



Designation: **E1251–17** E1251 – 17a

# Standard Test Method for Analysis of Aluminum and Aluminum Alloys by Spark Atomic Emission Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E1251; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

## 1. Scope

1.1 This test method describes the analysis of aluminum and its alloys by ~~atomic~~ ~~spark-atomic~~ emission spectrometry: spectrometry (Spark-AES). The aluminum specimen to be analyzed may be in the form of a chill cast disk, casting, foil, sheet, plate, extrusion, or some other wrought form or shape. The elements covered in the scope of this method are listed in the table below.

Element	Tested Concentration—Mass Fraction
	Range (Wt %)
Antimony	0.001 to 0.003
Arsenic	0.001 to 0.006
Beryllium	0.0004 to 0.24
Bismuth	0.03 to 0.6
Boron	0.0006 to 0.009
Calcium	0.0002 to –
Chromium	0.001 to 0.23
Cobalt	0.4 to –
Copper	0.001 to 5.5
Gallium	0.02 to –
Iron	0.2 to 0.5
Lead	0.04 to 0.6
Lithium	0.0003 to 2.1
Magnesium	0.03 to 5.4
Manganese	0.001 to 1.2
Nickel	0.005 to 2.6
Phosphorus	0.003 to –
Silicon	0.07 to 16
Sodium	0.003 to 0.02
Strontium	0.03 to –
Tin	0.03 to –
Titanium	0.001 to 0.12
Vanadium	0.002 to 0.022
Zinc	0.002 to 5.7
Zirconium	0.001 to 0.12

NOTE 1—The ~~concentration-mass fraction~~ ranges given in the above scope were established through cooperative testing (ILS) of selected reference materials. The range shown for each element does not demonstrate the actual usable analytical range for that element. The usable analytical range may be extended higher or lower based on individual instrument capability, spectral characteristics of the specific element wavelength being used, and the availability of appropriate reference materials.

NOTE 2—Mercury (Hg) is intentionally not included in the scope. Analysis of Hg in aluminum by ~~spark-atomic emission spectrometry (Spark-AES)~~ Spark-AES is not recommended. Accurate analysis of Hg using this technique is compromised by the presence of an intense iron interference. Inaccurate reporting of Hg due to these interference effects can jeopardize the current designation of aluminum production as a mercury-free process. To demonstrate compliance with legislated Hg content limits, use of an alternate method capable of analysis with a minimum reporting limit of 0.0001% or lower is recommended. Suitable techniques include but are not limited to ~~glow discharge mass spectrometry, XRF, and cold vapor AA~~ GD-MS, XRF (X-ray fluorescence), cold vapor AA, and ICP-MS.

1.2 This test method is suitable primarily for the analysis of chill cast disks as defined in Practices **E716**. Other forms may be analyzed, provided that: (1) they are sufficiently massive to prevent undue heating, (2) they allow machining to provide a clean,

<sup>1</sup> 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 **E01.04** on Aluminum and Magnesium.

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flat surface, which creates a seal between the specimen and the spark stand, and (3) reference materials of a similar metallurgical condition and chemical composition are available.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific safety and health statements are given in Section 10.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

[B985 Practice for Sampling Aluminum Ingots, Billets, Castings and Finished or Semi-Finished Wrought Aluminum Products for Compositional Analysis](#)

[E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)

[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 \(Withdrawn 2004\)<sup>3</sup>](#)

[E172 Practice for Describing and Specifying the Excitation Source in Emission Spectrochemical Analysis \(Withdrawn 2001\)<sup>3</sup>](#)

[E305 Practice for Establishing and Controlling Atomic Emission Spectrochemical Analytical Curves](#)

[E406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E716 Practices for Sampling and Sample Preparation of Aluminum and Aluminum Alloys for Determination of Chemical Composition by Spark Atomic Emission Spectrometry](#)

[E826 Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry](#)

[E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data \(Withdrawn 2003\)<sup>3</sup>](#)

[E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis](#)

[E1507 Guide for Describing and Specifying the Spectrometer of an Optical Emission Direct-Reading Instrument](#)

### 2.2 ANSI Standard:<sup>4</sup>

[ANSI H35.1/H35.1M American National Standard Alloy and Temper Designation Systems for Aluminum](#)

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in this Standard, refer to Terminology [E135](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *alloy-type calibration*—calibration curves determined using ~~calibrants~~ calibration materials from alloys with similar compositions.

3.2.2 *binary-type calibration*—calibration curves determined using binary ~~calibrants~~ calibration materials (primary aluminum to which has been added one specific element).

3.2.3 *global-type calibration*—calibration curves determined using ~~calibrants~~ calibration materials from many different alloys with considerable compositional differences.

3.2.4 *two-point drift correction*—the practice of analyzing a high and low ~~standardant~~ standardization materials for each calibration curve and adjusting the ~~counts or voltage values~~ intensities obtained back to the values obtained on those particular ~~standardants~~ standardization materials during the collection of the calibration data. The corrections are accomplished mathematically and are applied to both the slope and intercept. Improved precision may be obtained by using a multi-point drift correction as described in Practice [E1329](#).

3.2.5 *type standardization*—mathematical adjustment of the calibration curve's slope or intercept using a single ~~standardant~~ standardization materials (reference material) standardization materials at or close to the nominal composition for the particular alloy being analyzed. For best results, the ~~standardant~~ standardization material being used should be within  $\pm 10\%$  of the composition (for each respective element) of the material being analyzed.

## 4. Summary of Test Method

4.1 A unipolar triggered capacitor controlled electrical discharge is produced in an argon atmosphere between the prepared flat surface of a specimen and the tip of a semi-permanent counter electrode. The energy of the discharge is sufficient to ablate material

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

<sup>4</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

from the surface of the sample, break the chemical or physical bonds, and cause the resulting atoms or ions to emit radiant energy. The radiant energies of the selected analytical lines and the internal standard line(s) are converted into electrical signals by either photomultiplier tubes (PMTs) or a suitable solid state detector. The detector signals are electrically integrated and converted to a digitized value. The signals are ratioed to the proper internal standard signal and converted into concentrations by a computer mass fractions in accordance with Practice E158.

4.2 Three different methods of calibration defined in 3.2.2, 3.2.3, and 3.2.1 are capable of giving the same precision, accuracy, and detection limit.

4.2.1 The first method, *binary calibration*, employs calibration curves that are determined using a large number of high-purity binary ~~calibrants—calibration materials~~. This approach is used when there is a need to analyze almost the entire range of aluminum alloys. Because binary ~~calibrants—calibration materials~~ may respond differently from alloy ~~calibrants—calibration materials~~, the latter are used to improve accuracy by applying a slope and/or intercept correction to the observed readings.

4.2.2 The second method, *global calibration*, employs calibration curves that are determined using many different alloy ~~calibrants—calibration materials~~ with a wide variety of compositions. Mathematical calculations are used to correct for both alloy difference and inter-element effects. Like the method above, specific alloy ~~calibrants—calibration materials~~ may be used to apply a slope and/or intercept correction to the observed readings.

4.2.3 The third method, *alloy calibration*, employs calibration curves that are determined using different alloy ~~calibrants—calibration materials~~ that have similar compositions. Again, specific alloy ~~calibrants—calibration materials~~ may be used to apply a slope and/or intercept correction to the observed readings.

## 5. Significance and Use

5.1 The metallurgical properties of aluminum and its alloys are highly dependent on chemical composition. Precise and accurate analyses are essential to obtaining desired properties, meeting customer specifications, and helping to reduce scrap due to off-grade material.

5.2 This test method is applicable to chill cast specimens as defined in Practices E716 and can also be applied to other types of samples provided that suitable reference materials are available. Also, other sample forms can be melted down and cast into a disk, using an appropriate mold, as described in Practices E716. However, it should be noted that some elements (for example, magnesium) readily form oxides, while some others (for example, sodium, lithium, calcium, and strontium) are volatile, and may be lost to varying degrees during the melting process.

## 6. Recommended Analytical Lines and Potential Interferences

6.1 Table 1 lists the analytical lines commonly used for aluminum analysis. Other lines may be used if they give comparable results. Also listed are recommended ~~concentration—mass fraction~~ ranges, background equivalent ~~concentrations (BEC)~~, ~~mass fractions~~, detection limits, useful linear ranges, and potential interferences. The values given in this table are typical; actual values obtained are dependent on instrument design.

NOTE 3—The ~~BEC—background equivalent mass fraction~~ and detection limits listed in Table 1 have been attained with a spectrometer that has a reciprocal dispersion of 54 nm/mm and a working resolution of 3.5 nm, using an entrance slit width of 25 μm and exit slit widths of 50 μm.

## 7. Apparatus

### 7.1 Specimen Preparation Equipment:

7.1.1 *Sampling Molds*, for aluminum and the techniques of pouring a sample disk are described in Practices E716. Chill cast samples, poured and cast as described within Practices E716, shall be the recommended form in this test method.

7.1.2 *Lathe*, capable of machining a smooth, flat surface on the reference materials and samples. A variable speed cutter, a cemented carbide or polycrystalline diamond tool bit, and an automatic cross feed are highly recommended. Proper depth of cut and desired surface finish are described in Practices E716.

7.1.3 *Milling Machine*, a milling machine can be used as an alternative to a lathe.

7.1.4 ~~*Milling Machine*, a milling machine can be used as an alternative to a lathe.~~ It is strongly recommended that the same preparation machinery used to prepare the standards is also used to prepare the samples. Differences in surface characteristics may influence the analysis.

NOTE 4—It is strongly recommended that the same preparation machinery used to prepare the standards is also used to prepare the samples. Differences in surface characteristics may influence the analysis.

7.2 ~~*Excitation Source*, *Source*—capable of producing a unipolar, triggered capacitor discharge.~~ In today's instrumentation, the excitation source is computer controlled and is normally programmed to produce: (1) a high-energy pre-spark (of some preset duration), (2) a spark-type discharge (of some preset duration), (3) an arc-type discharge (of some preset duration), and (4) a spark-type discharge, during which, time-resolved measurements are made for improved detection limits (this may be optional on some instruments).

7.2.1 Typical parameters and ~~exposure~~ times are given in Table 2. It should be emphasized that the information presented is given as an example only and parameters may vary with respect to instrument model and manufacturer. For details on describing and specifying an excitation source, please refer to Practice E172.

**TABLE 1 Recommended Analytical Lines**

Element	Wavelength in Air (nm) <sup>A</sup>	Recommended Concentration/Mass Fraction Range, %	Background Equivalent, % <sup>B</sup>	Calculated Detection Limit, % <sup>C,D</sup>	High Concentration/Mass Fraction Index, % <sup>E</sup>	Interferences Element, λ(nm) and k, % <sup>F</sup>
Aluminum	I-256.799	70-100				
Aluminum	256.799 I	70-100				
	I-266.039	70-100				
	266.039 I	70-100				
	I-237.208	70-100				
	237.208 I	70-100				
Antimony	I-231.147	0.001-0.5	0.17	0.0002		Ge-231.166 0.6
Antimony	231.147 I	0.001-0.5	0.17	0.0002		Co 231.166 0.6
	I-259.806	0.001-0.5		0.0002		Fe-259.837
	259.806 I	0.001-0.5		0.0002		Fe 259.837
						Mn 259.817 0.01
Arsenic	234.984 I	0.005-0.1				
Beryllium	I-234.861	0.0001-0.05	0.001	0.00003		
Beryllium	234.861 I	0.0001-0.05	0.001	0.00003		
	II-313.042	0.0001-0.05	0.0035	0.00001		
	313.042 II	0.0001-0.05	0.0035	0.00001		
	332.134 I	0.0001-0.05		0.00001		
Bismuth	I-306.772	0.001-0.7	0.04	0.0002		
Bismuth	306.772 I	0.001-0.7	0.04	0.0002		
Boron	I-249.773	0.0001-0.05	0.002	0.0001*		Fe-249.782 0.001
Boron	249.773 I	0.0001-0.05	0.002	0.0001*		Fe 249.782 0.001
						Mn 249.778 0.007
	I-249.678	0.0001-0.05				
	249.678 I	0.0001-0.05				
	I-208.959	0.0001-0.05				Mo-208.952 0.13
	208.959 I	0.0001-0.05				Mo 208.952 0.13
Cadmium	I-228.802	0.001-1	0.05	<0.0001		As-228.812
Cadmium	228.802 I	0.001-1	0.05	<0.0001		As 228.812
	I-479.992	0.005-2	0.15	0.003		
	479.992 I	0.005-2	0.15	0.003		
Calcium	II-393.367 <sup>G</sup>	0.001-0.05	0.001	0.00005		Fe-393.361
Calcium	393.367 II <sup>G</sup>	0.001-0.05	0.001	0.00005		Fe 393.361
Chromium	I-425.435	0.001-1	0.015	<0.0001		
Chromium	425.435 I	0.001-1	0.015	<0.0001		
	II-267.716	0.001-1	0.004	0.0005*		
	267.716 II	0.001-1	0.004	0.0005*		
	II-276.654 <sup>G</sup>	0.005-1				
	276.654 II <sup>G</sup>	0.005-1				
Cobalt	I-345.351	0.0001-2	0.0001	<0.0001		
Cobalt	345.351 I	0.0001-2	0.0001	<0.0001		
Copper	I-327.396	0.001-1.5	0.005	<0.0001	0.7	
Copper	327.396 I	0.001-1.5	0.005	<0.0001	0.7	
	I-324.754	0.001-0.5				
	324.754 I	0.001-0.5				
	I-296.117	0.05-20	0.40	0.01*	>20	Fe-296.128
	296.117 I	0.05-20	0.40	0.01*	>20	Fe 296.128
	II-224.700	0.01-5	0.03	0.0005*	5	
	224.700 II	0.01-5	0.03	0.0005*	5	
	I-510.554	0.05-20	0.32	0.01*	>20	
	510.554 I	0.05-20	0.32	0.01*	>20	
Gallium	I-294.364	0.001-0.05	0.015	<0.0001		
Gallium	294.364 I	0.001-0.05	0.015	<0.0001		
	I-417.206 <sup>G</sup>	0.001-0.05				Fe-417.213
	417.206 I <sup>G</sup>	0.001-0.05				Ti-417.190
						Cr-417.167
						Fe 417.213
						Ti 417.190
						Cr 417.167
Iron	II-238.204	0.001-1.5	0.015	0.0008	1.0	
Iron	238.204 II	0.001-1.5	0.015	0.0008	1.0	
	II-259.940	0.001-1.5	0.005	0.0004		
	259.940 II	0.001-1.5	0.005	0.0004		
	I-259.957					
	259.957 I					
	II-273.955	0.01-3.5				
	273.955 II	0.01-3.5				
	I-374.949 <sup>G</sup>	0.001-3.5		0.0001		
	374.949 I <sup>G</sup>	0.001-3.5		0.0001		
	I-441.512	0.01-3.5		0.0004		
	441.512 I	0.01-3.5		0.0004		
	I-438.355	0.005-3.5				
	438.355 I	0.005-3.5				

TABLE 1 Continued

Element	Wavelength in Air (nm) <sup>A</sup>	Recommended Concentration Mass Fraction Range, %	Background Equivalent, % <sup>B</sup>	Calculated Detection Limit, % <sup>C,D</sup>	High Concentration Mass Fraction Index, % <sup>E</sup>	Interferences Element, λ(nm) and k, % <sup>F</sup>
Lead	I-405.782	0.002-0.7	0.04	0.0001		Mn 405.792 0.01
Lead	405.782 I	0.002-0.7	0.04	0.0001		Mg 405.763 0.001 Mn 405.792 0.01 Mg 405.763 0.001
Lithium	I-283.306	0.002-0.7	0.07	0.002		
Lithium	283.306 I	0.002-0.7	0.07	0.002		
Lithium	I-610.364	0.0001-3				
Lithium	610.364 I	0.0001-3				
Lithium	I-670.784	0.0001-0.02	0.0005	<0.0005		
Lithium	670.784 I	0.0001-0.02	0.0005	<0.0005		
Lithium	I-323.261	0.01-3				Fe 323.279 Sb 323.250
Lithium	323.261 I	0.01-3				Fe 323.279 Sb 323.250
Magnesium	II-279.553	0.0005-0.3	0.0006	0.00003	0.04	
Magnesium	279.553 II	0.0005-0.3	0.0006	0.00003	0.04	
Magnesium	I-285.213	0.0005-0.3	0.008	<0.0001	0.25	
Magnesium	285.213 I	0.0005-0.3	0.008	<0.0001	0.25	
Magnesium	I-277.669	0.05-11	0.08	0.01	>11	
Magnesium	277.669 I	0.05-11	0.08	0.01	>11	
Magnesium	I-383.231 <sup>G</sup>	0.01-11	0.015	0.002*	>11	
Magnesium	383.231 I <sup>G</sup>	0.01-11	0.015	0.002*	>11	
Magnesium	I-383.826	0.1-11				
Magnesium	383.826 I	0.1-11				
Magnesium	I-518.362	0.01-11	0.02	0.002*	>11	
Magnesium	518.362 I	0.01-11	0.02	0.002*	>11	
Magnesium	I-403.076 <sup>G</sup>	0.001-0.1	0.028	0.0001*		
Magnesium	403.076 I <sup>G</sup>	0.001-0.1	0.028	0.0001*		
Magnesium	II-259.373	0.0005-0.5	0.004	0.00005	0.2	
Magnesium	II-293.306	0.001-2	0.006	0.0002*	>1.1	
Magnesium	259.373 II	0.0005-0.5	0.004	0.00005	0.2	
Magnesium	293.306 II	0.001-2	0.006	0.0002*	>1.1	
Magnesium	II-346.033 <sup>B</sup>	0.01-2				
Magnesium	346.033 II	0.01-2				
Nickel	I-341.476	0.001-2	0.02	<0.0001	>2.5	Zr 341.466 0.01
Nickel	341.476 I	0.001-2	0.02	<0.0001	>2.5	Zr 341.466 0.01
Nickel	I-310.188	0.005-4	0.05	0.001*	>5	
Nickel	310.188 I	0.005-4	0.05	0.001*	>5	
Nickel	II-231.604	0.001-2	0.015	0.0005*	<2.5	
Nickel	231.604 II	0.001-2	0.015	0.0005*	<2.5	
Phosphorus	I-178.231 <sup>H</sup>	0.0001-0.1	0.084	0.0001		
Phosphorus	178.231 I <sup>H</sup>	0.0001-0.1	0.084	0.0001		
Silicon	I-288.158	0.001-1.5	0.01	0.0001	1.5	Cr 288.123
Silicon	288.158 I	0.001-1.5	0.01	0.0001	1.5	Cr 288.123
Silicon	I-251.612	0.001-1.5	0.006	0.0001	1.5	
Silicon	251.612 I	0.001-1.5	0.006	0.0001	1.5	
Silicon	I-390.553 <sup>G</sup>	0.05-24	0.25	0.01	>24	Cr 390.566 0.09
Silicon	390.553 I <sup>G</sup>	0.05-24	0.25	0.01	>24	Cr 390.566 0.09
Silicon	I-212.415	0.05-24	0.5	0.05	>24	
Silicon	212.415 I	0.05-24	0.5	0.05	>24	
Silver	I-328.068	0.0005-0.1				
Silver	328.068 I	0.0005-0.1				
Silver	I-338.289	0.0001-0.1			>10	
Silver	338.289 I	0.0001-0.1			>10	
Silver	I-466.848	0.05-1.5				
Silver	466.848 I	0.05-1.5				
Sodium	I-588.995	0.0001-0.05	0.0015	<0.0001		
Sodium	588.995 I	0.0001-0.05	0.0015	<0.0001		
Strontium	II-421.552 <sup>G</sup>	0.0001-0.1	0.0004	0.0001		
Strontium	421.552 II <sup>G</sup>	0.0001-0.1	0.0004	0.0001		
Strontium	I-460.733	0.0005-0.06				
Strontium	460.733 I	0.0005-0.06				
Tin	I-317.502	0.001-7.5	0.04	0.0001	>10	
Tin	317.502 I	0.001-7.5	0.04	0.0001	>10	
Titanium	II-334.904	0.0005-0.5	0.004	<0.0001		
Titanium	334.904 II	0.0005-0.5	0.004	<0.0001		
Titanium	II-337.280	0.001-0.5	0.002	<0.00010		
Titanium	337.280 II	0.001-0.5	0.002	<0.00010		
Titanium	I-363.545	0.0005-0.05	0.030	0.003*		
Titanium	363.545 I	0.0005-0.05	0.030	0.003*		
Vanadium	I-318.341	0.001-0.15	0.06	0.0003*		
Vanadium	318.341 I	0.001-0.15	0.06	0.0003*		

TABLE 1 Continued

Element	Wavelength in Air (nm) <sup>A</sup>	Recommended Concentration/Mass Fraction Range, %	Background Equivalent, % <sup>B</sup>	Calculated Detection Limit, % <sup>C,D</sup>	High Concentration/Mass Fraction Index, % <sup>E</sup>	Interferences Element, λ(nm) and k, % <sup>F</sup>
Zinc	I-437.924	0.001-0.25				
	437.924 I	0.001-0.25				
	II-310.230	0.001-0.15	0.014	<0.0001		
	310.230 II	0.001-0.15	0.014	<0.0001		
	I-213.856	0.0005-0.1	0.035	0.0001*	0.05	
	213.856 I	0.0005-0.1	0.035	0.0001*	0.05	
	I-334.502	0.001-10.0	0.065	0.0004	>8	
	334.502 I	0.001-10.0	0.065	0.0004	>8	
	I-481.053	0.01-10	0.07	0.001*	>10	
	481.053 I	0.01-10	0.07	0.001*	>10	
Zirconium	I-472.216	0.01-10	0.26	0.0015	>10	
	472.216 I	0.01-10	0.26	0.0015	>10	
	II-339.198	0.001-1	0.02	0.001*		
	339.198 II	0.001-1	0.02	0.001*		
Zirconium	II-349.621 <sup>G</sup>	0.001-1	0.006	<0.0001		
	349.621 II <sup>G</sup>	0.001-1	0.006	<0.0001		

<sup>A</sup> I = atom line, II = ion line. Second (2nd) indicates that the second order shall be used, where available.  
<sup>B</sup> Background Equivalent Concentration (BEC) Mass Fraction—The concentration-mass fraction at which the signal due to the element is equal to the signal due to the background.  
<sup>C</sup> In this test method, the calculated detection limit was measured by calculating the standard deviation of ten consecutive burns on a specimen with element concentration(s)-mass fraction(s) at levels below ten times the expected detection limit.  
<sup>D</sup> See footnote C. For values marked with an asterisk (\*) the available data were for a concentration-mass fraction greater than ten (10) times but less than a hundred (100) times the expected detection limit.  
<sup>E</sup> High Concentration-Mass Fraction Index—The concentration-mass fraction at which the slope of the calibration curve drops below 0.75.  
<sup>F</sup> Interference Factor, k—The apparent increase in the concentration-mass fraction of the element being determined, expressed in percent, due to the presence of 1.0 % of the interfering element.  
<sup>G</sup> Useful analytical lines with improved signal to background ratios due to the complete removal of C-N background by the argon atmosphere.  
<sup>H</sup> If phosphorus is to be determined, the most sensitive line appears to be the 178.231 nm in the second order which requires either a vacuum or a gas filled spectrometer. The vacuum spectrometer should be operated at a pressure of 25 microrr or less. The gas filled spectrometer will be charged with nitrogen to a positive pressure of slightly over one atmosphere (101 k pa). Optimum results are obtained by using a background channel that has been profiled "off peak" of the first order 178.231 nm phosphorus line as the internal standard. The ratio of P 178.231 nm (2nd) / background near the 178.231 nm (1st) is plotted against % phosphorus. Even with this compensation for variability in background, alloys with highly different compositions of major alloying elements, particularly silicon, require separate reference materials and analytical curves.

TABLE 2 Typical Excitation Source Electrical Parameters

Parameter	High Energy Pre-spark	Spark	Arc
Resistance, Ω	1	1	15
Inductance, μH	30	130	30
Volts, V	400	400	400
Frequency, Hz	300	300	300
Capacitance, μF	12	3	5
Time, s	10	5	5

7.3 Excitation Chamber, shall be designed with an upper plate that is smooth and flat so that it will mate (seal) perfectly with the prepared surface of the sample specimen. The seal that is formed between the two will exclude atmospheric oxygen from entering the discharge chamber. The excitation chamber will contain a mounting clamp to hold the counter electrode. The excitation stand assembly will also have some type of clamp or device designed to hold the sample firmly against the top plate. Some manufacturers may provide for the top plate to be liquid cooled to minimize sample heat-up/heating during the excitation cycle. The excitation chamber will also be constructed so that it is flushed automatically with argon gas during the analytical burn cycle. The excitation chamber's design should allow for a flow of argon gas to prevent the deposition of ablated metal dust on the inner-chamber quartz-window(s). The excitation chamber will be equipped with an exhaust system that will safely dispose of the argon gas and the metal dust created during the excitation cycle. For reasons of health and cleanliness, the exhausted gas and dust should not be vented directly into the laboratory. To help with this situation, manufacturers have designed their instruments with some type of exhaust/filter system to deal with minimize this problem. The exhaust can then be vented into an efficient hood system.

7.4 Gas Flow System, will be designed so that it can deliver pure argon gas to the excitation chamber. The purity of the argon gas will affect the precision of the results. Generally, precision improves as the purity of the argon gas gets higher. Argon gas with a minimum purity of 99.995 % has been found to be acceptable. The gas shall be delivered by a flow system as described in Practice E406. The argon gas source can be from high-purity compressed gas cylinders, a cryogenic-type cylinder that contains liquid argon or possibly from a central supply (liquid only). It is essential that only argon gas meeting the minimum purity of 99.995 % be used. A lower purity grade of argon, such as a "welding grade," should not be used. The delivery system shall be