



Designation: E 1251 – 94 (Reapproved 1999)

# Standard Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Argon Atmosphere, Point-to-Plane, Unipolar Self-Initiating Capacitor Discharge<sup>1</sup>

This standard is issued under the fixed designation E 1251; 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.

## 1. Scope

1.1 This test method provides for the optical emission spectrometric analysis of aluminum and aluminum alloys, in cast or wrought form, for the following elements in the concentration ranges indicated:

Element	Concentration, Range, %
Silicon	0.001 to 24.0
Copper	0.001 to 20.0
Magnesium	0.001 to 11.0
Zinc	0.001 to 10.0
Tin	0.001 to 7.5
Nickel	0.001 to 4.0
Iron	0.001 to 3.5
Lithium	0.005 to 3.0
Manganese	0.001 to 2.0
Cobalt	0.001 to 2.0
Silver	0.001 to 1.5
Chromium	0.001 to 1.0
Zirconium	0.001 to 1.0
Lead	0.002 to 0.7
Bismuth	0.001 to 0.7
Cadmium	0.001 to 0.5
Beryllium	0.0001 to 0.5
Titanium	0.001 to 0.5
Antimony	0.001 to 0.5
Vanadium	0.001 to 0.15
Strontium	0.0001 to 0.07
Gallium	0.001 to 0.05
Sodium	0.0001 to 0.05
Boron	0.0001 to 0.05
Barium	0.0001 to 0.05
Calcium	0.001 to 0.05
Phosphorus	0.0001 to 0.01

1.2 This test method is suitable primarily for the control analysis of chill-cast specimens. Other forms may be analyzed, provided that (1) they are sufficiently massive to prevent undue heating, (2) they permit machining flat surfaces having a minimum diameter of approximately 15 mm, and (3) reference materials of similar metallurgical condition and chemical composition are available.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.04 on Aluminum and Magnesium.

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1.3 This standard does not purport to address all of the safety problems, 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. Specific safety hazard statements are given in 8.2 and Section 10.

## 2. Referenced Documents

### 2.1 ASTM Standards:

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

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 227 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique<sup>2</sup>

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

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)<sup>3</sup>

E 406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis<sup>4</sup>

E 607 Test Method for Optical Emission Spectrometric Analysis of Aluminum and Aluminum Alloys by the Point-to-Plane Technique, Nitrogen Atmosphere<sup>4</sup>

E 716 Practices for Sampling Aluminum and Aluminum Alloys for Spectrochemical Analysis<sup>4</sup>

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data<sup>4</sup>

## 3. Terminology

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

<sup>2</sup> Annual Book of ASTM Standards, Vol 03.05.

<sup>3</sup> Annual Book of ASTM Standards, Vol 14.02 (excerpts in all other volumes).

<sup>4</sup> Annual Book of ASTM Standards, Vol 03.06.

### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *alloy-type calibration*—the grouping of alloys of similar composition for the purpose of calibration so as to permit standardization with a minimum number of reference materials, for example, high silicon-aluminum casting alloys, American Aluminum Association alloy numbers 308 to 380, may be standardized with 380 standardant.

3.2.2 *standardization*—See Terminology E 135. Two types of standardization are commonly used, as follows

3.2.2.1 *two-point standardization*—adjustment of a gain control of a channel for an individual spectral line in a manner that reproduces the readings that the high and low standardants displayed during the collection of calibration data. In computer applications, correction is done mathematically by applying a slope and intercept correction, that is, a multiplication to correct the amount of difference between the high and low standardant readings followed by the addition or subtraction of a constant to finally restore readings to expected values.

3.2.2.2 *single-point standardization*—adjustment of a channel for an individual spectral line using a single standardant. Usually the single standardant is a high reference material used to set the gain. If the analytical interest is just in low concentrations near the detection limit, a low standardant is used and either a gain or a background control may be adjusted.

## 4. Summary of Test Method

4.1 A unipolar self-initiating capacitor discharge is produced in an argon atmosphere between a prepared flat surface of the specimen and the tip of a semi-permanent thoriated-tungsten or other counter electrode. The radiant energies of selected analytical lines and an internal standard line are converted into electrical currents by photomultipliers. The currents are integrated during the exposure time either by charging integrating capacitors and measuring the cumulative voltages at the end of the exposure, or by interrogating the capacitors during the exposure and converting the voltages into

digital counts by means of a computer. The ratios of the voltages of the analytical lines to the voltage of the internal standard line are converted into concentrations either manually by graphs, tables, or X-Y recorders, or by a computer in accordance with Practice E 158.

4.2 Two alternative procedures for calibration may be employed. The two procedures can give the same precision, accuracy, and detection limits.

4.2.1 The first procedure, *Binary Calibration*, is used when there is a need to analyze almost the entire range of aluminum alloys. This procedure employs calibration curves for each element that cover the entire concentration range that has to be determined in all alloys. Fifty or more binary calibrants may be required for calibration. *Type* reference materials are used to adjust the concentrations read from the binary curves in order to report the correct concentrations for specific alloys.

4.2.2 The second procedure, *Alloy-Type Calibration*, is more appropriate for analyzing only a relatively few alloys of similar composition. It employs an analytical curve for each element that covers a relatively limited concentration range. Calibration may require only 5 to 20 calibrants.

## 5. Significance and Use

5.1 The physical and chemical properties of high-purity aluminum and aluminum alloys depend on chemical composition, which must be determined and controlled. Accurate, high-speed analysis of aluminum, before it is poured from the furnace, can prevent scrapped heats and minimize the cost of expensive alloying metals.

5.2 This test method is applicable to chill-cast specimens prepared for routine production control. It can be applied to other types of specimens if there are appropriate reference materials, or if the specimens are remelted.

## 6. Interferences

6.1 Known line interferences due to other elements are listed in Table 1.

**TABLE 1 Analytical Lines and Concentration Ranges**

Element	Wavelength In Air, Å <sup>A,B,C</sup>	Recommended Concentration Range, %	Background Equivalent, % <sup>D</sup>	Detection Limit, % <sup>E,F</sup>	High- Concentration Index, % <sup>G</sup>	Interferences	
						Element, λ, Å, and k, % <sup>H</sup>	
Silicon	I 2881.58	0.001–1.5	0.01	0.0001	1.5	Cr 2881.93	
	I 2516.12	0.001–1.5	0.006	0.0001	1.5		
	I 3905.53 <sup>B</sup>	0.05–24	0.25	0.01	>24	Cr 3905.66	0.09
	I 2124.15	0.05–24	0.5	0.05	>24		
Iron	II 2382.04	0.001–1.5	0.015	0.0008	1.0		
	II 2599.40	0.001–1.5	0.005	0.0004			
	I 2599.57						
	I 3749.49 <sup>B</sup>	0.001–3.5		0.0001 <sup>I</sup>			
	I 4415.12	0.01–3.5		0.0004			
Copper	I 4383.55	0.005–3.5	0.05				
	I 3273.96	0.001–1.5	0.005	<0.0001	0.7	Fe 2961.28	
	I 2961.17	0.05–20	0.40	0.01 <sup>F</sup>	>20		
Manganese	II 2247.00	0.01–5	0.03	0.0005 <sup>F</sup>	5		
	I 5105.54	0.05–20	0.32	0.01 <sup>F</sup>	>20		
	I 4030.76 <sup>B</sup>	0.001–2	0.028	0.0001 <sup>F</sup>			
	II 2593.73	0.0005–0.5	0.004	0.00005	0.2		
	II 2933.06	0.001–2	0.006	0.0002 <sup>F</sup>	>1.1		
Magnesium	II 3460.33 <sup>B</sup>	0.01–2					
	II 2795.53	0.0005–0.3	0.0006	0.00003	0.04		

Element	Wavelength In Air, Å <sup>A,B,C</sup>	Recommended Concentration Range, %	Background Equivalent, % <sup>D</sup>	Detection Limit, % <sup>E,F</sup>	High- Concentration Index, % <sup>G</sup>	Interferences	
						Element, λ, Å, and k, % <sup>H</sup>	
Chromium	I 2852.13	0.0005–0.3	0.008	<0.0001	0.25		
	I 2776.69	0.05–11	0.08	0.01	>11		
	I 3832.31 <sup>B</sup>	0.01–11	0.015	0.002 <sup>F</sup>	>11		
	I 5183.62	0.01–11	0.02	0.002 <sup>F</sup>	>11		
	I 4254.35	0.001–1	0.015	<0.0001			
	II 2677.16	0.001–1	0.004	0.0005 <sup>F</sup>			
Nickel	II 2766.54	0.005–1					
	I 3414.76	0.001–2	0.02	<0.0001	>2.5		
	I 3101.88	0.005–4	0.05	0.001 <sup>F</sup>	>5		
Zinc	II 2316.04 (2nd)	0.001–2	0.015	0.0005 <sup>F</sup>	<2.5		
	I 2138.56 (2nd)	0.0005–0.1	0.035	0.0001 <sup>F</sup>	0.05		
	I 3345.02	0.001–10.0	0.065	0.0004	>8		
	I 4810.53	0.01–10	0.07	0.001 <sup>F</sup>	>10		
Titanium	I 4722.16	0.01–10	0.26	0.0015	>10		
	II 3349.04	0.0005–0.5	0.004	<0.0001			
	II 3372.80	0.001–0.5	0.002	<0.0001			
Vanadium	I 3635.45	0.0005–0.05	0.030	0.0003 <sup>F</sup>			
	I 3183.41	0.001–0.15	0.06	0.0003 <sup>F</sup>			
	II 3102.30	0.001–0.15	0.014	<0.0001			
Lead	I 4057.82	0.002–0.7	0.04	0.0001		Mn 4057.92	0.01
	I 2833.06	0.002–0.7	0.07	0.002			
Tin	I 3175.02	0.001–7.5	0.04	0.0001	>10		
Bismuth	I 3067.72	0.001–0.7	0.04	0.0002			
Gallium	I 2943.64	0.001–0.05	0.015	<0.0001			
	I 4172.06 <sup>B</sup>	0.001–0.05					
Boron	I 2497.73 (2nd)	0.0001–0.05	0.002	0.0001 <sup>F</sup>		Fe 2497.82	0.001
						Mn 2497.78	0.007
Beryllium	I 2089.59 (2nd)	0.0001–0.05				Mo 2089.52	0.13
	I 2348.61	0.0001–0.05	0.001	0.00003			
	II 3130.42	0.0005–0.05	0.0035	0.0001			
	I 3321.34	0.0001–0.05		0.0001			
Sodium	I 5889.95	0.0001–0.05	0.0015	<0.0001			
Calcium	II 3933.67 <sup>B</sup>	0.001–0.05	0.001	0.00005		Fe 3933.61	
Zirconium	II 3391.98	0.001–1	0.02	0.001 <sup>F</sup>			
	II 3496.21 <sup>B</sup>	0.001–1	0.006	<0.0001			
Cobalt	I 3453.51 <sup>B</sup>	0.0001–2		<0.0001			
Cadmium	I 2288.02	0.001–0.3	0.05	<0.0001		As 2288.12	
	I 4799.92	0.005–2	0.15	0.003			
Lithium	I 6707.84	0.0001–0.02	0.0005	<0.00005		Sb 3232.50	
	I 3232.61	0.01–3				Fe 3232.79	
Antimony	I 2311.47 (2nd)	0.001–0.5	0.17	0.0002		Co 2311.66	0.6
	I 2598.06	0.001–0.5		0.0002		Fe 2598.37	
Strontium	II 4215.52 <sup>B</sup>	0.0001–0.1	0.0004	0.0001			
Barium	II 4554.04	0.0001–0.1	0.0004	0.0001			
Phosphorous	I 1782.31 (2nd) <sup>I,J</sup>	0.0001–0.1	0.084	0.0001			
Silver	I 3280.68	0.0005–0.1					
	I 3382.89	0.0001–0.1			>10		
	I 4668.48	0.05–1.5					
Aluminum	I 2567.99	70–100					
	I 2660.39	70–100					
	I 2372.08	70–100					

<sup>A</sup> I = atom line, II = ion line.

<sup>B</sup> Useful analytical lines with improved signal to background ratios due to the complete removal of C-N background by the argon atmosphere.

<sup>C</sup> Second (2nd) indicates that the second order shall be used, where available.

<sup>D</sup> Background Equivalent—The concentration at which the signal due to the element is equal to the signal due to the background.

<sup>E</sup> In this test method, the detection limit was measured by calculating the standard deviation of ten concentration printouts of a specimen between three and ten times the expected detection limit.

<sup>F</sup> See footnote E. For the values marked with an F, the available data was for a concentration greater than ten times but less than 100 times the expected detection limit.

<sup>G</sup> High-Concentration Index—The concentration at which the tangent to the calibration curve plotted on log-log paper drops from 45° theoretical to 37°, that is, the response of  $I_a/I_s$  as ordinate versus % a as abscissa has dropped from approximately 1.0 to 0.75. It is recommended that a second, less sensitive line with close to 45° response be employed before this concentration is reached.

<sup>H</sup> Interference Factor, k—The apparent increase in the concentration of the element being determined, expressed in percent, due to 1.0 % of the interfering element.

<sup>I</sup> The detection limit for iron is determined more by possible segregation in the specimen and by contamination during machining than by spectrochemical sensitivity.

<sup>J</sup> If phosphorous is to be determined, the most sensitive line appears to be 1782.31 Å in the second order and this requires a vacuum spectrometer. Optimum results are obtained by using as an internal standard a background channel profiled off the peak of phosphorous 1782 Å in the first order. The ratio of P1782.31 Å (2nd)/background near 1782 Å (1st) is plotted against % P. 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.

## 7. Apparatus

### 7.1 Specimen Preparation Equipment:

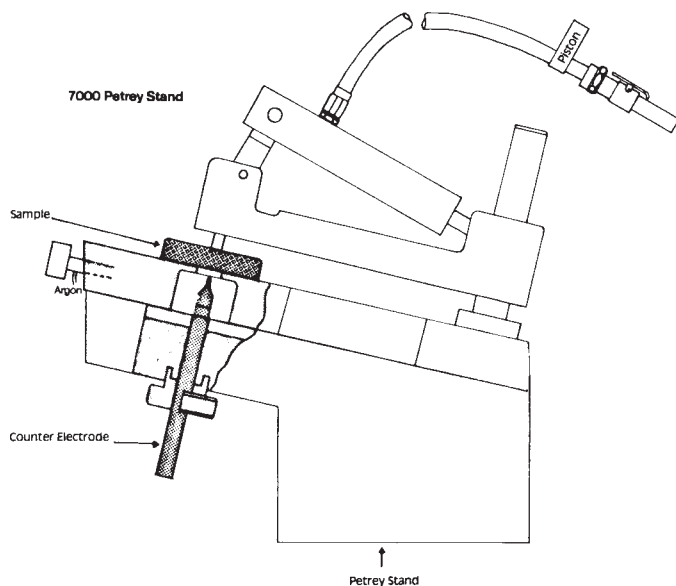
7.1.1 *Specimen Molds* for aluminum and methods for pouring specimens are described in Practice E 716. Chill-cast specimens, poured as described therein, shall be used in this test method.

7.1.2 *Lathe*, capable of machining a smooth, flat surface on the reference materials and specimens. A variable-speed cutter, a cemented-carbide tool, and an automatic cross-feed are recommended.

7.1.3 *Milling Machine*—A vertical milling machine with fly-cutter and vise for holding the specimen can be used as an alternative to the lathe.

7.2 *Excitation Source*, capable of producing a unipolar self-initiating capacitor discharge, utilizing the parameters described in 13.2 or equivalent, and in accordance with Practice E 172. Each high-frequency wave train is fully rectified, so that the specimen is always the cathode, and only specimen material is introduced into the analytical gap. The counter electrode is not attacked, and can be used for analyzing many specimens.

7.3 *Excitation Chamber*, similar to that shown in Fig. 1, for mounting a machined, flat surface of the specimen so that it forms a seal to exclude atmospheric oxygen from the discharge. The excitation chamber shall be provided with a counter electrode holder and with a clamp to hold the specimen firmly against the upper plate. The upper plate shall be inclined at an angle of 10 to 15° to the light path. Depending upon the manufacturer, the excitation chamber may require water-cooling, or a diaphragm may be inserted in the chamber to occlude any radiation from the hot specimen or the counter electrode, or both may be required. The excitation chamber shall be designed so that it can be flushed automatically with argon gas. Argon gas shall be introduced just inside the fused quartz window, flowing toward the discharge, to prevent any of the metal powder produced at the discharge from coating the inside of the window. Argon also may be introduced around the



**FIG. 1 Excitation Stand**

counter electrode, but the flow rate shall not confine the discharge to an area less than 5 mm in diameter, to provide adequate sampling of the specimen. The argon, with most of the metal dust, shall be exhausted to the atmosphere from the back or side of the chamber.

7.4 *Gas Flow System*, to supply pure argon gas (99.995 %) to the excitation chamber. The gas shall be delivered by a flow system as described in Practice E 406 from either high-purity compressed gas, or liquid argon bottles, or from a welding grade supply that has been purified to the required 99.995 % level. The system shall include: a two-stage regulator of all-metal construction with two pressure gages; copper tubing with all-metal seals; a solenoid and valve operated automatically by the control system, to start the full argon flow for the pre-flush and stop it at the end of the exposure; and a needle valve to maintain a very slow bleed of argon through the excitation chamber when it is not in active use (Note 1). The copper tubing shall be kept as short as possible—preferably only a few feet. All connections shall be leak-free. A flow gage is required to check the flow rates. The flow gage need not be incorporated in the system permanently, but may be connected at the outlet temporarily while initiating adjusting the operating flow, and then can be removed. This keeps the system as simple as possible and eliminates a possible source of organic contaminants or leaks.

NOTE 1—All-metal connections are required because the discharge is adversely affected by organic contamination, or by as little as 2 ppm of oxygen or a few ppm of water vapor.

7.5 *Spectrometer*, with a reciprocal linear dispersion of 10 Å/mm, or better, and a combination of entrance and exit slit widths that provides a working resolution of at least 0.7 Å (Note 2). The wavelength coverage must include the analytical lines required for the needed concentration ranges, as listed in Table 1.

NOTE 2—According to Practice E 380, the Angstrom, Å, is a deprecated unit. However, it is employed here for the convenience of the users of this test method who may wish to refer to the wavelength tables which contain Angstrom units. An *Angstrom* is defined as 0.10 of a nanometre.

NOTE 3—If phosphorus is to be measured, the optimum line is 1782 Å, which is strongly absorbed by oxygen; therefore, a vacuum spectrometer is required, operating at a pressure of 25 microtorr or less.

NOTE 4—The background equivalents and detection limits listed in Table 1 can be attained with a reciprocal linear dispersion of 5.4 Å/mm and a working resolution of 0.35 Å, using an entrance slit width of 25 μm and an exit slit width of 50 μm.

7.6 *Measuring System*, consisting of photomultipliers having individual voltage adjustments, integrating capacitors, or digital counting circuitry, an amplifier and some form of readout system. The system also may contain the program circuitry that times the flush, pre-burn and exposure intervals, turns the source on and off, etc. The measuring system may read only intensity ratios (voltage ratios), which then have to be converted into concentrations manually, or the system may use a computer or programmable calculator to convert intensity ratios into concentrations.

7.6.1 *Manual Measuring System*, with an individual voltage or amplifier gain control and a zero adjustment control for each channel, to permit standardization as required. Note 5



NOTE 5—The gain and zero controls can be omitted if the analytical program is restricted to a few alloy types, the concentration ranges in each are restricted, and the gain and background levels do not vary widely from day to day. Then some mechanism for single point standardization, such as a calculating board with movable concentration scales, may be used to compensate for instrumental drift.

7.6.1.1 *Intensity Readout System*, which displays the ratio of the radiant energy of the analytical line to the radiation of the internal standard line, either as a digital number read visually or typed automatically, or as a deflection on a strip chart recorder or X-Y recorder.

7.6.1.2 *Graphs, Flip Charts, or Calculating Boards*, used in conjunction with an intensity readout system, to convert digital intensity ratios into concentrations. With a strip chart or X-Y recorder, concentration scales may be preprinted on the paper, as illustrated in Test Method E 227.

7.6.2 *Computer Readout System or Programmable Calculator*, which stores the analytical curves in memory and converts the intensity ratios into concentrations (see X1.1 of Appendix X1). A printout of the analysis is recommended because it provides a permanent record and eliminates clerical errors, but display of results on a video scope is acceptable. Manual adjustments are not needed during standardization because gain and background adjustment factors can be calculated automatically.

8. Materials

8.1 *Counter Electrode*—The preferred counter electrode, consisting of a needle-point thoriated-tungsten wire mounted in a copper rod, is illustrated in Fig. 2(a). An alternative counter electrode, a 6.3-mm diameter tungsten or silver rod with a conical tip, is illustrated in Fig. 2(b).

8.2 *Argon Gas*, of 99.995 % purity. Cylinders of either compressed argon gas or liquid argon may be employed. Alternatively, argon gas of the requisite purity can be produced from a welding grade supply by passing it through a titanium

furnace which removes oxygen, nitrogen, water, and oil vapors, and other impurities. See Practice E 406 for precautions to be used in handling gases.

NOTE 6—A magnesium furnace is not acceptable because it will introduce variable magnesium contamination.

9. Reference Materials

9.1 *Calibrants* shall be homogeneous and free of cracks or porosity, with a metallurgical condition similar to that of the unknown specimens to be analyzed. They shall be used to produce the analytical curves for various elements being determined.

9.1.1 If an alloy-type calibration structure is used, it is desirable to have at least four calibrants for each element (see 3.2.1). The concentrations should be fairly evenly spaced over the anticipated analytical range, to establish a statistically sound smooth curve through all points.

9.1.1.1 If analytical lines are to be used above the concentrations at which they may start to self-absorb, it is necessary to have more calibration reference materials in the self-absorbed region, to define the shape of the curve.

9.1.2 The composition of the calibrants shall have been determined chemically at least to the accuracy required for the final analytical results. A few *benchmark Certified Reference Materials* are available from the National Institute of Standards and Technology<sup>5</sup> and many other reference materials are available commercially in both chill-cast and wrought forms.<sup>6</sup>

9.1.3 For several trace elements, available reference materials contain variable concentrations of the trace element in a typical alloy of constant composition. These reference materials can be used for establishing an analytical curve, but will not reveal potential interferences from nearby lines of other elements, or matrix effects that change the background or response. For optimum usefulness, several of the calibrants should have concentrations of the other elements that cover the ranges to be expected in the unknown specimens.

NOTE 7—Fortunately with aluminum, there are relatively few interferences because the aluminum spectrum itself and the spectra of the major alloying elements (silicon, copper, magnesium, zinc, and tin) are simple. The alloying elements with complex spectra (nickel, manganese, iron, cobalt, chromium, zirconium, titanium, and vanadium) are present at concentrations so low that only the more intense lines can cause interferences.

NOTE 8—Reference materials and specimens modified with sodium may produce blacker burns with deeper penetration of the spark into the surface. This may result in a drop in the intensity of the aluminum internal standard below its normal level, and a corresponding increase in the intensity ratios for the analytical lines. This, in turn, may produce concentration readings for silicon that are too high by 0.3 % at the 5 % to 7 % level, and a similar increase for other elements. If nonmodified specimens are to be analyzed with calibration curves established with modified reference materials (or vice versa), corrections for such effects shall be applied.

<sup>5</sup> Office of Standard Reference Materials, National Institute of Standards and Technology, U. S. Department of Commerce, Gaithersburg, MD 20899.

<sup>6</sup> Aluminum reference materials are available from Aluminum Company of America, Technology Marketing Division, 18th Floor, Alcoa Building, Pittsburgh, PA 15219, and Analytical Reference Materials International, Inc., P.O. Box 2246, Evergreen, CO 80439-2246.



(a) Needle-Point, Thoriated-Tungsten Wire Mounted in Copper Rod (b) Tungsten or Silver Rod with Conical Tip

FIG. 2 Counter Electrodes

## 9.2 Standardants:

9.2.1 *High-Concentration Standardants*— These may be selected commercially available reference materials or, preferably, reference materials prepared *in-house*, that have been tested for homogeneity and have among them concentration levels near or above the median concentration for each element. A single standardant for each matrix is preferable, but several may be used. The exact composition need not be known, since the standardants shall be used only to adjust the instrument to reproduce the intensity ratios which they produced at the time the analytical curves were established.

9.2.2 *High-Purity or Blank Standardants*— These shall be homogeneous, with the lowest available concentrations of the elements being determined, but their exact compositions need not be known. Usually, the background readings of a spectrometer remain relatively constant. Therefore, a blank standardant for an element does not need to be used in routine standardization unless the lowest concentration that needs to be determined is within ten times the detection limit of that element. For specimens with such low concentrations, single point standardization, either with the blank or another specimen, may give more consistent results.

## 10. Safety Precautions

10.1 All high-voltage sources can give painful shocks if the operating personnel come in contact with the high voltage when the source is turned on. The excitation chamber shall be enclosed and shall be provided with a safety interlock so that the source cannot be turned on until the door is closed. Access to the power supply shall be permitted only by removing panels which contain safety interlocks or require the use of tools.

10.2 Fumes of fine metallic powders exhausted from the excitation chamber can be poisonous if specimens contain significant levels of heavy metals such as cadmium or lead. Such metals shall be trapped by bubbling the exhaust through water or a filter system, or the exhaust shall be vented to the atmosphere at a nonhazardous location.

## 11. Sampling

11.1 For the sampling of molten aluminum metal and the casting of disk specimens, see Practice E 716.

## 12. Preparation of Reference Materials and Specimens

12.1 *Preparation of Reference Materials*— The reference material shall be machined in the same manner as the specimens.

12.2 *Preparation of Specimens*—For the preparation of chill-cast disk specimens and other types of aluminum specimens, see Practice E 716.

NOTE 9—For optimum precision and accuracy, the reference materials and specimens shall have been freshly machined no longer than 1 h before use, to avoid the formation of aluminum oxide.

## 13. Preparation of Apparatus

13.1 Prepare and operate the spectrometer in accordance with the manufacturer's instructions.

NOTE 10—It is not within the scope of this test method to prescribe minute details relative to the preparation of the apparatus. For a descrip-

tion and specific details concerning the operation of a particular spectrometer, refer to the manufacturer's manual.

13.1.1 Program the spectrometer to accommodate the internal standard line or lines (or background settings) and the pertinent analytical lines listed in Table 1. Connect the photomultipliers, capacitors, and related measuring system.

NOTE 11—The argon atmosphere eliminates the molecular bands of cyanogen (CN) that are produced with excitations in air (see Test Method E 227) or nitrogen (see Test Method E 607). The absence of CN bands provides improved signal to background ratios for some analytical lines and permits the use of other lines. The lines listed have proven satisfactory for the elements and concentration ranges prescribed in the Scope. Other internal standard and analytical lines may be used, provided it is shown that the results obtained are comparable.

13.1.2 Profile the spectrometer in accordance with the manufacturer's instructions (see 13.1).

13.1.3 Position or test the position of the spectrometer exit slits, secondary mirrors, if used, and photomultipliers to ensure that the peak radiation passes through each slit and illuminates the centers of the photomultipliers. This shall be done initially and as often as necessary thereafter to maintain proper alignment.

NOTE 12—Modern direct-reading spectrometers should show little drift in the response of channels with time. However, if at any time the gain adjustment of any channel drops below 0.5 or increases above 2, or if the background reading changes more than 0.5 to 2 $\times$ , that channel should be checked for alignment or deterioration of components. It is recommended that all gains and backgrounds be checked 1 month after installation and thereafter at least once a year as a formal inspection procedure.

13.2 *Electrical Parameters*—Various sets of electrical parameters in a rectified-capacitor discharge source produce somewhat similar high-frequency oscillatory unidirectional waveforms. These have been found to give comparable analytical performance. Use one of the sets of electrical parameters given in Table 2.

13.3 *Typical Exposure Conditions*—Exposure conditions will depend on the specific equipment, and a tradeoff that the individual laboratory selects between speed of analysis and slightly improved precision and accuracy with longer times. Typical time ranges are:

Flush period, s	2 to 7
Preburn period, s	2 to 10
Exposure period, s	5 to 20

13.4 *Gas Flow*—Gas flow rates will vary from laboratory to laboratory, but shall be in the following ranges:

Standby, L/min (standard ft <sup>3</sup> /h)	0.05 to 0.09 (0.1 to 0.2)
During exposure, L/min (standard ft <sup>3</sup> /h)	4.5 to 8 (9 to 15)

The compressed argon gas cylinder shall be replaced when the pressure falls to 7 kgf/cm<sup>2</sup> (100 psi). See Practice E 406 for precautions to be used in handling gases.

13.5 *Electrode System*—Position the flat, prepared surface of the specimen on the top of the excitation chamber so that the discharge will impinge on the area lying at least 5 mm from the edge of the specimen. The specimen shall seal the 8 to 15 mm opening in the top of the excitation chamber. The specimen serves as one electrode, the cathode. Mount the counter electrode opposite the specimen. Use only one type of counter