



Designation: 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 (ε) 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 spark-atomic emission 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 Mass Fraction Range<br>(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 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.

<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.

Current edition approved Oct. 1, 2017. Published October 2017. Originally approved in 1988. Last previous edition approved in 2017 as E1251 – 17. DOI: 10.1520/E1251-17A.

Analysis of Hg in aluminum by 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 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, 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

<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.

- 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
- 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 calibration materials from alloys with similar compositions.

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

3.2.3 *global-type calibration*—calibration curves determined using 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 standardization materials for each calibration curve and adjusting the intensities obtained back to the values obtained on those particular 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 standard-

ization materials at or close to the nominal composition for the particular alloy being analyzed. For best results, the 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 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 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 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 calibration materials. This approach is used when there is a need to analyze almost the entire range of aluminum alloys. Because binary calibration materials may respond differently from alloy 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 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 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 calibration materials that have similar compositions. Again, specific alloy 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 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, which may be removed

<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>.

(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 mass fraction ranges, background equivalent 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 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  $\mu\text{m}$  and exit slit widths of 50  $\mu\text{m}$ .

## 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 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*—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 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 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 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 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 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. (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.) Argon supply shall be sufficient to support required flow during analysis and bleed during idle periods. All connections must be leak-free.

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 mass fractions. Data should be accessible to the operator throughout all steps of the calculation process. 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 discharge.