordance with Practice E406.

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 E406.

7.2 *Counter Electrodes,* can vary in diameter from 1.5 mm to 6.5 mm (depending on the instrument manufacturer) and typically are machined to a 90° or 120° angled tip. Silver or thoriated tungsten rods are typically used. 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[6] 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.

8.3 The reference materials shall cover the concentration ranges of the elements being sought. A minimum of three reference materials shall be used for each element.

9. Preparation of Samples

9.1 The specimens and reference materials must be prepared in the same manner. A specimen cut from a large sample section must be of sufficient size and thickness for preparation and to properly fit the spectrometer stand.

9.2 Ensure the specimens are homogenous and free from voids and pits in the region to be excited. Grind the surface with an abrasive belt or disc. Refer to 6.1.2. Perform the final grind with a dry abrasive belt or disc.

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[6] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

10. Preparation of Apparatus

10.1 Follow the manufacturer's instructions for verifying the optical alignment of the entrance slit and programming the appropriate wavelengths (Table 1).

11. Excitation and Integration

11.1 *Electrical Parameters*—Two different types of sources were employed in the testing of this test method.

11.1.1 *Directional Self-Initiating Capacitor Discharge Source:*

Capacitance, µF	0.015
Inductance, L_1, µH	310
Inductance, L_2, µH	20
Resistance, Ω	residual
Potential, V	13 500
Peak Current, A	90
First Valley Current, A	60
Current pulse duration, µs	120
Number of discharges/s	240

11.1.1.1 *Excitation Conditions:*

Flush, s	7	Argon Flow 0.42 m^3/h
Preburn, s	20	Argon Flow 0.42 m^3/h
Integration, s	20	Argon Flow 0.42 m^3/h

11.1.2 *Triggered Capacitor Discharge Source:*

	Preburn	Integration
Pulse Output:		
Capacitance, µF (d-c charged)	7.5	2.5
Inductance, µH	50	50
Resistance, Ω	residual	residual
Potential, V	950	950
Peak Current, A	275	100
Current pulse duration, µs	250	130
Number of discharges/s	120	120
Trigger:		
Capacitance (d-c charged), µF	1.2	...
Inductance, µH	residual	...
Resistance, Ω	residual	...
Potential, V	425	...

11.1.2.1 *Excitation Conditions:*

Flush, s	2	Argon Flow 0.56 m^3/h
Preburn, s	10	Argon Flow 0.56 m^3/h
Integration, s	10 or 15	Argon Flow 0.56 m^3/h

12. Calibration, Standardization, and Verification

12.1 *Calibration*—Using the conditions given in 11.1, excite each calibrant and drift correction sample two to four times and bracket these with similar excitations of any verifiers. A verifier may be used as a calibrant even though it is burned only as a verifier. There shall be at least three calibrants for each element, spanning the required concentration range. If the spectrometer system and software permits, perform random excitations of each calibrant and drift correction sample and repeat with different random sequences at least four times. Follow the spectrometer manufacturer's software procedures to convert sample intensities into mass fraction %. Using the averages of the data for each point, determine analytical curves in accordance with Practice E305.

12.2 *Standardization*—Following the manufacturer's recommendations, standardize on an initial setup or anytime that it is known or suspected that readings have shifted. Make the necessary corrections either by adjusting the controls on the readout or by applying arithmetic corrections. Standardization will be done anytime verification indicates that readings have gone out of statistical control.

12.3 *Verification*—Analyze verifiers in replicate to confirm that they read within expected confidence interval, in accordance with 12.4.

12.3.1 Each laboratory should determine the frequency of verification necessary based on statistical analysis. Typically, every 4 h to 8 h is practical and adequate (or if the instrument has been idle for more than 1 hour). If the results are not within the control limits established in 12.4, perform a standardization and then repeat verification. Repeat standardization as necessary so verifications are within control limits or investigate further for instrument problems.

12.4 The confidence interval will be established from observations of the repeatability of the verifiers by utilizing the upper and lower limit of a control chart in accordance with Practice E1329 or ASTM Manual MNL 7.

13. Procedure for Excitation and Radiation Measurement

13.1 Produce and record the spectra using the conditions in 11.1.

13.2 *Replicate Excitation*—Make duplicate excitations of each specimen and report the average. Place the freshly surfaced specimen on the excitation stand in a manner to effect a gas-tight seal and adequate argon flushing. Position the specimen so there will be a uniform pattern of excitations around its face. For example, a disk-shaped specimen should have a ring of excitation marks around its outer edge and approximately 6 mm (0.25 in.) from the edge. Avoid the center of cast specimens because of possible quench cracks and segregation. Make a good electrical ground. If required, cool the specimen after two excitations to prevent overheating. Examine the specimen after each excitation to evaluate the quality of excitation. Cracks, voids, pits, moisture, or inclusions will limit the sampling and the accuracy of a determination. Successive excitations shall be sufficiently separated so that the discharge patterns do not overlap.

14. Calculation of Results

14.1 Average the readings obtained for each specimen. If the readout is not in direct mass fraction % units, use this value to obtain the mass fraction % from the curves, or related scale values and mass fraction % by reference to a table that has been previously prepared.

15. Precision and Bias[2]

15.1 *Precision*—The precision of this test method was determined by submitting three stainless alloy samples to five different laboratories. The interlaboratory testing was conducted in accordance with Practice E1060. Instrument calibrations were performed utilizing (1) reference materials supplied with the unknowns, (2) in-house reference materials, and (3) NIST and British Certified reference materials. The unknowns and in-house reference materials were run on three separate days. The precision data for the three unknowns are shown in Table 2.

15.1.1 *Accuracy*—The three unknowns were analyzed by alternate chemical methods by two laboratories independent of

TABLE 2 Precision Data

Element	Sample[A]	Average Mass Fraction %	Standard Deviation, Single Value		Range of Duplicates, 95 % Confidence	
			Within Laboratory, %	Between Laboratories, %	E1060, (R_1)[B] , %	E1060, (R_2)[C] , %
Carbon	CT-304	0.062	0.0011	0.0033	0.0035	0.0100
	CT-305	0.067	0.0015	0.0052	0.0046	0.0159
	CT-316	0.061	0.0013	0.0015	0.0040	0.0047
Manganese	CT-304	0.765	0.0038	0.0212	0.0120	0.0643
	CT-305	1.84	0.016	0.037	0.049	0.111
	CT-316	1.66	0.012	0.022	0.036	0.067
Phosphorus	CT-304	0.025	0.0004	0.0025	0.0014	0.0077
	CT-305	0.025	0.0004	0.0010	0.0011	0.0029
	CT-316	0.031	0.0004	0.0012	0.0014	0.0036
Sulfur	CT-304	0.022	0.0007	0.0017	0.0021	0.0052
	CT-305	0.022	0.0007	0.0018	0.0023	0.0054
	CT-316	0.024	0.0006	0.0011	0.0018	0.0034
Silicon	CT-304	0.55	0.005	0.006	0.015	0.019
	CT-305	0.56	0.004	0.008	0.013	0.026
	CT-316	0.72	0.008	0.016	0.025	0.047
Copper	CT-304	0.33	0.005	0.022	0.016	0.067
	CT-305	0.29	0.005	0.022	0.016	0.019
	CT-316	0.24	0.003	0.012	0.011	0.038
Nickel	CT-304	9.63	0.055	0.237	0.17	0.72
	CT-305	11.97	0.105	0.191	0.33	0.58
	CT-316	12.54	0.073	0.242	0.23	0.73
Chromium	CT-304	18.65	0.075	0.148	0.24	0.45
	CT-305	18.55	0.107	0.108	0.34	0.33
	CT-316	17.48	0.053	0.150	0.17	0.46
Molybdenum	CT-304	0.316	0.0035	0.0082	0.0109	0.0250
	CT-305	0.470	0.0020	0.0116	0.006	0.0366
	CT-316	2.44	0.016	0.019	0.052	0.057

[A] This is a composite of each of the five one-week daily results. This represents a total of 15 determinations. The results reported by each laboratory are on file at ASTM International Headquarters.

[B] Within-laboratory, repeatability, degrees of freedom = 10.

[C] Between-laboratories, reproducibility, degrees of freedom = 14.

the testing of this test method. The agreement between the results obtained by chemical methods and those obtained by this test method is displayed in Table 3. These data were obtained by procedures outlined in Practice E1060.

15.2 *Bias*—There is no known bias in this test method.

16. Keywords

16.1 austenitic stainless steel; spark atomic emission; spectrometric analysis; spectrometry

TABLE 3 Accuracy Data

Element	Sample	Mass Fraction %		Accuracy, % (95 % Confidence)		
		Determined[A]	Assumed True Value	A7, S_a[B]	E1060, $S_{(w)}$[C]	E1060, $S_{(SR)}$[D]
Carbon	CT-304	0.062	0.063	0.0025	0.0011	0.0033
	CT-305	0.067	0.067	0.0052	0.0015	0.0052
	CT-316	0.061	0.061	0.0026	0.0013	0.0015
Manganese	CT-304	0.765	0.78	0.025	0.0038	0.0212
	CT-305	1.84	1.85	0.027	0.016	0.037
	CT-316	1.66	1.67	0.022	0.012	0.022
Phosphorus	CT-304	0.025	0.026	0.0015	0.0004	0.0025
	CT-305	0.025	0.025	0.0009	0.0004	0.0010
	CT-316	0.031	0.029	0.0021	0.0004	0.0012
Sulfur	CT-304	0.022	0.023	0.0021	0.0007	0.0017
	CT-305	0.022	0.022	0.0017	0.0007	0.0018
	CT-316	0.024	0.023	0.0018	0.0006	0.0011
Silicon	CT-304	0.55	0.56	0.013	0.005	0.006
	CT-305	0.56	0.55	0.014	0.004	0.008
	CT-316	0.72	0.69	0.013	0.008	0.016
Copper	CT-304	0.33	0.34	0.023	0.005	0.022
	CT-305	0.29	0.29	0.007	0.005	0.006
	CT-316	0.24	0.25	0.017	0.003	0.012
Nickel	CT-304	9.63	9.60	0.223	0.055	0.237
	CT-305	11.97	11.95	0.181	0.105	0.191
	CT-316	12.54	12.61	0.78	0.073	0.242
Chromium	CT-304	18.65	18.57	0.165	0.075	0.148
	CT-305	18.55	18.58	0.114	0.107	0.108
	CT-316	17.48	17.60	0.184	0.053	0.150
Molybdenum	CT-304	0.316	0.33	0.017	0.0035	0.0082
	CT-305	0.470	0.45	0.019	0.0020	0.0116
	CT-316	2.44	2.45	0.020	0.016	0.019

[A] All mass fractions consist of a 15-determination average, the “apparent” outliers were left in because a statistical evaluation of the data is not a satisfactory reason for eliminating the data values. The results for each individual laboratory are on file at ASTM International Headquarters.

[B] Estimate of the overall accuracy for a single analysis from any laboratory.

[C] Estimate of the standard deviation of any single random analysis within laboratories.

[D] Variable use to calculate R_2 in Table 2 and comprised of the $\sqrt{}$ of the sum of *(1)* estimate of variance between laboratories and *(2)*S_w^2 divided by the number of analyses that are averaged to obtain the reported value.