**Designation: E1251 – 11**

# Standard Test Method for
# Analysis of Aluminum and Aluminum Alloys by Spark Atomic Emission Spectrometry[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

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

## 1. Scope

1.1 This test method describes the analysis of aluminum and its alloys by atomic emission spectrometry. 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 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 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) 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.

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

## 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]

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.04 on Aluminum and Magnesium.

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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.

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 Spectrochemical Analysis
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)[3]
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

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

3.2.2 *global type calibration*—calibration curves determined using calibrants from many different alloys with considerable compositional differences.

3.2.3 *alloy type calibration*—calibration curves determined using calibrants from alloys with similar compositions.

3.2.4 *two point drift correction*—the practice of analyzing a high and low standardant for each calibration curve and adjusting the counts or voltage values obtained back to the values obtained on those particular standardants 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 (reference material) at or close to the nominal composition for the particular alloy being analyzed. For best results the standardant being used should be within ±10 % of the composition (for each respective element) of the material being analyzed.

## 4. Summary of Test Method

4.1 A unipolar triggered capacitor 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 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 in accordance with Practice E158.

4.2 Three different methods of calibration defined in 3.2.1, 3.2.2 and 3.2.3, 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. This approach is used when there is a need to analyze almost the entire range of aluminum alloys. Because binary calibrants may respond differently from alloy calibrants, 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 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 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 that have similar compositions. Again, specific alloy calibrants 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 Practice 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 Practice 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 ranges, background equivalent concentrations (BEC), 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 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.

TABLE 1 Recommended Analytical Lines

| Element | Wavelength in Air (nm)[A] | Recommended Concentration Range, % | Background Equivalent, %[B] | Calculated Detection Limit, %[C,D] | High Concentration Index, %[E] | Interferences Element, λ(nm) and k, %[F] | |
|---|---|---|---|---|---|---|---|
| Aluminum | I 256.799 | 70-100 | | | | | |
| | I 266.039 | 70-100 | | | | | |
| | I 237.208 | 70-100 | | | | | |
| Antimony | I 231.147 | 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 | |
| | | | | | | Mn 259.817 | 0.01 |
| Arsenic | 234.984 | 0.005-0.1 | | | | | |
| Beryllium | I 234.861 | 0.0001-0.05 | 0.001 | 0.00003 | | | |
| | II 313.042 | 0.0001-0.05 | 0.0035 | 0.00001 | | | |
| | 332.134 | 0.0001-0.05 | | 0.00001 | | | |
| Bismuth | I 306.772 | 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 |
| | | | | | | Mn 249.778 | 0.007 |
| | I 249.678 | 0.0001-0.05 | | | | | |
| | I 208.959 | 0.0001-0.05 | | | | Mo 208.952 | 0.13 |
| Cadmium | I 228.802 | 0.001-1 | 0.05 | <0.0001 | | As 228.812 | |
| | I 479.992 | 0.005-2 | 0.15 | 0.003 | | | |
| Calcium | II 393.367[G] | 0.001-0.05 | 0.001 | 0.00005 | | Fe 393.361 | |
| Chromium | I 425.435 | 0.001-1 | 0.015 | <0.0001 | | | |
| | II 267.716 | 0.001-1 | 0.004 | 0.0005* | | | |
| | II 276.654[G] | 0.005-1 | | | | | |
| Cobalt | I 345.351 | 0.0001-2 | | <0.0001 | | | |
| Copper | I 327.396 | 0.001-1.5 | 0.005 | <0.0001 | 0.7 | | |
| | I 324.754 | 0.001-0.5 | | | | | |
| | I 296.117 | 0.05-20 | 0.40 | 0.01* | >20 | Fe 296.128 | |
| | II 224.700 | 0.01-5 | 0.03 | 0.0005* | 5 | | |
| | I 510.554 | 0.05-20 | 0.32 | 0.01* | >20 | | |
| Gallium | I 294.364 | 0.001-0.05 | 0.015 | <0.0001 | | | |
| | I 417.206[G] | 0.001-0.05 | | | | Fe 417.213 | |
| | | | | | | Ti 417.190 | |
| | | | | | | Cr 417.167 | |
| Iron | II 238.204 | 0.001-1.5 | 0.015 | 0.0008 | 1.0 | | |
| | II 259.940 | 0.001-1.5 | 0.005 | 0.0004 | | | |
| | I 259.957 | | | | | | |
| | II 273.955 | 0.01-3.5 | | | | | |
| | I 374.949[G] | 0.001-3.5 | | 0.0001 | | | |
| | I 441.512 | 0.01-3.5 | | 0.0004 | | | |
| | I 438.355 | 0.005-3.5 | | | | | |
| Lead | I 405.782 | 0.002-0.7 | 0.04 | 0.0001 | | Mn 405.792 | 0.01 |
| | | | | | | Mg 405.763 | 0.001 |
| | I 283.306 | 0.002-0.7 | 0.07 | 0.002 | | | |
| Lithium | I 610.364 | 0.0001-3 | | | | | |
| | I 670.784 | 0.0001-0.02 | 0.0005 | <0.0005 | | | |
| | I 323.261 | 0.01-3 | | | | Fe 323.279 | |
| | | | | | | Sb 323.250 | |
| Magnesium | II 279.553 | 0.0005-0.3 | 0.0006 | 0.00003 | 0.04 | | |
| | I 285.213 | 0.0005-0.3 | 0.008 | <0.0001 | 0.25 | | |
| | I 277.669 | 0.05-11 | 0.08 | 0.01 | >11 | | |
| | I 383.231[G] | 0.01-11 | 0.015 | 0.002* | >11 | | |
| | I 383.826 | 0.1-11 | | | | | |
| | I 518.362 | 0.01-11 | 0.02 | 0.002* | >11 | | |
| Manganese | I 403.076[G] | 0.001-0.1 | 0.028 | 0.0001* | | | |
| | II 259.373 | 0.0005-0.5 | 0.004 | 0.00005 | 0.2 | | |
| | II 293.306 | 0.001-2 | 0.006 | 0.0002* | >1.1 | | |
| | II 346.033B | 0.01-2 | | | | | |
| Nickel | I 341.476 | 0.001-2 | 0.02 | <0.0001 | >2.5 | Zr 341.466 | 0.01 |
| | I 310.188 | 0.005-4 | 0.05 | 0.001* | >5 | | |
| | II 231.604 | 0.001-2 | 0.015 | 0.0005* | <2.5 | | |
| Phosphorus | I 178.231[H] | 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 | |
| | I 251.612 | 0.001-1.5 | 0.006 | 0.0001 | 1.5 | | |
| | I 390.553[G] | 0.05-24 | 0.25 | 0.01 | >24 | Cr 390.566 | 0.09 |
| | I 212.415 | 0.05-24 | 0.5 | 0.05 | >24 | | |
| Silver | I 328.068 | 0.0005-0.1 | | | | | |
| | I 338.289 | 0.0001-0.1 | | | >10 | | |
| | I 466.848 | 0.05-1.5 | | | | | |
| Sodium | I 588.995 | 0.0001-0.05 | 0.0015 | <0.0001 | | | |
| Strontium | II 421.552[G] | 0.0001-0.1 | 0.0004 | 0.0001 | | | |
| | I 460.733 | 0.0005-0.06 | | | | | |
| Tin | I 317.502 | 0.001-7.5 | 0.04 | 0.0001 | >10 | | |
| Titanium | II 334.904 | 0.0005-0.5 | 0.004 | <0.0001 | | | |
| | II 337.280 | 0.001-0.5 | 0.002 | <0.00010 | | | |
| | I 363.545 | 0.0005-0.05 | 0.030 | 0.003* | | | |

**TABLE 1** *Continued*

| Element | Wavelength in Air (nm)[A] | Recommended Concentration Range, % | Background Equivalent, %[B] | Calculated Detection Limit, %[C,D] | High Concentration Index, %[E] | Interferences Element, λ(nm) and k, %[F] |
|---|---|---|---|---|---|---|
| Vanadium | I 318.341 | 0.001-0.15 | 0.06 | 0.0003* | | |
| | I 437.924 | 0.001-0.25 | | | | |
| | II 310.230 | 0.001-0.15 | 0.014 | <0.0001 | | |
| Zinc | I 213.856 | 0.0005-0.1 | 0.035 | 0.0001* | 0.05 | |
| | I 334.502 | 0.001-10.0 | 0.065 | 0.0004 | >8 | |
| | I 481.053 | 0.01-10 | 0.07 | 0.001* | >10 | |
| | I 472.216 | 0.01-10 | 0.26 | 0.0015 | >10 | |
| Zirconium | II 339.198 | 0.001-1 | 0.02 | 0.001* | | |
| | II 349.621[G] | 0.001-1 | 0.006 | <0.0001 | | |

[A] I = atom line, II = ion line. Second (2nd) indicates that the second order shall be used, where available.

[B] *Background Equivalent Concentration (BEC)*—The concentration at which the signal due to the element is equal to the signal due to the background.

[C] 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) at levels *below* ten times the expected detection limit.

[D] See footnote C. For values marked with an asterisk (*) the available data were for a concentration greater than ten (10) times but less than a hundred (100) times the expected detection limit.

[E] *High Concentration Index*—The concentration at which the slope of the calibration curve drops below 0.75.

[F] *Interference Factor, k*—The apparent increase in the concentration of the element being determined, expressed in percent, due to the presence of 1.0 % of the interfering element.

[G] Useful analytical lines with improved signal to background ratios due to the complete removal of C-N background by the argon atmosphere.

[H] 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 microtorr 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.

## 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 Practice E716. Chill cast samples, poured and cast as described within Practice 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 Practice E716.

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

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,* 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.

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 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 this problem. The exhaust can then be vented into an efficient hood system.

**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 |

7.4 *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 composed of a two-stage type (high/low pressure) regulator of all-metal construction with two-pressure gages. Delivery tubing must not produce any contamination of the argon stream. Refrigerator grade copper tubing is recommended. The gages on the regulator will allow for the adjustment of the gas pressure to the instrument. Delivery pressure specifications will vary with instrument manufacturer. Please note that the delivery tube connections should be made with all metal seals and the delivery tubing itself should be kept as short as possible (Note 5). Argon supply shall be sufficient to support required flow during analysis and bleed during idle periods. All connections must be leak-free.

NOTE 5—All metal connections are strongly recommended 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*—For details on specifying the spectrometer please refer to Guide E1507.

7.6 *Measuring and Control System* of the instrument consists of either photomultiplier and integrating electronics or solid-state photosensitive arrays (CCD or CID) that convert observed light intensities to a digitizable signal. A dedicated computer and/or microprocessor is used to control burn conditions, source operation, data acquisition and the conversion of intensity data to concentrations. Data should be accessible to the operator throughout all steps of the calculation process. Concentration data may be automatically transferred to a site mainframe computer or server for further data storage and distribution. The instrument's control software should include functions for routine instrument drift correction (standardization), type standardization and the application of these functions to subsequent analyses.

## 8. Materials

8.1 *Counter-Electrode*—The counter-electrode and specimen surface are the two terminus points of the spark discharge. The counter electrode should be made from thoriated tungsten or silver and have a pointed end. The gap distance between the specimen surface and the tip of the counter electrode is specified by the manufacturer. The diameter and geometry of the counter electrode is also application and vendor dependent. If different designs and/or configurations are offered, it is recommended that the prospective purchaser test each design to determine which one performs the best for the intended analytical task. The counter electrode configuration and auxiliary gap distance must not be altered subsequent to spectrometer calibration or calibration adjustments. Electrode maintenance (frequent brushing of the counter electrode) to maintain its configuration, gap distance and minimize surface contamination are critical to accurate, precise analytical results. It is recommended that the instrument purchaser order several spare counter electrodes so that they can be replaced when necessary.

## 9. Reference Materials

9.1 *Calibrants*—All calibrants shall be homogeneous and free of cracks or porosity. These materials should also possess a metallurgical condition that is similar to the material(s) that are being analyzed. The calibrants shall be used to produce the analytical curves for the various elements being determined.

9.1.1 It is recommended that a calibration curve for any particular element be composed of a minimum of four calibrants. The concentrations of these calibrants should be fairly evenly spaced over the calibrated analytical range so that a mathematically valid calibration curve can be established using all of the points.

9.1.1.1 The calibrants used shall be of sufficient quality, purchased from a recognized reputable source, and have certified values to the required accuracy for the anticipated analytical tasks to be performed. A few SRM's are available from the National Institute of Standards and Technology (NIST). Also, there are other commercial sources for aluminum reference materials.

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

NOTE 6—Atomic emission analysis is a comparative technique that requires a close match of the metallurgy, structure and composition between the reference material and the test material. Differences in structure, such as result from the sodium modification of high silicon alloys, or differences in metallurgical history, due to extruding, rolling or heat treating induce a variety of effects that can influence the analytical results. To ensure analytical accuracy, care must be taken to match the characteristics of the reference material to that of the test material or suitable corrections to adjust for these influences must be established.

9.2 *Standardants:*

9.2.1 *Standardants for Drift Correction*—Both high and low concentration standardants are available from several commercial sources. The low standardant is usually high purity (smelter grade) aluminum. The high standardant(s) should have concentrations near or above the median concentration for the calibrated range of each spectral line. The commercially available standardants are tested for homogeneity and reproducibility of spectral response but are not necessarily certified for composition of individual elements. Composition certification is not required because these materials are only used to adjust intensity ratios back to those obtained during the initial calibration of the instrument. Care should be exercised when replacing depleted standardants with new ones that are from different heats or lots since the actual concentration of the individual element(s) may be different from the standardant currently in use. Whenever standardants are replaced, appropriate procedures must be followed to reference the intensities obtained from the new standardant to the intensities obtained from the standardant being replaced. See 14.3 for details.

9.2.2 *High Purity Standardants*—These shall be homogeneous and shall consist of aluminum with the lowest available concentration of the elements being determined. These materials are used to establish the background readings of the spectrometer for most elements. Their exact compositions need not be known.

9.2.3 *Blank Standardants*—These materials shall be homogeneous and of similar composition to the alloy type calibrants as described in 9.1 but will contain the lowest available concentrations of the trace elements being determined. They may be used if the lowest concentration of the element being determined is within ten times the detection limit of that element.

9.2.4 *Type Standardants*—Type standardants are certified reference materials that are traceable to a recognized certification agency such as NIST. These materials are certified for composition and homogeneity. In use, a type standardant usually provides a nominal concentration reference point which the instrument's computer software can use to calculate a slope and/or intercept correction to the observed readings to fine-tune the instrument's calculated response for each element of interest. This correction is then applied to each subsequent analysis. When using this approach it is assumed that the composition(s) of the unknown(s) will be essentially similar to the composition of the type standardant.

## 10. Hazards

10.1 The spark discharge presents a potential electrical shock hazard. The spark stand and/or the sample clamping device shall be provided with a safety interlock system to prevent energizing the electrode whenever contact can be made with the electrode. The instrument should be designed so access to the power supply is restricted by the use of safety interlocks.

10.2 Fumes of the fine metallic powder that are exhausted from the excitation chamber can be poisonous if the sample specimens contain significant levels of hazardous elements. Therefore, the instrument shall be designed with an internal exhaust system that is equipped with its own set of filters. Additionally, the instrument exhaust (after being filtered), may be vented directly to a non-hazardous location. To keep the instrument running properly, the filters should be cleaned and/or changed according to the manufacturer's recommendations.

## 11. Sampling

11.1 *Chill Cast Disks and Other Aluminum Forms*—For the techniques used to sample, melt and cast molten aluminum metal into a chill cast disk suitable for analysis, refer to Practice E716.

## 12. Preparation of Reference Materials and Specimen

12.1 *Preparation of Reference Materials*—All reference materials shall have their surfaces prepared for analysis according to Practice E716 with the cutting depth usually limited to that required to produce a fresh surface (about 0.010 in. or 250 µm). The surfaces of the reference materials and the surfaces of the specimens that are to be analyzed shall be prepared in the same manner. See Practice E716 for details.

12.2 *Preparation of Specimens*—For techniques on how to select and prepare for both chill cast samples and other forms of aluminum, such as sheet, plate, extrusions and castings refer to Practice E716.

NOTE 7—To achieve the best analytical results, both reference materials and sample specimen should have fresh surfaces. Surfaces that are clearly dirty, look "old" or oxidized, have porosity, inclusions or other foreign substances, or have been contaminated by repeated handling should not be used.

## 13. Preparation of Apparatus

13.1 Prepare the spectrometer for operation accordance with the manufacturer's instructions supplied with the instrument.

NOTE 8—It is not within the scope of this method to prescribe all of the details that are associated with the correct operation of any spectrometer. The reader is referred to the manufacturer's manual that is supplied with the instrument. Additionally, it is recommended that the purchaser of the spectrometer determine if training courses are offered at the manufacturer's facility. In many instances a manufacturer will offer specific spectrometer training courses several times yearly.

13.1.1 *Instrument Configuration*—Instruments are usually pre-configured for the analytical program (elements), concentration ranges and alloy families according to specifications that have been requested by the purchaser. Optionally, the purchaser may also choose to specify that the instrument come completely pre-calibrated for all alloys and all intended analytical tasks. The purchaser also has the option of completely configuring and calibrating the instrument. When this is done, great care must be exercised in the selection of the correct analytical conditions, analytical channels, internal standard channels, calibration ranges and calibrants to meet the specific analytical tasks. Whether the vendor or the end user calibrates an instrument, it is the responsibility of the end user to verify that the instrument is performing according to the specifications that have been set forth in the initial agreement or according to the performance as stated by the vendor. It is beyond the scope of this test method to describe the intricacies of complete instrument configuration. The user should consult the manufacturer's hardware and software manuals for specific configuration requirements.

13.1.2 *Profiling the Instrument*—Refer to Guide E1507 and profile the instrument according to the manufacturer's instructions. If the instrument is newly installed, it is recommended that the profile be checked several times during the first few weeks of operation to determine the stability of the unit. Record all profile settings in a logbook. Compare the differences in the settings to the tolerance variability allowed by the manufacturer.

13.1.3 *Checking Optical Alignment*—Position or test the position of the spectrometer exit slits, secondary mirrors (if used) or refractor plates (if used) and photomultipliers to ensure that the peak radiation passes through each slit and illuminates the centers of the phototubes. This shall be done by a trained expert initially and as often as necessary thereafter to assure proper alignment.

NOTE 9—Modern direct reading spectrometers should show little drift in the response 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 changes by more than 0.5 to 2×, that channel should be checked for alignment or deterioration of components.

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 produce comparable analytical performance. Refer to 7.2 for typical parameters.

13.3 *Exposure Conditions*—Exposure conditions vary with the manufacturer of the equipment. Conditions may have to be selected. A longer pre-spark and exposure may result in better precision and accuracy with less sample through-put while a shorter pre-spark and exposure will increase sample throughput but may decrease precision and accuracy. Typical time ranges are:

| | |
|---|---|
| Flush period, s | 2 to 7 |
| Pre-burn period, s | 2 to 20 |
| Exposure (spark) period, s | 2 to 10 |
| Exposure (arc) period, s | 2 to 10 |

13.4 *Gas Flow*—Argon flow rate requirements will vary from manufacturer to manufacturer and possibly from laboratory to laboratory. The following ranges are presented as a guide.

| | |
|---|---|
| Standby, L/min | 0.03 to 0.5 |
| During Exposure, L/m | 3.0 to 10 |

13.4.1 The high-pressure compressed gas cylinder should be changed when the pressure falls below 7 kg/cm$^2$ (100 kPa). If the gas is supplied from a cryogenic cylinder, caution should be exercised so that the cylinder is not allowed to "run dry." Consult with your local gas supplier to get their recommendation as to when a cryogenic tank should be changed. See Practice E406 for precautions to be used when handling gases.

13.5 *Electrode System*—The sample specimen serves as one electrode, the cathode. The thoriated tungsten or other suitable electrode serves as the counter electrode. Since the discharge is essentially unidirectional, the counter electrode is not attacked and therefore can be used for many burns. Because the electrode is semi-permanent, continual gapping is not required. It is recommended that the gap of the electrode be checked periodically. The gapping frequency is dependent on the number of burns. Consult with the manufacturer to determine the optimum gapping frequency for each instrument type. However, material ablated from the sample surface tends to build up on the tip of some types of electrodes. This buildup will change the gap and may adversely affect results. The counter electrode therefore should be cleaned (brushed) with a wire brush that is normally supplied with the instrument. For best performance it is strongly recommended that the counter electrode be cleaned after every burn. Also, with continued use the shape of the electrode may change due to this buildup of material. Frequent close inspection of the electrode is recommended. Some instruments use pin type electrodes that are not affected by a buildup of ablated material. Pin electrodes should not be cleaned with a brush. Pin electrodes tend to erode faster and should be gapped and/or replaced more frequently.

13.6 *Reference Material / Sample Placement*—Reference materials and samples should be placed on the spark stand so that the hole in the top plate is completely covered. Completely covering the hole will prevent air leaks into the discharge area. Air can cause "bad" burns and adversely affect precision and accuracy. The hole should be covered during idle periods for the same reason. Samples and reference materials should be sparked approximately 7 to 10 mm from their outer edge. This can be best accomplished by placing them so that the outer edge of the machined surface just covers the hole in the top plate. Overlapping the burns may adversely affect precision and accuracy.

NOTE 10—It is essential that operators learn the difference between a "good" burn and a "bad" burn. Bad burns can be caused by an air leak between the sample and the top plate, a poor quality sample, poor quality argon and various other reasons. A "good" burn will have a deeply pitted area in the center surrounded by a blackish ring. The actual appearance of a burn will vary with source conditions and alloy. A "bad" burn will tend to have shallow pits surrounded by a white or silver colored ring. Usually the intensity of the aluminum internal standard channel for a "bad" burn will be considerably lower than a good burn. All "bad" burns should be rejected and replaced.

13.7 *Warm-up*—After any prolonged interval of instrument non-use, several warm-up burns should be taken. In most cases two to four burns are sufficient to check for proper gas flow and consistency of results.

## 14. Drift Correction

14.1 *Need for Drift Correction*—Atomic emission spectrometric analyses depend upon relative measurements that are subject to drift over time. To correct for drift, a suite of reference materials that include both high and low concentrations of the elements is used to standardize the readout whenever a correction is required. Failure to routinely correct for instrument drift will adversely affect analysis results. The frequency for drift correction should be determined by statistical process control methods based on periodic measurement of a control sample.

14.2 *Drift Correction*—Select a suite of drift correction standardants that will cover the analytical array and anticipated element concentration ranges of the instrument to be drift corrected. It is highly recommended that the purchaser of a new instrument specify that the appropriate drift correction standards be included with the purchase of the spectrometer. If the instrument comes pre-calibrated, then these materials should automatically be included with the instrument. It is the responsibility of the purchaser to make sure that the correct standardants are included with the instrument. Follow the manufacturer's instructions when drift correcting the instrument. The spectrometer's software should have a program that will guide the operator through the drift correction process. If the instrument is newly installed, give the unit sufficient time to stabilize in its new environment before proceeding with a drift correction. It is recommended that the spectrometer be allowed to stabilize under vacuum (if so equipped) and to rest in its final controlled environment surroundings for at least two days before a drift correction is performed. Remember, the instrument must be profiled before being drifted corrected. Refer to Practice E1329 for further details.

14.3 *Number of Burns*—It is recommended that four burns be taken on each of the standardants during the drift correction process.

14.4 *Checking Homogeneity of Candidate Standardants*—If the homogeneity of the standardant(s) being used is questionable; the material(s) can be tested for homogeneity. To determine the material's homogeneity follow instructions as given in Method E826.

14.5 *Recording the Drift Correction Readings:*

14.5.1 Instruments that come pre-calibrated will have the initial drift corrected response factors entered into the instrument's computer memory.

14.5.2 If the instrument does not come pre-calibrated, then follow the instructions of the manufacturer regarding establishing the initial drift correction responses/factors. Initial drift correction responses should be established immediately after calibration.

14.5.3 If one of the drift correction materials must be replaced because it has become unusable (too thin), follow the instructions as set-forth in the instrument's manual regarding the replacement and recording of the new standardant's responses. Failure to properly replace drift correction standards may adversely affect analysis accuracy.

## 15. Calibration

15.1 *Obtaining Calibration Data*—The following procedure is designed to allow the analyst to collect accurate data for the purpose of generating analytical calibration curves. For details on establishing and controlling spectrochemical analytical curves, refer to Practice E305. Any recently installed, laboratory grade spectrometer should show minimal drift over an 8 to 24 h time period when placed in a laboratory with a tightly controlled environment.

15.1.1 Select the reference materials that are to be used as the calibrants.

15.1.2 Follow the manufacturer's operating manual and use the instrument's software to design, and name the analytical program that you wish to create. Using the software, enter the identities of the selected calibrants and their associated concentrations for the elements you wish to include in this calibration.

15.1.3 Before starting the collection of calibration data, thoroughly clean the excitation chamber and gap or replace the electrode as needed. Prepare fresh surfaces on the selected calibrants.

15.1.4 Profile the instrument.

15.1.5 Burn the calibrants and collect the data.

15.2 Refer to Practice E305 and calibrate the instrument using the instrument's software following the instructions in the manufacturer's manual. Use the appropriate program that allows for the calculation of the calibration curves. Care should be taken when using 3rd and 4th order regressions that enough standards are available to adequately cover the entire range.

15.3 *Verifying the Accuracy of Calibration*—After completing a calibration re-burn several of the calibrants as unknowns and compare the measured concentrations for each element with the certified values. Check for clerical errors, elemental interferences or biases if results do not compare favorably.

15.3.1 If individual calibrants give consistently high readings for an element, check for possible interferences from other elements. Manually calculate or, using the instrument's software, have the software calculate, the appropriate factors for the interference(s). If the factors are calculated manually, see Practice E158 for details.

## 16. Procedure for Analyzing Specimens

16.1 *Excitation*—Burn the specimens in accordance with the conditions given in 13.2, 13.3, 13.4, and 13.5.

16.2 *Replicate Burns*—Burn the specimens from one to eight times, depending on the complexity of the alloy, specimen homogeneity, and the level of confidence required. A single burn is frequently employed for qualitative analysis of primary aluminum to detect major changes in the composition of aluminum from individual Hall cells to assess performance. A single burn shall not be used for determining compliance with composition specifications. Two to four burns are recommended for most alloys where homogeneity is fair and accuracy becomes important. In very complex alloys or in alloy systems that are noted for their segregation additional burns may be required.

16.2.1 The determinations from all burns should be averaged unless a burn produces a very abnormal internal standard count or appears visually to be bad (see 13.5, Note 10). When a burn is rejected, it should be replaced in order to maintain the normal number of burns to be averaged.

## 17. Calculation of Results

17.1 After performing the test material analyses, print out the concentration data directly. Further display or manipulation of the data should not be necessary.

## 18. Data Reporting

18.1 *Number of Significant Figures*—The composition of alloys shall not be reported with more significant figures or higher precision than that of the calibrants used to calibrate the spectrometer.

## 19. Precision and Bias[4]

19.1 *Precision*—Eight laboratories cooperated in the interlaboratory study using either the binary calibration approach or the alloy-type calibration technique. Since an attempt was made to include all general alloy types, not all laboratories could analyze all materials or all concentration ranges. Testing was done in accordance with Practice E691. A summary of the inter-laboratory test is shown in Table 3.

19.2 *Bias*—There is no evidence of bias since all acceptable individual test results are within one R of the assumed concentrations.

## 20. Keywords

20.1 aluminum; aluminum alloys; Spark Atomic Emission Spectrometry (Spark-AES)

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[4] Supporting data for strontium have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1113.

**TABLE 3 Summary of Interlaboratory Tests**

| Test Sample | Concentration | | $S_T$ | $S_R$ | R | 2R |
|---|---|---|---|---|---|---|
| | Certified | Observed | | | | |
| Antimony | | | | | | |
| WA1199 | 0.0013 | 0.0012 | 0.00041 | 0.00041 | 0.0011 | 0.0022 |
| SS1075 | 0.0033 | 0.0036 | 0.00024 | 0.00024 | 0.00067 | 0.0013 |
| Arsenic | | | | | | |
| L1 | 0.0014 | 0.0012 | 0.00021 | 0.00021 | 0.00059 | 0.0012 |
| L5 | 0.0064 | 0.0055 | 0.00083 | 0.00083 | 0.0023 | 0.0046 |
| Beryllium | | | | | | |
| 3004-3 | 0.0004(4) | 0.00046 | 0.000010 | 0.000040 | 0.00011 | 0.0002 |
| 5056 | 0.004 | 0.0037 | 0.000011 | 0.00037 | 0.0010 | 0.002 |
| 358 | 0.24 | 0.237 | 0.0021 | 0.0034 | 0.0095 | 0.02 |
| Bismuth | | | | | | |
| 1000 | 0.034 | 0.0335 | 0.0005 | 0.0010 | 0.0029 | 0.006 |
| 2011 | 0.56 | 0.567 | 0.0048 | 0.021 | 0.058 | 0.12 |
| Boron | | | | | | |
| 1075 | 0.0006 | 0.00055 | 0.000024 | 0.00006 | 0.00016 | 0.0003 |
| 18 | 0.009 | 0.0107 | 0.00026 | 0.0012 | 0.0033 | 0.007 |
| Calcium | | | | | | |
| 3004-3 | 0.0002(5) | 0.00027 | 0.000012 | 0.000072 | 0.0002 | 0.0004[A] |
| Chromium | | | | | | |
| 3003 | 0.001 | 0.00146 | 0.00009 | 0.0023 | 0.0064 | 0.013 |
| 1000 | 0.036 | 0.0347 | 0.00025 | 0.0033 | 0.0092 | 0.018 |
| 5056 | 0.12 | 0.1208 | 0.00093 | 0.0050 | 0.0140 | 0.028 |
| 7075 | 0.23 | 0.2264 | 0.0036 | 0.0067 | 0.0189 | 0.038 |
| Cobalt | | | | | | |
| 7091 | 0.44 | 0.4423 | 0.0054 | 0.0054 | 0.015 | 0.030 |
| Copper | | | | | | |
| 1075 | 0.001 | 0.00084 | 0.00014 | 0.00061 | 0.0017 | 0.0034 |
| 1000 | 0.030 | 0.0306 | 0.00027 | 0.00123 | 0.0035 | 0.007 |
| 3003 | 0.15 | 0.154 | 0.00074 | 0.00435 | 0.0122 | 0.024 |
| 7075 | 1.58 | 1.564 | 0.0179 | 0.0388 | 0.109 | 0.22 |
| 2011 | 5.49 | 5.492 | 0.0231 | 0.0538 | 0.151 | 0.30 |
| Gallium | | | | | | |
| 1075 | 0.022 | 0.0219 | 0.00030 | 0.00053 | 0.0015 | 0.0030 |
| Iron | | | | | | |
| 5056 | 0.20 | 0.195 | 0.00532 | 0.01655 | 0.046 | 0.09 |
| 2011 | 0.53 | 0.530 | 0.00264 | 0.01275 | 0.036 | 0.07 |
| Lead | | | | | | |
| 1000 | 0.036 | 0.035 | 0.0003 | 0.0009 | 0.003 | 0.005 |
| 2011 | 0.56 | 0.580 | 0.0060 | 0.0461 | 0.129 | 0.26 |
| Lithium | | | | | | |
| 3004-3 | 0.0003(4) | 0.00034 | 0.000006 | 0.000025 | 0.00007 | 0.00014 |
| 2090 | 2.14 | 2.13 | 0.023 | 0.041 | 0.114 | 0.23 |
| Magnesium | | | | | | |
| 1000 | 0.030 | 0.0317 | 0.00044 | 0.00158 | 0.0044 | 0.009 |
| 356 | 0.36 | 0.354 | 0.00426 | 0.00860 | 0.024 | 0.05 |
| 7075 | 2.61 | 2.596 | 0.0219 | 0.0283 | 0.079 | 0.16 |
| 5056 | 5.36 | 5.364 | 0.0400 | 0.0441 | 0.123 | 0.25 |
| Manganese | | | | | | |
| 1075 | 0.001 | 0.00102 | 0.00008 | 0.00024 | 0.001 | 0.013[A] |
| 1000 | 0.032 | 0.0316 | 0.00022 | 0.00131 | 0.0037 | 0.007 |
| 5056 | 0.10 | 0.1006 | 0.00044 | 0.0039 | 0.0109 | 0.022 |
| 3003 | 1.21 | 1.208 | 0.00850 | 0.0139 | 0.039 | 0.08 |
| Nickel | | | | | | |
| 3003 | 0.005 | 0.00474 | 0.00007 | 0.00028 | 0.0008 | 0.0016 |
| 1000 | 0.031 | 0.0299 | 0.00028 | 0.00177 | 0.0050 | 0.0099 |
| 850 | 1.21 | 1.219 | 0.0044 | 0.0082 | 0.023 | 0.048 |
| 336 | 2.60 | 2.596 | 0.0216 | 0.0247 | 0.069 | 0.139 |
| Phosphorus | | | | | | |
| AP-4 | 0.003 | 0.00269 | 0.00016 | 0.00028 | 0.0008 | 0.0016 |
| Silicon | | | | | | |
| 1075 | 0.068 | 0.0699 | 0.0004 | 0.00266 | 0.0074 | 0.015 |
| 2011 | 0.28 | 0.288 | 0.00194 | 0.00784 | 0.022 | 0.044 |
| 356 | 7.18 | 7.16 | 0.0414 | 0.0811 | 0.23 | 0.45 |
| 390A | 16.36 | 16.29 | 0.13 | 0.242 | 0.68 | 1.35 |
| Sodium | | | | | | |
| 3004-3 | 0.0002(9) | 0.00026 | 0.000011 | 0.000017 | 0.00005 | 0.00010 |
| 18 | 0.021 | 0.024 | 0.0016 | 0.0036 | 0.010 | 0.020 |
| Strontium[B] | | | | | | |
| 336 | 0.027 | 0.0271 | 0.0005 | 0.0008 | 0.00229 | 0.00458 |
| 356 | 0.028 | 0.0277 | 0.0003 | 0.0016 | 0.00435 | 0.00870 |
| Tin | | | | | | |
| 1000 | 0.028 | 0.0287 | 0.0002 | 0.0011 | 0.003 | 0.006 |
| Titanium | | | | | | |
| 1075 | 0.001 | 0.00083 | 0.000031 | 0.000341 | 0.0010 | 0.0019[A] |

**TABLE 3** *Continued*

| Test Sample | Concentration | | $S_T$ | $S_R$ | R | 2R |
|---|---|---|---|---|---|---|
| | Certified | Observed | | | | |
| 2011 | 0.003 | 0.00321 | 0.000041 | 0.000185 | 0.0005 | 0.0010[A] |
| 1000 | 0.031 | 0.0318 | 0.000161 | 0.000799 | 0.0022 | 0.0045 |
| 356 | 0.12 | 0.1176 | 0.00058 | 0.00353 | 0.010 | 0.020 |
| Vanadium | | | | | | |
| 1075 | 0.002 | 0.00195 | 0.000055 | 0.00016 | 0.0005 | 0.0009 |
| 1000 | 0.022 | 0.0218 | 0.000237 | 0.00058 | 0.0016 | 0.0033 |
| Zinc | | | | | | |
| 1075 | 0.002 | 0.00229 | 0.00040 | 0.00069 | 0.0019 | 0.004 |
| 1000 | 0.030 | 0.0302 | 0.00065 | 0.00071 | 0.0020 | 0.004 |
| 356 | 0.01 | 0.1001 | 0.00059 | 0.00111 | 0.0031 | 0.006 |
| 7075 | 5.74 | 5.741 | 0.0285 | 0.0927 | 0.26 | 0.52 |
| Zirconium | | | | | | |
| 1075 | 0.001 | 0.00093 | 0.00007 | 0.00017 | 0.0005 | 0.0009 |
| 2090 | 0.12 | 0.120 | 0.012 | 0.0023 | 0.006 | 0.013 |

[A] Values are below minimum quantifiable limit calculated based on the data in this ILS.

[B] Supporting data for strontium have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1113.

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