Designation: E1999 – 11

Standard Test Method for Analysis of Cast Iron by Spark Atomic Emission Spectrometry[1]

This standard is issued under the fixed designation E1999; 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 analysis of cast iron by spark atomic emission spectrometry for the following elements in the concentration ranges shown (Note 1):

Elements	Concentration Ranges, % Applicable Range, %	Quantitative Range, %[A]
Carbon	1.9 to 3.8	1.90 to 3.8
Chromium	0 to 2.0	0.025 to 2.0
Copper	0 to 0.75	0.015 to 0.75
Manganese	0 to 1.8	0.03 to 1.8
Molybdenum	0 to 1.2	0.01 to 1.2
Nickel	0 to 2.0	0.02 to 2.0
Phosphorus	0 to 0.4	0.005 to 0.4
Silicon	0 to 2.5	0.15 to 2.5
Sulfur	0 to 0.08	0.01 to 0.08
Tin	0 to 0.14	0.004 to 0.14
Titanium	0 to 0.12	0.003 to 0.12
Vanadium	0 to 0.22	0.008 to 0.22

[A]Quantitative range in accordance with Practice E1601.

NOTE 1—The concentration ranges of the elements listed have been established through cooperative testing of reference materials. These concentration ranges can be extended by the use of suitable reference materials.

1.2 This test method covers analysis of specimens having a diameter adequate to overlap the bore of the spark stand opening (to effect an argon seal). The specimen thickness should be sufficient to prevent overheating during excitation. A heat sink backing may be used. The maximum thickness is limited only by the height that the stand will permit.

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:*[2]

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for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis (Withdrawn 2004)[3]

E172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis (Withdrawn 2001)[3]

E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves

E351 Test Methods for Chemical Analysis of Cast Iron—All Types

E406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis

E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry

E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques

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

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

E1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods

[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] 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] The last approved version of this historical standard is referenced on www.astm.org.

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

2.2 *Other Documents:*

MNL 7A Manual on Presentation of Data and Control Chart Analysis[4]

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 capacitor discharge is produced between the flat, ground surface of the disk specimen and a conically shaped electrode. The discharge is terminated at a predetermined intensity of a selected iron line, or at a predetermined time, and the relative radiant energies of the analytical lines are recorded and converted to concentration.

4.2 Carbon, phosphorus, sulfur and tin emit in the vacuum ultraviolet region. The absorption of the radiation by air in this region is overcome by flushing the spark chamber with argon or argon-hydrogen gas mixture and either evacuating the spectrometer or filling the spectrometer with an inert gas such as nitrogen or argon. A capacitor discharge is produced between the flat, ground surface of the disk specimen and a conically shaped electrode. The discharge is terminated at a predetermined intensity of a selected iron line, or at a predetermined time, and the relative radiant energies of the analytical lines are recorded and converted to concentration.

NOTE 2—It is not within the scope of this test method to prescribe specific details of every instrument that could be used for the analysis of cast iron by spark atomic emission spectrometry. The parameters listed in this test method represent the parameters of the specific instruments used during the interlaboratory study to produce the precision and bias listed in this test method. Other spark atomic emission spectrometers with different parameters may be used provided that they produce equivalent or better precision and bias data

5. Significance and Use

5.1 The chemical composition of cast iron alloys shall be determined accurately in order to insure the desired metallurgical properties. This procedure is suitable for manufacturing control and inspection testing.

6. Interferences

6.1 Interferences may vary with spectrometer design and excitation characteristics. Direct spectral interferences may be present on one or more of the wavelengths listed in a method. Frequently, these interferences shall be determined and proper corrections made by the use of various reference materials. Refer to Table 1 for possible interferences. The composition of the sample being analyzed should match closely the composition of one or more of the reference materials used to prepare and control the calibration curve. Alternatively, mathematical corrections may be used to solve for interelement effects (refer to Practice E158). Various mathematical correction procedures are commonly utilized. Any of these correction procedures that produce precision and accuracy results equal to or better than the results in the interlaboratory study for this test method are acceptable.

TABLE 1 Analytical and Internal Standard Lines, Possible Interferences

Element	Wavelength, nm	Reported Possible Interfering Elements
Carbon	193.09	Al, Mo, Cu, S
Chromium	267.72	Mo, S, Mn
	265.86	
Copper	211.21	Ni
	221.81	
	327.40	Mo, P
	510.55	V
Manganese	293.31	Cr, Mo, W
Molybdenum	202.03	Ni
	281.61	Mn
Nickel	243.79	Mn
	231.60	Mn
	341.48	
	352.45	Mo
Phosphorus	178.29	Cr, Mn, Mo, Cu
Silicon	212.41	Mo, Cu, Ni
	251.61	
	288.16	Mo, Cr
Sulfur	180.73	Mn, Cu, Cr
Tin	189.99	Mn, Mo, Fe
Titanium	334.90	Cr
	337.28	Fe
	334.19	
Vanadium	310.23	Ni
	311.07	
Iron[A]	273.07	
	271.44	
	281.33	
	360.89	

[A]Internal standard.

7. Apparatus

7.1 When required, use sample preparation equipment as follows:

7.1.1 *Sample Mold,* to produce graphite-free white chilled 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 chill-cast disk approximately 40 mm (1 ½ in.) in diameter and 3-mm to 12-mm (⅛-in. to ½-in.) thick is satisfactory. A sample mold made from copper with a low oxygen content has proven to be optimum for this purpose. Refer to Practice E1806 for iron sampling procedures.

7.1.2 *Surface Grinder or Sander with Abrasive Belts or Disks,* capable of providing a flat, clean, uniform surface on the reference materials and specimens.

7.2 *Excitation Source,* capable of providing sufficient energy to sample the specimen and excite the analytes of interest.

[4] ASTM Manual Series, ASTM, 6th Edition, 1990.

See Practice E172. Any other excitation source whose performance has been proven to be equivalent may be used.

7.3 *Excitation Chamber,* automatically flushed with argon or other inert gas. Clean the excitation chamber when the counter electrode is replaced.

NOTE 3—Clean the lens or protective window as recommended by the instrument manufacturer.

7.4 *Spectrometer,* having sufficient resolving power and linear dispersion to separate clearly the analytical lines from other lines in the spectrum in the spectral region 170.0 nm to 520.0 nm. The spectrometers used to test this method had a dispersion of 0.3 nm/mm to 0.6 nm/mm and a focal length of 0.5 m to 0.75 m. Spectral lines are listed in Table 1. The primary slit width is 15 µm to 50 µm. Secondary slit width is 15 µm to 200 µm. The spectrometer shall be provided with one or more of the following:

7.4.1 An air/gas inlet and a vacuum outlet. The spectrometer shall be operated at a vacuum of 25 µm of mercury or below.

7.4.2 A gas inlet and a gas outlet.

7.4.3 Sealed with nitrogen or other inert gas.

7.5 *Measuring System,* consisting of photomultipliers having individual voltage adjustment, capacitors on which the output of each photomultiplier 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.

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

7.7 *Gas System,* consisting of an argon or argon-hydrogen 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 gas system shall be in accordance with Practice E406.

7.8 *Vacuum Pump,* if required, capable of maintaining a vacuum of 25 µm Hg or less.

NOTE 4—A pump with a displacement of at least 0.23 m^3/min (8 ft^3/min) is usually adequate.

8. Reagents and Materials

8.1 *Inert Gas (Argon, Nitrogen), or Hydrogen,* as required, shall be of sufficient purity to permit proper excitation of the analytical lines of interest in the excitation chamber or light transmittance in the spectrometer chamber. Use in accordance with Practice E406.

8.2 *Counter Electrodes*—A silver or thoriated tungsten rod of 2-mm to 6-mm diameter ground to a 30° to 90° conical tip. Other material may be used provided it can be shown experimentally that equivalent precision and accuracy are obtained.

NOTE 5—A black deposit may build up on the tip of the electrode, thus reducing the overall intensity of the spectral radiation. The number of acceptable excitations on an electrode varies from one instrument to another and should be determined in each laboratory. Cleaning electrodes after each burn significantly reduces this buildup and gives more consistent results.

9. Calibrants

9.1 Calibrants can come in three forms: certified reference materials, reference materials, and analyzed production samples. In selecting calibrants, use caution with compositions that are unusual. One element may adversely influence the radiant energy of another element or its uniformity of distribution within the material. Tests should be made to determine if interrelations exist between elements in the calibrants. To compensate for inter-element effects, it is suggested that the calibrants approximate the composition of the material to be tested. The metallurgical history of the calibrants should be similar to that of the specimens being analyzed in accordance with the recommendations of Practice E305.

9.2 *Certified Reference Materials (CRMs),* used as calibrants for chill-cast iron alloys are available commercially.

9.3 *Reference Materials (RM's),* used as calibrants for chill-cast iron alloys are available commercially.

NOTE 6—The distinction is made between CRMs and production materials because there are commercially available RMs produced by reputable producers that do not claim to be CRMs but in all other respects fit the definition of CRMs.

9.4 *Analyzed Production Samples* shall be chemically analyzed test specimens taken from production heats produced according to Practice E1806. They shall cover the concentration ranges of the elements to be determined and shall include all of the specific types of alloys being analyzed. These calibrants shall be homogeneous and free of voids and porosity. Refer to Test Methods E351 and E1019 or other nationally accepted test methods for chemical analysis of iron base alloys. Refer to Practice E826 for information on homogeneity testing of reference materials.

10. Preparation of Calibrants and Specimens

10.1 *Specimens,* cast graphite-free specimens from molten metal into a suitable mold and cool. Refer to Practice E1806 for information on the preparation of specimens for analysis.

10.2 *Preparation,* prepare the surface to be analyzed on a suitable belt or disk grinder. Prepare the surface of the specimens and calibrants in a similar manner. All specimens shall be free of moisture, oil, and residue for proper excitation.

10.3 Specimen porosity is undesirable because it leads to the "diffuse-type" rather than the desired "concentrated-type" discharge. The specimen surface should be kept clean because the specimen is the electron emitter, and electron emission is inhibited by oily, dirty surfaces.

10.4 Calibrants and specimens shall be refinished dry on a belt or disc sander before being re-excited on the same area.

11. Specimen Excitation Parameters

11.1 Operate the spectrometer according to the manufacturer's instructions.

NOTE 7—When parameters are established, maintain them carefully. The variation of the power supply voltage shall not exceed ±5 % and preferably should be held within ±2 %.

11.1.1 An example of excitation parameters for a high-energy unidirectional spark source is listed below:

	Preburn	Exposure
Capacitance, μF	10	10
Inductance, μH	20	20
Resistance, Ω	0	4.4
Potential, V	550	350
Number of discharges/s	120	60

11.2 *Spark Conditions (Note 8)*—An example of spark parameters is listed below:

Flush period, s	2 to 10	
Preburn period, s	5 to 20	
Exposure period, s	5 to 20	
Gas Flow	ft^3/h	L/min
Flush	5 to 45	2.5 to 25
Preburn	5 to 45	2.5 to 25
Exposure	5 to 30	2.5 to 15

NOTE 8—Select preburn and exposure periods after a study of volatization rates during specimen excitation. Once established, maintain the parameters consistently. The instrument manufacturer can normally provide this information.

11.3 *Electrode System*— For conventional capacitor discharge excitation systems, the specimen, electrically negative, serves as one electrode. The opposite electrode or counter electrode is a thoriated tungsten or silver rod. Use a 3-mm to 6-mm (0.125-in. to 0.25-in.) analytical gap. Once a gap size is selected, maintain it consistently. Condition a fresh counter electrode with 2 excitations to 6 excitations. A high-purity argon atmosphere is required for the analytical gap. Molecular gas impurities, nitrogen, oxygen, hydrocarbons, or water vapor, either in the gas system or from improperly prepared specimens should be minimized.

12. Preparation of Apparatus

12.1 Prepare the spectrometer in accordance with the manufacturer's instructions. Program the spectrometer to accommodate the internal standard lines and one of the analytical lines for each element listed in Table 1.

12.2 Test the positioning of the spectrometer entrance slit to ensure that peak radiation is entering the spectrometer chamber. This shall be done initially and as often as necessary to maintain proper entrance slit alignment. Follow the manufacturer's recommended procedures. The laboratory shall determine the frequency of positioning the alignment based on instrument performance.

12.3 Exit slit positioning and alignment is normally performed by the manufacturer at spectrometer assembly. Under normal circumstances, further exit slit alignment is not necessary (Note 9).

NOTE 9—The manner and frequency of positioning or checking the position of the exit slits will depend on such factors as the type of spectrometer and the frequency of use. Each laboratory should establish a suitable check procedure.

13. Calibration, Standardization, and Verification

13.1 *Calibration*— Using the parameters in Section 11, excite each calibrant and potential standardant two to four times in random sequence, bracketing these with excitations of any materials intended for use as verifiers (a verifier may be used as a calibrant even though it is used principally as a verifier). There should be at least seven calibrants for each element, spanning the required concentration range. Repeat with different random sequences at least two times. Using the average intensity of the data for each point, determine analytical curves as described in Practices E158 and E305. (Note 10)

13.2 *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 shall be done anytime verifications indicate that readings have gone out of statistics control.

13.3 Verification shall be done at least at the beginning of instrument operation. A number of warm-up burns may be necessary. Analyze verifiers with duplicate burns to confirm that the average of the two burns falls within the control limits established in 17.1.

13.3.1 Check the verification after standardizing. Each laboratory should determine the frequency of verification necessary based on statistical analysis. Refer to 17.1. Typically, every 4 or 8 hours is practical and adequate. If results are not within the control limits established in 17.1, perform a standardization and then repeat verification. Repeat standardization as necessary so verifications are within control limits or investigate further for instrument problems.

NOTE 10—Modern instruments are very stable, and the software may not permit more than one set of intensity data to be averaged for the calibration curves since it is unnecessary.

14. Procedure for Excitation and Radiation Measurement

14.1 Check the standardization by verification as listed in 13.3.

14.2 Produce and record the radiation intensities for each element using the conditions given in Section 11.

14.3 *Replicate Excitation*—Make a minimum of two burns on each specimen. Average the replicate readings for each element if their difference does not exceed twice the established standard deviation for the element. If their difference exceeds this value, analyze the specimen two more times and average all four readings. In all cases, discard readings caused by observable defects in the specimen and replace it with another reading. When placing the freshly surfaced specimen on the excitation stand, position it to effect a gas tight seal and adequate gas flushing. Position the specimen so that there will be a uniform pattern of burns around its surface. For example, a disk-shaped specimen should have a ring of burn marks around its outer edge and approximately 6 mm (¼ in.) from the edge. Avoid burning the center of cast specimens where there is more likely to be quench cracks and segregation. Make certain there is a good electrical connection between the specimen and the specimen ground. Cool the specimen after two burns to prevent overheating, if required. Successive burns shall be sufficiently separated so that the burn patterns do not overlap.

14.4 Examine the specimen and instrument measurements after each burn to evaluate the quality of excitation. Cracks, voids, pit, moisture, or inclusions will invalidate the sampling and accuracy of a determination.

15. Calculation of Results

15.1 Average the readings obtained for each element.

16. Precision and Bias

16.1 *Precision:*

16.1.1 Seven laboratories cooperated in performing this test method and obtained the statistical information summarized in Tables 2-4.[5] The interlaboratory data were evaluated in accordance with Practices E1601 and E1763. An approximate value for the expected reproducibility index, R, can be calculated for carbon with the following equation:

$$R = C_c \times 0.059 \quad (1)$$

where: C_c is the expected carbon content in the range 1.9 % to 3.8 %.

TABLE 2 Precision Data

Test Material	Number of Laboratories	Found, %	Minimum SD (S_M, Practice E1601)	Reproducibility SD (S_R, Practice E1601)	Reproducibility Index (R, Practice E1601)	R_{rel} %
			Carbon			
D	7	1.970	0.0125	0.0364	0.1019	5.17
C	7	2.426	0.0204	0.0577	0.1616	6.66
A	7	2.986	0.0151	0.0685	0.1919	6.42
E	7	3.063	0.0192	0.0478	0.1337	4.36
B	7	3.495	0.0221	0.0818	0.2289	6.55
F	7	3.717	0.0208	0.1641	0.4596	12.36
			Chromium			
F	7	0.1044	0.0028	0.0045	0.0126	12.08
A	7	0.3089	0.0019	0.0107	0.0301	9.74
C	7	0.5350	0.0031	0.0182	0.0510	9.52
B	7	0.7153	0.0028	0.0133	0.0373	5.22
E	7	1.091	0.0050	0.0152	0.0425	3.89
D	7	2.048	0.0068	0.0500	0.1401	6.84
			Copper			
F	7	0.0145	0.00082	0.00249	0.00698	47.98
C	7	0.1386	0.00164	0.01001	0.02802	20.22
E	7	0.4935	0.0064	0.0204	0.0570	11.55
B	7	0.5404	0.0045	0.0194	0.0543	10.04
A	7	0.7611	0.0069	0.0131	0.0367	4.82
D[A]	7	0.9820	0.0123	0.0657	0.1841	18.74
			Manganese			
F	7	0.2019	0.0018	0.0061	0.0172	8.50
D	7	0.6932	0.0055	0.0232	0.0650	9.38
A	7	0.8060	0.0039	0.0128	0.0357	4.43
E	7	0.990	0.0048	0.0132	0.0369	3.72
B	7	1.201	0.0092	0.0161	0.0451	3.76
C	7	1.813	0.0094	0.0376	0.1052	5.80
			Molybdenum			
A	7	0.0269	0.00061	0.00172	0.00481	17.87
F	7	0.1031	0.0017	0.0042	0.0118	11.47
E	7	0.3018	0.0027	0.0050	0.0139	4.60
C	7	0.4459	0.0051	0.0146	0.0408	9.14
D	7	0.5015	0.0027	0.0139	0.0389	7.75
B	7	1.151	0.0048	0.0128	0.0358	3.11
			Nickel			
F	7	0.0654	0.00108	0.00299	0.00837	12.79
A	7	0.0876	0.00132	0.00396	0.01108	12.65
B	7	0.5722	0.0042	0.0266	0.0745	13.01
E	7	0.7498	0.0077	0.0303	0.0849	11.33
D	7	1.259	0.0129	0.0543	0.1520	12.08
C	7	1.981	0.0181	0.0759	0.2125	10.73
			Phosphorus			
F	6	0.0037	0.00019	0.00129	0.00360	96.95
A	7	0.0230	0.00038	0.00124	0.00347	15.04
C	7	0.0300	0.00051	0.00166	0.00465	15.50
E	7	0.0502	0.00074	0.00354	0.00990	19.71
D	7	0.0784	0.00148	0.00419	0.01173	14.96
B	6	0.4141	0.00361	0.02530	0.07084	17.11
			Silicon			
F	7	0.5272	0.0051	0.0354	0.0991	18.80
E	7	1.082	0.0082	0.0300	0.0841	7.77
A	7	1.917	0.0120	0.0432	0.1209	6.30
C	7	2.058	0.0226	0.1071	0.2999	14.57
B	7	2.224	0.0123	0.0562	0.1574	7.08
D	7	2.519	0.0131	0.0894	0.2504	9.94
			Sulfur			
F	6	0.0023	0.00028	0.00114	0.00318	141.31
E	7	0.0058	0.00062	0.00166	0.00464	79.93
A	7	0.0464	0.00158	0.00595	0.01667	35.91
C	7	0.0554	0.00418	0.00665	0.01862	33.60
D	7	0.0576	0.00219	0.00501	0.01402	24.35
B	7	0.0776	0.00449	0.01176	0.03294	42.43
			Tin			
A	7	0.0119	0.00025	0.00076	0.00214	17.98
D	7	0.0318	0.00042	0.00196	0.00363	11.42
B	7	0.0541	0.00044	0.00247	0.00692	12.79
C	7	0.0561	0.000537	0.00290	0.00812	14.47
E	7	0.1367	0.00109	0.00335	0.00937	6.85
			Titanium			
A	6	0.0105	0.00028	0.00108	0.00304	28.95
B	6	0.0339	0.00033	0.00138	0.00387	11.42
D	6	0.0805	0.00046	0.00993	0.02779	34.52
C	6	0.0865	0.00133	0.00520	0.01456	16.83
E	6	0.1114	0.00586	0.00720	0.02015	18.09
			Vanadium			
A	7	0.0071	0.00015	0.00150	0.00419	59.01
D	7	0.0501	0.00068	0.00298	0.00836	16.69
E	7	0.0851	0.00053	0.00255	0.00715	8.40
C	7	0.1199	0.00163	0.00483	0.01352	11.28
B	7	0.2166	0.00099	0.00672	0.01883	8.69

[A] Sample D is not included in determining the scope of testing in 1.1.

TABLE 3 Constants for Reproducibility Index Equation

A	K_R	K_{rel}	B
Chromium	0.0126	0.062	2.0
Copper	0.007	0.067	0.75
Manganese	0.016	0.050	1.80
Molybdenum	0.005	0.031	1.20
Nickel	0.0064	0.11	2.0
Phosphorus	0.0024	0.17	0.40
Silicon	0.066	0.075	2.5
Sulfur	0.0032	0.35	0.08
Tin	0.002	0.067	0.14
Titanium	0.00138	0.17	0.12
Vanadium	0.0042	0.087	0.22

16.1.2 An estimate of the reproducibility index for the other analytes, R_A, can be calculated with the following equation:

$$R_A = \sqrt{\left[K_R^2 + \left(C_A \times K_{rel}\right)^2\right]} \text{ over the analyte range of 0 to } B \quad (2)$$

where:

C_A = expected analyte content in %,

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1027.

B = upper limit of the analyte range in % (from Table 3), and

K_R and K_{rel} are constants for each analyte from Table 3.

16.1.3 Laboratories participating in the interlaboratory study used the same set of calibration specimens. Users are warned that comparisons of results between laboratories using different sets of calibration materials may experience greater differences in results than is implied by the calculated values for R from the equation or Table 2.

16.2 *Bias*—The accuracy of this test method at certain concentration levels may be judged by comparing the accepted reference values with the arithmetic average obtained by interlaboratory testing (see Table 4). Users are warned that the accuracy of results from applying the method depend upon the accuracy of the calibration materials used and the care with which the calibration is performed.

TABLE 4 Bias Information

Test Material	Assumed True Value, %	Average Spectrometer Value, %	Difference, %	Material Identification, Uncertainty or (SD)	
		Carbon			
D	1.94	1.970	0.030	BS 2C	0.02
C	2.36	2.426	0.066	BS 1C	0.03
A	2.97	2.986	0.016	BS 290A	0.05
E	3.01	3.063	0.053	BS 3C	0.03
B	3.50	3.495	0.005	CKD U	(0.007)
F	3.82	3.717	–0.103	BS 4C	0.03
		Chromium			
F	0.11	0.1044	–0.0056	BS 4C	0.005
A	0.320	0.3089	–0.0111	BS 290A	0.005
C	0.52	0.5350	0.0150	BS 1C	0.03
B	0.725	0.7153	–0.0096	CKD U	(0.008)
E	1.11	1.091	–0.019	BS 3C	0.02
D	2.03	2.048	0.018	BS 2C	0.02
		Copper			
F	0.014	0.0145	0.0005	BS 4C	0.002
C	0.133	0.1386	0.0056	BS 1C	0.005
E	0.48	0.4935	0.0135	BS 3C	0.01
B	0.551	0.5404	–0.0106	CKD U	(0.0095)
A	0.75	0.7611	0.0111	BS 290A	0.01
D[A]	0.90	0.982	0.082	BS 2C	0.02
		Manganese			
F	0.21	0.2019	–0.008	BS 4C	0.01
D	0.67	0.6932	0.023	BS 2C	0.01
A	0.80	0.806	0.006	BS 290A	0.01
E	0.98	0.990	0.010	BS 3C	0.01
B	1.21	1.201	–0.009	CKD U	(0.012)
C	1.79	1.813	0.023	BS 1C	0.02
		Molybdenum			
A	0.024	0.0269	0.0029	BS 290A	0.002
F	0.105	0.1031	–0.0019	BS 4C	0.005
E	0.30	0.3018	0.0018	BS 3C	0.01
C	0.43	0.4459	0.0159	BS 1C	0.015
D	0.50	0.5015	0.0015	BS 2C	0.02
B	1.16	1.151	–0.009	CKD U	(0.0125)
		Nickel			
F	0.068	0.0654	–0.0026	BS 4C	0.002
A	0.088	0.0876	–0.0004	BS 290A	0.004
B	0.576	0.5722	–0.0043	CKD U	(0.0044)
E	0.75	0.7498	–0.0002	BS 3C	0.02
D	1.26	1.259	–0.001	BS 2C	0.03
C	1.99	1.981	–0.009	BS 1C	0.02
		Phosphorus			
F	0.003	0.0037	0.0007	BS 4C	0.001
A	0.024	0.0230	–0.0010	BS 290A	0.002
C	0.029	0.0300	0.0010	BS 1C	0.002
E	0.051	0.0502	–0.0008	BS 3C	0.003
D	0.078	0.0784	0.0004	BS 2C	0.002
B	0.414	0.4141	0.0001	CKD U	(0.007)
		Silicon			
F	0.52	0.527	0.007	BS 4C	0.02
E	1.08	1.082	0.002	BS 3C	0.02
A	1.89	1.917	0.027	BS 290A	0.02
C	2.02	2.058	0.038	BS 1C	0.02
B	2.25	2.224	–0.025	CKD U	(0.014)
D	2.51	2.519	0.009	BS 2C	0.05
		Sulfur			
F	0.001	0.0023	0.0013	BS 4C	0.0005
E	0.003	0.0058	0.0028	BS 3C	0.001
A	0.051	0.0464	–0.0046	BS 290A	0.002
C	0.060	0.0554	–0.0046	BS 1C	0.003
D	0.062	0.0576	–0.0044	BS 2C	0.004
B	0.077	0.0776	0.0007	CKD U	(0.0008)
		Tin			
A	0.011	0.0119	0.0009	BS 290A	0.001
D	0.031	0.0318	0.0008	BS 2C	0.002
B	0.057	0.0541	–0.0029	CKD U	(0.0010)
C	0.054	0.0561	0.0021	BS 1C	0.002
E	0.136	0.1367	0.0007	BS 3C	0.005
		Titanium			
A	0.012	0.0105	–0.0014	BS 290A	(0.0009)
B	0.035	0.0339	–0.0011	CKD U	(0.0011)
D	0.080	0.0805	0.0005	BS 2C	0.005
C	0.083	0.0865	0.0035	BS 1C	0.004
E	0.111	0.1114	0.0004	BS 3C	0.004
		Vanadium			
A	0.007	0.0071	0.0001	BS 290A	0.001
D	0.049	0.0500	0.0010	BS 2C	0.004
E	0.086	0.0851	–0.0009	BS 3C	0.008
C	0.12	0.1199	0.0001	BS 1C	0.01
B	0.221	0.2166	–0.0044	CKD U	(0.0057)

[A]Sample D is not included in determining the scope of testing in 1.1.

17. Maintaining Analytical Credibility

17.1 Users are encouraged to include this test method in an accountability and quality control program. Refer to Practice E1329 for procedures to control analysis, including the use of control charts. Support for the use of control charts with respect to a given standard appear in MNL 7A.

18. Keywords

18.1 cast iron; spark atomic emission; spectrometric analysis