Designation: E3061 − 17

Standard Test Method for Analysis of Aluminum and Aluminum Alloys by Inductively Coupled Plasma Atomic Emission Spectrometry (Performance Based Method)[1]

This standard is issued under the fixed designation E3061; 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.

1. Scope

1.1 This test method describes the inductively coupled plasma atomic emission spectrometric analysis of aluminum and aluminum alloys for the following elements:

Elements	Application Range, % Minimum	Application Range, % Maximum
Si	0.02	16.8
Fe	0.02	3.06
Cu	0.005	7.0
Mn	0.003	1.41
Mg	0.006	8.2
Cr	0.004	0.52
Ni	0.004	2.71
Zn	0.02	9.65
Ti	0.009	0.20
Ag	0.003	0.4
As	0.005	0.012
B	0.009	0.027
Ba	0.002	0.03
Be	0.002	0.11
Bi	0.01	0.59
Ca	0.003	0.048
Cd	0.002	0.055
Co	0.002	0.034
Ga	0.01	0.019
Li	0.001	2.48
Mo	0.02	0.15
Na	0.008	0.026
P	0.01	0.025
Pb	0.009	0.51
Sb	0.01	0.28
Sc	0.01	0.065
Sn	0.008	6.28
Sr	0.0008	0.028
Ti	0.005	0.20
Tl	0.009	0.13
V	0.01	0.12
Zr	0.004	0.25

1.2 This test method has only been interlaboratory tested for the elements and ranges specified. It may be possible to extend this test method to other elements or different composition ranges if method validation, which includes evaluation of method sensitivity and precision and bias (as described in Section 14), is performed. Additionally, the validation study must evaluate the acceptability of sample preparation methodology using reference materials and/or spike recoveries. The user should carefully evaluate the validation data against the laboratory's data quality objectives. Method validation of scope extensions is also a requirement of ISO/IEC 17025.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.* Safety hazard statements are given in Section 10 and specific warning statements are given in Sections 15, 17, 18, 19, 20 and 21.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B985 Practice for Sampling Aluminum Ingots, Billets, Castings and Finished or Semi-Finished Wrought Aluminum Products for Compositional Analysis
D1193 Specification for Reagent Water
E34 Test Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys
E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E406 Practice for Using Controlled Atmospheres in Spectrochemical Analysis
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

[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.

E716 Practices for Sampling and Sample Preparation of Aluminum and Aluminum Alloys for Determination of Chemical Composition by Spark Atomic Emission Spectrometry
E1329 Practice for Verification and Use of Control Charts in Spectrochemical Analysis
E1452 Practice for Preparation of Calibration Solutions for Spectrophotometric and for Spectroscopic Atomic Analysis (Withdrawn 2005)[3]
E1479 Practice for Describing and Specifying Inductively Coupled Plasma Atomic Emission Spectrometers
E2857 Guide for Validating Analytical Methods

2.2 *ISO Standards*[4]

ISO/IEC 17025 General Requirements for the Competence of Calibration and Testing Laboratories
ISO Guide 98-3 Uncertainty of Measurement Part 3: Guide to the Expression of Uncertainty in Measurement (GUM:1995) - First Edition

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E135.

4. Summary of Test Method

4.1 The test specimen, in the form of drillings, chips, millings, turnings, small pieces or powder, is dissolved in a caustic solution or a mixture of dilute mineral acids and hydrogen peroxide or sodium nitrite and the resulting solutions are measured using inductively coupled plasma atomic emission spectrometry. The spectrometer is calibrated using calibration solutions prepared to match the sample matrix, using a pure aluminum stock solution prepared in 15.2 and stock solutions traceable to an SI unit through a national metrology laboratory or stock solutions prepared as directed in Practice E1452.

5. Significance and Use

5.1 This test method for the analysis of aluminum and aluminum alloys is primarily intended to test material for compliance with The Aluminum Association Inc.[5] registered composition limits or other specified composition limits for aluminum and aluminum alloys.

5.2 It is assumed that all who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely, and that the work will be performed in a properly equipped laboratory.

5.3 This is a performance-based test method that relies more on the demonstrated quality of the test result than on strict adherence to specific procedural steps. It is expected that laboratories using this test method will prepare their own work instructions. These work instructions should include detailed operating instructions for the specific laboratory, the specific reference materials employed, and performance acceptance criteria.

6. Interferences

6.1 The effect of potential spectral overlap interferences and background will vary based on the wavelengths selected, instrument design, and may vary from instrument to instrument of the same design. Variation of excitation conditions or operating parameters may enhance or minimize these interferences. For these reasons, the effect of the potential interferences must be thoroughly investigated for each element and matrix on the instrument chosen for analysis. Practice E1479 describes the typical physical and spectral interferences encountered during the inductively coupled plasma spectrometric analysis of metal alloys. Potential spectral interferences for recommended wavelengths are given in Table 1. The user is responsible for ensuring the absence of, or for compensating for, interferences that may bias test results obtained using their particular spectrometer.

6.2 The use of an internal standard may compensate for the physical interferences resulting from differences between sample and calibration solutions transport efficiencies.

6.3 Shifts in background intensity levels because of, for example, recombination effects or molecular band contributions, or both, may be corrected by the use of an appropriate background correction technique. Direct spectral overlaps are best addressed by selecting alternative wavelengths. If alternate wavelengths are not available, spectral interference studies should be conducted on all new matrices to determine the interference correction factor(s) that must be applied to compositions obtained from certain spectral line intensities to minimize biases. Some instrument manufacturers offer software options that mathematically correct for direct spectral overlaps, but the user should carefully evaluate this approach to spectral correction.

6.4 Modern ICP spectrometers typically have software that allows comparison of a sample spectrum to the spectrum obtained from a blank solution. The user of this test method must examine this information to ascertain the need for background correction and the correct placement of background points.

6.5 Table 1 recommends wavelengths from the NIST Atomic Spectra Database[6] that may be used for the analysis of aluminum and aluminum alloys. In this database, wavelengths of less than 200 nm were measured in vacuum and wavelengths greater than or equal to 200 nm were measured in air. Software tables for individual instruments may list wavelengths somewhat differently, as instrument optical path atmospheric conditions may vary.

6.6 Information on potential spectral interfering elements typically found in aluminum alloys was provided by some of the laboratories participating in the interlaboratory study and

[3] The last approved version of this historical standard is referenced on www.astm.org.
[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St. 4th Floor, New York, NY 10036, http//www.ansi.org
[5] Available from The Aluminum Association Inc., 1400 Crystal Drive, Arlington, VA 22202, http://www.aluminum.org/
[6] Available from The National Institute of Standards and Technology 100 Bureau Dr., Gaithersburg, MD 20899 https://www.nist.gov/

TABLE 1 Analytical Lines and Possible Interferences

Element	Wavelength, nm	Possible Interferences
Antimony	206.833	W, Fe, Ni, Be
	259.805	Fe
Arsenic	189.042	Cr
	193.759	Zr
	197.262	Pb
Barium	455.403	Zr
	493.409	
Beryllium	234.861	Fe, Zr
	313.042	Ti, V
	313.107	Ti
Bismuth	222.825	Cr, Cu, Ti
	223.061	Cu, Ni, Ti
	306.772	
Boron	208.959	Sn, Fe
	249.678	Sn, Fe, Ni, Ca
	249.773	Ni, V
Cadmium	226.502	Co, Ni
	228.802	As
Calcium	315.887	Cr, Zr
	317.933	W
	393.366	Zr
Chromium	205.552	Be, Cu, Ni
	267.716	
	283.563	
	357.869	Zr
Cobalt	228.616	Mo, Ni, Fe
	238.892	Fe, Mo
Copper	221.458	Cr
	221.810	Si
	223.008	Bi, Mn, Ti, V
	224.700	Ni
	324.754	
	327.396	
Gallium	294.364	Fe, Ti, Cr
	417.206	Ni, Fe, Co
Indium	410.172	Cr, Ti
	451.131	Mo
Iron	238.204	V, Zr
	239.562	
	259.837	
	259.940	
Lead	182.203	
	220.353	Bi
	283.306	Cr
Lithium	670.784	Co, Mo, Fe
Magnesium	257.610	Mn, Ti
	259.373	Mn
	260.569	Mn, Ti, V
	293.306	Fe, Zr
	293.930	Zr
Manganese	257.610	
	259.373	
	260.569	Ti
	293.306	Cr
	293.930	
Molybdenum	202.030	Ni, Co, Mn
	277.540	Al