



# Standard Test Method for Analysis of Cast Iron Using Optical Emission Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E 1999; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

<sup>ε1</sup> NOTE—Editorial changes were made in October 2000.

## 1. Scope

1.1 This test method covers the optical emission spectrometric analysis of cast iron by use of the point-to-plane technique for the following elements in the concentration ranges shown (Note 1):

Elements	Concentration Ranges, %	
	Applicable Range, %	Quantitative Range, % <sup>4</sup>
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

<sup>4</sup>Quantitative range in accordance with Practice E 1601.

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:

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials<sup>2</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E-01 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 03.05.

E 158 Practice for Fundamental Calculations to Convert Intensities into Concentrations in Optical Emission Spectrochemical Analysis<sup>2</sup>

E 172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis<sup>2</sup>

E 305 Practice for Establishing and Controlling Spectrochemical Analytical Curves<sup>2</sup>

E 351 Test Methods for Chemical Analysis of Cast Iron—All Types<sup>2</sup>

E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis<sup>3</sup>

E 826 Practice for Testing Homogeneity of Materials for the Development of Reference Materials<sup>3</sup>

E 1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys<sup>3</sup>

E 1059 Practice for Designating Shapes and Sizes of Non-graphite Counter Electrodes<sup>3</sup>

E 1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis<sup>3</sup>

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method<sup>3</sup>

E 1763 Guide for Interpretation and Use of Results from Interlaboratory Testing of Chemical Analysis Methods<sup>3</sup>

E 1806 Practice for Sampling Steel and Iron for Determination of Chemical Composition<sup>3</sup>

### 2.2 Other Documents:

MNL 7 Manual on Presentation of Data and Control Chart Analysis<sup>4</sup>

## 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 The most sensitive lines for carbon, phosphorus, sulfur and tin lie in the 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 evaluating all or portions of the spectrometer or filling all or portions of the spectrometer with an inert gas. A capacitor

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.06.

<sup>4</sup> *ASTM Manual Series, ASTM, 6th Edition, 1990.*

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.

## 5. Significance and Use

5.1 The chemical composition of cast iron alloys must 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 must be determined and proper corrections made by the use of various reference materials. 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 which is employed. Alternatively, mathematical corrections may be used to solve for interelement effects (refer to Practice E 158). Various mathematical correction procedures are commonly utilized. Any of these is acceptable, which will achieve analytical accuracy equivalent to that provided by this test method.

## 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 1/2 in.) in diameter and 3 to 12-mm (1/8 to 1/2-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 E 1806 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. See Practice E 172. 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 support gas. Gases and electrodes are described in 8.1 and 8.2.

NOTE 2—Clean the excitation chamber when the counter electrode is replaced. Clean the lens or protective window after approximately 200 to 300 excitations, or at a statistically determined time based on intensity loss, to minimize transmission losses.

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 to 500.0 nm. The spectrometers used to test this method had a dispersion of 0.3 to 0.6 nm/mm and a focal length of 0.5 to 0.75 m.

Spectral lines are listed in Table 1. The primary slit width is 15 to 50  $\mu\text{m}$ . Secondary slit width is 15 to 200  $\mu\text{m}$ . 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  $\mu\text{m}$  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*, 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 *Flushing System*, consisting of argon tanks or an argon-hydrogen gas mixture, a pressure regulator, and a gas flowmeter. Automatic sequencing shall be provided to actuate the flow of argon or argon-hydrogen mixture at a given flow rate for a

**TABLE 1 Analytical and Internal Standard Lines, Possible Interference**

Element	Wavelength, nm	Reported Possible Interfering Elements
Carbon	193.093	A1, Mo, Cu, S
Chromium	267.716 265.859	Mo, S, Mn
Copper	211.209 221.81 327.4 510.5	Ni  Mo, P V
Manganese	293.306	Cr, Mo, W
Molybdenum	202.03 281.61	Ni Mn
Nickel	243.789 231.604 341.4 352.45	Mn Mn  Mo
Phosphorus	178.287	Cr, Mn, Mo, Cu
Silicon	212.411 251.612 288.16	Mo, Cu, Ni  Mo, Cr
Sulfur	180.731	Mn, Cu, Cr
Tin	189.989	Mn, Mo, Fe
Titanium	334.904 337.2 334.2	Cr Fe
Vanadium	310.23 311.07	Ni
Iron <sup>A</sup>	273.074 271.4 281.33 360.89	

<sup>A</sup>Internal standard.

given time interval and to start the excitation at the end of the flush period. Means of changing the flow rate of argon or argon-hydrogen mixture shall be provided. The flushing system shall be in accordance with Practice E 406.

7.8 *Vacuum Pump*, if required, capable of maintaining a vacuum of 25  $\mu\text{m Hg}$ .

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

## 8. Reagents and Materials

8.1 *Inert gas, argon, nitrogen, and hydrogen*, as required, must 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 E 406.

8.2 *Counter Electrodes*—A silver or thoriated tungsten rod of 2 to 6-mm diameter ground to a 30 to 90° included angle conical tip, which conforms to Practice E 1059, has been found satisfactory.

NOTE 4—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. With a thoriated tungsten electrode, it has been reported that a hundred or more excitations usually can be made before replacement. Cleaning electrodes after each burn significantly reduces this buildup and gives more consistent results.

## 9. Reference Materials

9.1 *Certified Reference Materials*, used as calibrants, for chill-cast iron alloys are available commercially.

9.2 *Other Calibrants*, shall be chemically analyzed test specimens taken from production heats. 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. The metallurgical history of the calibrants should be similar to that of the specimens being analyzed. Refer to Test Methods E 351 and E 1019 or other nationally accepted test methods for chemical analysis of iron base alloys. Refer to Practice E 826 for information on homogeneity testing of reference materials.

9.2.1 In selecting calibrants, use caution with compositions that are unusual. One element may influence adversely 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 interelement effects, it is suggested that the calibrants approximate the composition of the material to be tested.

## 10. Preparation of Calibrants and Specimens

10.1 Cast graphite-free specimens from molten metal into a suitable mold and cool. The molten metal must be at a high enough temperature for all carbon to be in solution. Prepare the surface to be analyzed on a suitable belt or disk grinder. Prepare the surface of the specimens and reference materials in a similar manner. All specimens must be moisture-free for proper excitation in the argon atmosphere.

NOTE 5—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.

NOTE 6—Reference materials and specimens shall be refinished dry on a belt or disc sander before being re-excited on the same area.

## 11. Excitation and Exposure

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  $\pm 5\%$  and preferably should be held within  $\pm 2\%$ .

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

	Preburn	Exposure
Capacitance, $\mu\text{F}$	10	10
Inductance, $\mu\text{H}$	20	20
Resistance, $\Omega$	0	4.4
Potential, V	550	350
Number of discharges/s	120	60

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

Preflush period, s	2 to 10	
Preburn period, s	5 to 20	
Exposure period, s	5 to 20	
Argon Flow (Note 9)	$\text{ft}^3/\text{h}$	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 volatilization rates during specimen excitation. Once established, maintain the parameters consistently.

NOTE 9—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.

11.3 *Electrode System*— For conventional capacitor discharge excitation systems, the specimen, electrically negative, serves as one electrode. The opposite electrode is a thoriated tungsten or silver rod, the tip of which has been sharpened to a 30 to 90° included angle cone. Use a 3 to 6-mm (0.125 to 0.25-in.) gap. Once a gap size is selected, maintain a consistent gap. Center the analytical gap on the optical axes of the spectrometer. Condition a fresh counter electrode with 2 to 6 excitations using the conditions given in 11.1 and 11.2.

## 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.

NOTE 10—It is not within the scope of this test method to prescribe all details of equipment to be used. Equipment varies between laboratories.

NOTE 11—The lines listed in Table 1 have been proven satisfactory for the elements and concentration ranges described in the scope. Other internal standard and analytical lines may be used provided that it can be shown experimentally that equivalent precision and accuracy are obtained.

12.2 If required by the spectrometer, position or test the position of the spectrometer exit slits to ensure that peak radiation passes through each slit and is incident on the photomultiplier. This shall be done initially and as often as necessary thereafter to maintain proper alignment.

NOTE 12—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 conditions given in Section 10, make duplicate burns on the calibrants and potential standardants in a random sequence, bracketing these burns 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 averages of the data for each point, determine analytical curves as described in Practices E 158 and E 305.

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. If confirmation is not obtained, run another standardization or investigate why the system may be malfunctioning.

13.3.2 Repeat the verification at least every 4 h or if the instrument has been idle for more than 1 h. If readings are not within control limits, repeat the standardization.

### 14. Procedure for Excitation and Radiation Measurement

14.1 Produce and record the spectra using the conditions given in Section 11.

14.2 Check the standardization by verification as listed in 13.3.

14.3 *Replicate Exposure*—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 (Note 13). When placing the freshly surfaced specimen on the excitation stand, position it to effect a gas tight seal and adequate argon 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 (1/4 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.

NOTE 13—Examine the specimen and instrument measurements after each burn to evaluate the quality of excitation. Cracks, voids, pits, moisture, or inclusions will invalidate the sampling and accuracy of a determination.

### 15. Calculation of Results

15.1 Average the readings obtained for each specimen.

### 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.<sup>5</sup> The interlaboratory data were evaluated in accordance with Practices E 1601 and E 1763. 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<sub>c</sub>* 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 ( <i>S<sub>M</sub></i> , Practice E 1601)	Reproducibility SD ( <i>S<sub>R</sub></i> , Practice E 1601)	Reproducibility Index ( <i>R</i> , Practice E 1601)	<i>R<sub>rel</sub></i> %
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 <sup>A</sup>	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

<sup>5</sup> Supporting data have been filed at ASTM Headquarters. Request RR:E01-1027 to obtain a copy of this data.

TABLE 2 Continued

Test Material	Number of Laboratories	Found, %	Minimum SD ( $S_M$ , Practice E 1601)	Reproducibility SD ( $S_R$ , Practice E 1601)	Reproducibility Index ( $R$ , Practice E 1601)	$R_{rel}$ %
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

<sup>A</sup>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

	A	$K_R$	$K_{rel}$	B
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:

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

where:

$C_A$  = expected analyte content in %,

$B$  = 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 <sup>A</sup>	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

**TABLE 4** *Continued*

Test Material	Assumed True Value, %	Average Spectrometer Value, %	Difference, %	Material Identification, Uncertainty or (SD)	
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)

**TABLE 4** *Continued*

Test Material	Assumed True Value, %	Average Spectrometer Value, %	Difference, %	Material Identification, Uncertainty or (SD)	
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)

<sup>A</sup>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 E 1329 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 7.

## 18. Keywords

18.1 cast iron; optical emission; spectrometric analysis.

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