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	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

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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.

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

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[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

TABLE 1 *Continued*

Element	Wavelength, nm	Possible Interferences
Nickel	221.647	
	231.604	
	232.003	
	239.452	
Phosphorus	177.499	Cu, Be
	178.287	Al
Potassium	404.721	Ba
	766.490	Mo, Cr
Scandium	361.384	Mo, Zr, Cr
	363.075	Ca, Fe
Silicon	212.412	V
	250.690	V
	251.612	V, Zn
	288.158	Cr, Zr
Silver	328.068	Mn
	338.289	Cr, Sb
Sodium	330.237	
	589.592	Cr, Zn
Strontium	407.771	Fe
	421.552	Cr, Cu
Tin	189.989	Ti
	242.949	Fe
Thallium	276.787	
	190.896	
Titanium	323.452	Ni, Zr
	323.657	Mn, Zr
	334.904	
	334.941	
	336.121	Ni
	337.280	Zr
Vanadium	290.646	Ti
	290.882	Cr
	292.402	Cr
	310.230	Ni
	311.838	Cr, Ti
Zinc	202.548	Cr, Cu, Mg, Ni
	206.200	Bi, Cr, Ti
	213.856	Cu, Ni, Ti, V
	472.216	Bi
	481.053	
Zirconium	327.305	
	339.198	
	343.823	
	349.621	Mn

may have originated from sources such as recognized wavelength reference tables, instrument manufacturer's software wavelength tables, an individual laboratory's wavelength research studies, or a combination thereof.

6.7 The user must verify that the selected wavelength performs acceptably in their laboratory, preferably during method validation (see Section 23). Total dissolved solids, pH, and viscosity should be similar between sample solutions and calibration solutions. The user also may choose to use multiple wavelengths to help verify that line selection is optimized for the particular alloy being analyzed. It is recommended that when wavelengths and appropriate spectral corrections are determined, the user of this test method should specify this information or reference instrument programs that include this information in their laboratory analysis procedures.

7. Apparatus

7.1 *Inductively Coupled Plasma Atomic Emission Spectrometers*—Refer to Practice E1479 for attributes to consider when selecting an appropriate instrument.

8. Reagents and Materials

8.1 *Reagents:*

8.1.1 *Purity and Composition of Reagents*[7]—The purity and composition of chemical reagents shall conform to the requirements prescribed in Practices E50. Reagent grade chemicals or better shall be used in all tests.

8.1.2 *Alcohol,* ethanol or methanol.

8.1.3 *Boric Acid* (H_3BO_3).

8.1.4 *Hydrogen Peroxide* (H_2O_2), 30 %.

8.1.5 *10.5 N Sodium Hydroxide (NaOH) Solution.*

8.1.6 *4 % Sodium Nitrite* ($NaNO_2$) *Solution.*

8.1.7 Metals of the highest purity available and having known impurity content should be used if preparing stock solutions as directed in Practice E1452.

8.1.8 *Purity of Water*—References to water shall be understood to mean reagent water, Type II grade, as defined by Specification D1193. The water purification method used must be capable of removal of all elements that might bias the test results.

8.1.9 *Argon,* of 99.998 % purity, has been found satisfactory. For information on gas handling, refer to Practice E406.

8.1.10 *Stock Solutions,* if needed, shall be prepared as directed in Practice E1452.

8.1.11 *Certified Reference Materials (CRMs),* in chip form are available from some national metrology organizations and commercial sources.

8.1.12 *Single Element Certified Reference Material Solutions*—are available from some national metrology organizations and commercial sources.

8.1.13 *Aluminum Metal (Al),* for matrix matching calibration solutions. Aluminum that is at least 99.999 % is recommended. Aluminum that is less pure may be used provided the impurities are not present at levels that affect the measured amount for elements of interest or the internal standard if used.

8.2 *Internal Standard*—The use of an internal standard is not required but is recommended. The use of an internal standard may compensate for the physical interferences resulting from differences in sample and calibration solutions transport efficiency. Lanthanum, Co, Sc, Be, and Y were used by participants in the Interlaboratory Study (ILS). Wavelengths used and potential interferences are given in Table 2. It is important that the element chosen for an internal standard is not present in the samples at a level that will affect the analysis.

9. Control Materials

9.1 A laboratory may procure or produce a chip material with a composition that is similar to the samples for use as a control material. These chips should have low heterogeneity and be well blended. Users of this test method may also use certified reference materials as control materials.

9.2 A laboratory may find it difficult to procure or produce the materials for all of the necessary analytes or alloys. Here, it is acceptable to prepare equivalent reference material solutions using the procedure described in Section 15 for use as control solutions.

10. Hazards

10.1 This test method involves the use of concentrated mineral acids. Read and follow label precautions carefully before using. **Warning—**This method involves the use of HF. HF is extremely dangerous. Read and follow label precautions, SDS information, and Practices E50 for HF handling. For precautions to be observed in the use of certain other reagents in this test method, refer to Practices E50.

11. Sampling

11.1 Refer to Practices B985, E34, and E716 for procedures to sample aluminum and aluminum alloys that provide a representative sample.

12. Sampling, Test Specimens, and Test Units

12.1 Test specimens should be obtained by milling or drilling to obtain drillings, chips, millings, or turnings that are clean and of sufficient quantity to generate test specimens of at least 0.1 g for dissolution and analysis. Powdered aluminum can typically be used as-is.

NOTE 1—Antimony, arsenic, bismuth, and phosphorus may be volatilized during the process of obtaining drillings, chips, millings, or turnings

TABLE 2 Wavelengths Used and Potential Interferences

Internal Standard Element	Wavelength, nm	Potential Interference
Beryllium	234.861	Fe, Zr
	313.042	Ti, V
	313.107	Ti
	228.616	Mo, Ni, Fe
Cobalt	238.892	Fe, Mo
Lanthanum	379.477	Fe
Scandium	361.384	Mo, Zr, Cr
Yttrium	363.075	Ca, Fe
	371.029	Ti

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[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

if the metal overheats. Lubricating the metal with methanol or ethanol during machining is recommended. Pin samples or small pieces may also be used to prevent the loss of volatile elements.

13. Preparation of Apparatus

13.1 Analytical instrumentation and sample preparation equipment shall be installed and operated as directed by the manufacturer.

13.2 Using the manufacturer's ICP-AES instrument operation software, conduct start-up procedures and adjustments to the analytical instrument as directed in the manufacturer's operation instructions. Prepare a method or program appropriate to calibrate and measure the elements of interest for the selected wavelengths. Optimize the ICP-AES instrument parameters. Profile the spectrometer as specified in the manufacturers' instructions to ensure optical alignment.

14. Sensitivity and Precision

14.1 Prior to calibration, establish that the instrument being used is capable of demonstrating acceptable sensitivity and precision for the elements being determined. Once it has been demonstrated that the instrument has acceptable sensitivity and precision for these elements, it is not necessary to routinely evaluate sensitivity and precision. Evaluate equipment sensitivity and precision as described in 14.2 and 14.3 .

14.2 *Sensitivity*—Sensitivity shall be evaluated by establishing two-point calibrations for each element being determined using the blank and a high calibration solution prepared as described in Section 15. After thorough rinsing, the blank solution is measured 10 times. Calculate 3 times the standard deviation of these measurements to approximate the limit of detection. Calculate 10 times the standard deviation to approximate the limit of quantification. If the instrument/parameter selection of the user does not produce an estimated limit of detection equal to or better than the lower scope limit of the method for the element(s) being determined, then it is probable the method user will be unable to meet the method's lower scope limit. If the instrument/parameter selection of the user does not produce a limit of quantification equal to or better than the lower scope limit of the method for the element(s) being determined, then it is possible the method user will be unable to consistently meet the method's lower scope limit.

14.3 *Precision*—The short-term precision shall be determined as follows. Using the two- point calibration generated in 14.2, measure the high calibration solution 10 times using the instrument/parameters selected by the method user. Calculate the % Relative Standard Deviation (% RSD) as follows:

$$\% \, RSD = 100 \, s/\bar{C} \qquad (1)$$

where:

s = estimated standard deviation of the 10 measurements, and

$\bar{C}$ = average of the 10 results for the measured composition.

The calculated % RSD should be approximately 1 %. However, as compositions decrease or as intensities approach detector saturation, % RSD may tend to increase, while not necessarily affecting the quality of the reported result. During the interlaboratory study, % RSD values were typically approximately 1 %, although some values approached 5 %. The user of this test method must decide if precision is adequate for meeting data quality objectives. Practice E1479 provides limited guidance for the parameters that may have an effect on instrument precision. Instrument troubleshooting manuals provided by the manufacturer of the equipment may also provide guidance for optimizing performance for the specific instrument being used.

15. Calibration

15.1 *Calibration Solutions and Preparation of Calibration Curve*—In this test method, calibration is based on laboratory-prepared matrix-matched calibration solutions. Matrix-matched calibration solutions are solutions that contain the approximate amount of aluminum and acid found in typical sample solutions. They are intended to model the physical behavior of sample solutions in the plasma. The matrix-matched solutions are prepared with Al stock solution prepared in 15.2 and various acids to match the sample matrix. These are spiked with aliquots of single element certified reference material (CRM) solutions or stock solutions prepared as directed in Practice E1452 and contain the analytes to be quantified and the internal standard if used.

15.1.1 *Calibration Solutions,* traceable to an SI unit through a national metrology organization, shall be prepared from certified stock solutions, typically 1000 mg/L or 10 000 mg/L, or stock solutions prepared as directed in Practice E1452.

15.1.2 The composition for each element in the calibration solution should bracket the expected level of the element in solution. Aluminum matrix solution (50 g/L), prepared in 15.2, and acid should be added to the calibration solutions to match the amount of aluminum and acid that is in the final sample solution.

NOTE 2—Paragraphs 15.1.1 and following describe the preparation of alloy matrix-matched calibration solutions for analysis of sample solutions that contain 0.5 g alloy/500 mL final dilution. It is acceptable to vary both the sample mass and final volume as long as the mass and volume chosen demonstrate the required sensitivity and precision as described in 14.2 and 14.3. It is recommended that sample mass should be at least 0.1 g to ensure representative sampling.

15.1.3 Determine the number and concentrations of the calibration solutions needed to cover the concentration range for each element. The calibration solutions should have the highest concentration slightly above the highest expected sample solution concentration, the lowest concentration near the lowest expected sample solution concentration, a concentration near the mid-range between the high and low calibration solutions, and a blank. A minimum of three calibration solutions and a blank should be used for calibration of each element.

15.2 *Aluminum Stock Solution for Matrix Matching:*

15.2.1 Prepare a 50 g/L Al matrix solution as follows: Weigh 50 g of Al drillings, chips, millings, turnings, or powder to the nearest milligram, transfer to a 1 L beaker and add about 150 mL of water and about 300 mL of HCl in small increments. **Warning—**If powdered aluminum is used, add the acid cautiously since powdered aluminum tends to be very reactive.

15.2.2 Place the beaker on a hot plate and heat the solution to approximately 90 °C to start the reaction. Remove the beaker from the hotplate when the reaction starts and cover with a watch glass. Pure aluminum dissolves slowly in HCl and complete dissolution may take several days.

15.2.3 After complete dissolution, add 2 mL to 3 mL of 30 % H_2O_2 and place the beaker on a hotplate. Heat the solution to about 110 °C and gently boil for about 5 min. Allow the solution to cool and transfer to a 1000 mL volumetric flask and dilute to volume. Transfer the solution to a polycarbonate container for storage. One milliliter of the solution will contain 50 mg of Al.

15.3 Set up the instrument for calibration as directed by the manufacturer's instructions.

15.4 Automatic calibration using the instrument operation software should be possible. Using the calibration solutions and a matrix matched blank, follow the manufacturer's procedure to perform the instrument calibration at the wavelengths specified in Table 1. Other wavelengths may be used provided they are validated for sensitivity and possible interferences. The calibration solutions and blank should be measured at least three times.

15.5 The average of the three measurements, assuming acceptable precision, should be used to construct calibration curves.

15.6 Calibration curves for ICP-AES are generally linear over several orders of magnitude. Typical calibration methods include calculation of a linear function: *(1)* using a calculated intercept, *(2)* forcing the intercept through zero, or *(3)* using concentration weighting. Method validation in accordance with Section 23 may help the laboratory to select an appropriate calibration algorithm.

15.7 Most, if not all, of the calibration curves should be linear (first order) over at least three orders of magnitude. Second order curves may be used if necessary but third order curves should not be utilized.

15.8 Verify the calibration by analyzing one or more CRM solutions as unknowns and compare the results to the certificates of analysis. Agreement should be determined based on the desired quality criteria for the results.

15.9 The user of this test method must verify the quality of the calibration fit.

15.10 Typical ICP-AES instrument software will calculate a correlation coefficient for each calibration curve. It is acceptable to rely upon the correlation coefficient as a demonstration of calibration fit. This coefficient should be 0.999 or better. If the user elects to use a linear equation with a calculated intercept then the correlation coefficient (r) is calculated by the following equation:

$$r_{xy} = \frac{n \sum_{i-1}^{n} XiYi - \sum_{i-1}^{n} Xi \sum_{i-1}^{n} Yi}{\sqrt{n \sum_{i-1}^{n} Xi^2 - \left(\sum_{i-1}^{n} X\ i \right)^2} \sqrt{n \sum_{i-1}^{n} Yi^2 - \left(\sum_{i-1}^{n} Y\ i \right)^2}} \tag{2}$$

where:

X = concentration,
Y = intensity,
n = number of calibration solutions including the blank, and
i = 1, 2,…n.

15.11 The user is cautioned that, when using this test method, it is possible to have a correlation coefficient of 1.0 and still have significant bias in the calibration curve. In this case, calculated composition values may be obtained for a verification solution(s) that disagree with the known values by a margin greater than the uncertainty goal set by the user. The user is advised to inspect all calibration curves and evaluate the potential for an unacceptable bias in certain alloys. The user of this method may choose to use other methods to judge the quality of the calibration fit, such as checking the residuals for trends and calculating a lack of fit parameter.

16. 15. Procedures for Preparing Samples

16.1 Five methods for dissolution of aluminum alloy samples are given in Sections 17 – 21. The dissolution method used is dependent on the element of interest and sometimes, the composition of the element in the alloy. Table 3 is a guide for selecting the proper dissolution method.

17. HNO_3/ HF/HCl Dissolution

17.1 Weigh approximately 0.5 g of sample to the nearest milligram into a 250 mL TFE-fluorocarbon polymer beaker. The weight, acid volume, and final volume can be adjusted to account for the composition of the alloy and the precision and sensitivity required for the analysis.

17.2 Add about 50 mL of water and 5 mL of HNO_3 to the beaker. **Warning—**If powdered aluminum is used, add the acid cautiously because powdered aluminum tends to be very reactive.

17.3 Place the beaker on a hot plate and warm the solution slowly to about 90 °C or until the reaction is complete. Do not allow the solution to boil.

17.4 Remove the beaker from the hot plate, place a watch glass on the beaker, and allow the solution to cool to room temperature.

17.5 Carefully add the amount of HF needed for the concentration of Si in the sample solution. The amount of HF required is dependent on the weight of sample and the composition of silicon in the sample. Typically, 0.2 mL of HF per 1 % Si in the alloy is sufficient if using a 0.5 g sample and a final volume of 250 mL.

17.6 Allow the reaction to complete (approximately 15 min). If the solution is not clear, add about half of the volume of HF added in 17.5, again allowing time for the reaction to complete.

17.7 Once the sample is completely in solution, add 10 mL of HCl and 2 g of H_3BO_3. Transfer the solution to a 250 mL volumetric flask. Add the internal standard if used in the calibration solutions and dilute to volume.

18. NaOH Dissolution

18.1 Weigh approximately 0.5 g of sample to the nearest milligram into a 250 mL TFE-fluorocarbon polymer beaker. The weight, acid volume, and final volume can be adjusted to

TABLE 3 Summary of Methods Applicable for Particular Elements

	Dissolution	NaOH	HF/HNO$_3$/HCl	HCl / HNO$_3$	HNO$_3$	HCl
1	Si ≥ 0.5 %	X	X			
2	Si ≤ 0.5 %			X		
3	Fe	X	X	X		
4	Cu	X	X	X		
5	Mn	X	X	X		
6	Mg	X	X	X		
7	Cr	X	X	X		
8	Ni	X	X	X		
9	Zn	X	X	X		
10	Ti	X	X	X		
11	Ag				X	X
12	As			X		
13	B			X		
14	Be	X	X	X		
15	Bi	X	X	X		
16	Ca			X		
17	Cd	X	X	X		
18	Ce			X		
19	Co	X	X	X		
20	Ga	X	X	X		
21	In			X		
22	La	X		X		
23	Li		X	X		
24	Na		X	X		
25	Pb	X	X	X		
26	Sb	X	X	X		
27	Sc			X		
28	Sn	X	X	X		
29	Sr	X	X	X		
30	V	X	X	X		
31	Zr	X	X	X		

account for the composition of the alloy and the precision and sensitivity required for the analysis. Using a plastic pipette, add 6.0 mL of 10.5 N NaOH solution and cover with a TFE-fluorocarbon polymer watch glass. **Warning—**If powdered aluminum is used, add the NaOH cautiously because powdered aluminum tends to be very reactive.

NOTE 3—Plastic pipets are recommended because NaOH will etch glass.

18.2 Wait until the reaction is complete then add 1 mL to 2 mL of H_2O_2 and again cover and wait until the reaction is complete.

18.3 Place the beaker on a hot plate and warm the solution slowly to about 110 °C. Gently boil the solution to near dryness.

18.4 Remove the beaker from the hotplate and allow the solution to cool to room temperature. Slowly add about 50 mL of warm water.

18.5 Add 15.0 mL HNO_3 (1 + 1) and 5.0 mL HCl (1 + 1) and place the beaker on a hot plate. Warm the solution slowly to approximately 110 °C.

18.6 If the solution is not clear, add a few drops of 4 % $NaNO_2$ or H_2O_2 and continue heating until the solution is clear.

18.7 Remove the beaker from the hot plate and allow the solution to cool to room temperature. Transfer the solution to a 500 mL volumetric flask. Add the internal standard if used in the calibration solutions then dilute to volume.

19. HCl /HNO$_3$ Dissolution

19.1 Weigh approximately 0.5 g of sample to the nearest milligram into a 250 mL TFE-fluorocarbon polymer beaker. The weight, acid volume, and final volume can be adjusted to account for the composition of the alloy and the precision and sensitivity required for the analysis.

19.2 Add about 50 mL of water and 5 mL of HNO_3 to the beaker. **Warning—**If powdered aluminum is used, add the acid cautiously because powdered aluminum tends to be very reactive.

19.3 Place the beaker on a hot plate and warm the solution slowly to about 90 °C or until reaction starts. Do not allow the solution to boil.

19.4 If the sample is sufficiently pure, the reaction may not start. If this happens, remove the beaker from the hot plate and add 5.0 mL of HCl. Return the beaker to the hot plate and again warm the solution slowly to about 90 °C or until the reaction starts. Do not allow the solution to boil. **Warning—**If powdered aluminum is used, add the acid cautiously because powdered aluminum tends to be very reactive.

19.5 Add 2 drops to 3 drops of H_2O_2 while the sample is reacting. Remove the beaker from the hot plate when the sample is completely dissolved and allow the solution to cool to room temperature.

19.6 Add an additional 5.0 mL of HCl.

19.7 Once the sample is completely in solution, transfer the solution to a 250 mL volumetric flask. Add the internal standard if used in the calibration solutions then dilute to volume.

20. HNO$_3$ Dissolution

20.1 Weigh approximately 0.5 g of sample to the nearest milligram into a 250 mL TFE-fluorocarbon polymer beaker.

The weight, acid volume, and final volume can be adjusted to account for the composition of the alloy and the precision and sensitivity required for the analysis.

20.2 Add about 50 mL of water and 25 mL of HNO_3 to the beaker. **Warning—**If powdered aluminum is used, add the acid cautiously because powdered aluminum tends to be very reactive.

20.3 Place the beaker on a hot plate and warm the solution slowly to about 90 °C or until the reaction is complete. Do not allow the solution to boil.

20.4 Once the sample is completely in solution, transfer the solution to a 250 mL volumetric flask. Add the internal standard if used in the calibration solutions then dilute to volume.

NOTE 4—Solutions containing Ag should be kept away from direct sunlight or stored in opaque labware.

21. HCl Dissolution

21.1 Weigh approximately 0.5 g of sample to the nearest milligram into a 250 mL TFE-fluorocarbon polymer beaker. The weight, acid volume, and final volume can be adjusted to account for the composition of the alloy and the precision and sensitivity required for the analysis.

21.2 Add about 50 mL of water and 30 mL of HCl to the beaker. **Warning—**If powdered aluminum is used, add the acid cautiously because powdered aluminum tends to be very reactive.

21.3 Place the beaker on a hot plate and warm the solution slowly to about 90 °C or until reaction starts. Do not allow the solution to boil.

21.4 Once the sample is completely in solution, transfer the solution to a 100 mL volumetric flask. Add the internal standard if used in the calibration solutions then dilute to volume (Note 4).

22. Control

22.1 Maintain control charts for elements of interest for each control sample. Refer to Practice E1329 for guidance.

22.2 Most ICP-AES instrument manufacturer's software allows the use of programmable control sample tolerances. It is acceptable to calculate control limits and to use these in the instrument software.

22.3 The individual laboratory's analysis procedures will typically specify reanalysis of affected samples if control samples indicate that the calibration is no longer valid.

23. Test Method Validation

23.1 A laboratory using this test method for the first time shall provide additional method validation data to demonstrate that the method as applied in their laboratory is yielding unbiased, repeatable results.

23.2 Initially, the laboratory should prepare and analyze solid CRMs, or RMs, or both, using this test method to obtain this data. If solid CRMs or RMs are not available for the alloys/analytes being determined, then spike recovery studies using alloy samples should be part of the validation process. The precision and bias data obtained for these materials should be compared to the precision and bias data stated in this test method.

23.3 Any laboratory demonstrating significantly worse data should attempt to identify and correct any problems associated with their application of this test method.

23.4 The test method user must weigh customer requirements and their data quality objectives and justify acceptance of the method validation data.

23.5 The test method validation study shall be documented. Consult Guide E2857 for guidance.

24. Calculations

24.1 If the user chooses to specify units in the ICP-AES instrument software to express the amount of analyte contained in the sample as a mass fraction, then no other calculations other than sample weight correction will be necessary.

24.1.1 Results may be taken directly from the instrument readout.

24.2 If the user specified analyte composition as a volume fraction in the software, it will be necessary to convert the analyte volume fraction obtained for the sample solution into analyte mass fraction contained in the sample. For example, if the sample is prepared as 1 g of sample diluted to a final volume of 100 mL solution, an analyte volume fraction of 1.00 mg analyte/L of solution corresponds to a mass fraction of 0.010 % analyte in the sample.

25. Report

25.1 Results shall be reported in accordance with customer requirements. When uncertainty estimates are required, results should be reported as directed in ISO Guide 98-3. This document explains that the analyst must obtain an estimate of the overall uncertainty of the result, and express that uncertainty as an expanded uncertainty $U = ku_c$, where u_c is a combined uncertainty expressed at the level of 1 *s* (one standard deviation), and *k* is an expansion factor typically chosen as $k = 2$ to approximate a 95 % level of confidence. It is expected that the laboratory will include all significant sources of uncertainty in their estimate of the combined uncertainty. Express the value of *U* with 3 significant digits. Then, express the reported result to the same number of decimal places.

25.2 On the basis of the performance embodied in the interlaboratory study results, it is clear that no results shall be reported with digits beyond the level of 0.0001 % (based on the ILS or the equivalent 1 mg/kg). No reported results shall exceed three significant figures. The individual laboratory's performance may dictate fewer decimal places in the results, especially for results of significantly greater magnitude than the limit of quantification of the method as implemented in the particular laboratory.

26. Precision and Bias

26.1 26.1 The precision of this test method is based on an interlaboratory study conducted in 2015. Eleven laboratories tested 30 different samples, at both high and low composition levels, for up to 32 elements. Every test result represents an individual determination. All participants provided test results in triplicate. Practice E691 was followed for the design and analysis of the data; the details are given in RR:E01-1126.[8]

26.1.1 *Repeatability, r*—The difference between repetitive results obtained by the same operator in a given laboratory applying the same test method with the same apparatus under constant operating conditions on identical test material within short intervals of time would in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

26.1.1.1 Repeatability can be interpreted as the maximum difference between two results, obtained under repeatability conditions, which is considered acceptable due to random causes under normal and correct operation of the test method.

26.1.1.2 Averaged repeatability limits are listed in Tables 4 and 5.

26.1.2 *Reproducibility, R*—The difference between two single and independent results obtained by different operators applying the same test method in different laboratories using different apparatus on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in 20.

26.1.2.1 Reproducibility can be interpreted as the maximum difference between two results, obtained under reproducibility conditions, which is considered acceptable due to random causes under normal and correct operation of the test method.

26.1.2.2 Averaged reproducibility limits are listed in Tables 4 and 5.

26.1.3 The above terms (*repeatability limit* and *reproducibility limit*) are used as specified in Practice E177.

26.1.4 Any judgment made according to statements 26.1.1 and 26.1.2 would have an approximate 95 % probability of being correct.

26.2 *Bias*—Relative bias can be found in Tables 4 and 5.

26.3 The precision statement was determined through statistical examination of averaged results from 11 laboratories, on 30 different materials, measured for as many as 32 elements, at both high and low compositions.

27. Keywords

27.1 aluminum alloys; ICP; inductively coupled plasma atomic emission spectrometry; performance based method

TABLE 4 Average Reproducibility Limits - Low Level

NOTE 1—All results are expressed as mass fraction (%) .

Element	Sample ID	Certified Value	Xbar, $\bar{X}$	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Range, *r*	Reproducibility Range, *R*	Percent Relative Bias
Ag	11880S	0.003	0.00325	0.00010	0.00052	0.00027	0.00145	8.4
As	372	0.0085	0.00715	0.00049	0.00088	0.00136	0.00245	–15.9
B	BN3	0.012	0.01138	0.00023	0.00152	0.000629	0.004255	–5.2
Ba	11880S	0.0009	0.001030	0.000030	0.00043	0.00009	0.00121	14.8
Be	11880S	0.0009	0.000840	0.000050	0.000320	0.000140	0.000890	–6.9
Bi	11880S	0.0058	0.00607	0.00040	0.002208	0.001132	0.006183	4.7
Ca	11880S	0.001	0.001170	0.000070	0.00047	0.00019	0.00131	16.7
Cd	11880S	0.0011	0.001096	0.000033	0.000277	0.000093	0.000777	–0.3
Co	11880S	0.0011	0.001181	0.000033	0.000278	0.000093	0.000779	7.4
Cr	11880S	0.007	0.00657	0.00026	0.000695	0.000727	0.001947	–6.1
Cu	11880S	0.0052	0.00488	0.00028	0.00083	0.00078	0.00231	–6.2
Fe	1000	0.011	0.00905	0.00051	0.003431	0.00143	0.009608	–17.7
Ga	1000	0.01	0.00991	0.00030	0.00244	0.00083	0.00684	–0.9
Li	11880S	0.0009	0.00073	0.00002	0.00026	0.00006	0.00074	–19
Mg	11880S	0.0051	0.00550	0.00019	0.00104	0.00052	0.00291	7.8
Mn	11880S	0.0066	0.00675	0.00017	0.00049	0.00048	0.00136	2.3
Mo	11880S	0.01	0.01325	0.00029	0.00289	0.00081	0.00809	32.5
Na	337	0.003	0.00334	0.00015	0.00139	0.00041	0.00389	11.4
Ni	11880S	0.0073	0.00690	0.00020	0.00069	0.00055	0.00193	–5.5
P	488	0.007	0.00384	0.00090	0.00231	0.00251	0.00648	–45.2
Pb	11880S	0.0071	0.00574	0.00035	0.001616	0.000965	0.004525	–19.1
Sb	354	0.011	0.00956	0.00048	0.00224	0.00135	0.00626	–13.1
Sc	376	0.018	0.0169	0.00048	0.00192	0.00135	0.00537	–6.1
Si	1000	0.0105	0.00925	0.00039	0.0039	0.0011	0.01093	–11.9
Sn	11880S	0.0065	0.00705	0.00058	0.00135	0.00163	0.00378	8.5
Sr	11880S	0.0013	0.000940	0.000040	0.00014	0.0001	0.00039	–27.4
Ti	11880S	0.0072	0.00646	0.00014	0.000910	0.000380	0.002540	–10.3
Tl	11880S	0.0026	0.00276	0.00027	0.001687	0.000754	0.004725	6.1
V	11880S	0.02	0.01973	0.00044	0.00204	0.00124	0.00571	–1.3
Zn	11880S	0.007	0.00827	0.00046	0.00402	0.00128	0.01126	18.1
Zr	11880S	0.0026	0.00276	0.00021	0.00069	0.00058	0.00193	6.3

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[8] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1126. Contact ASTM Customer Service at service@astm.org.

TABLE 5 Average Reproducibility Limits - High Level

NOTE 1—All results are expressed as mass fraction (%)

Element	Sample ID	Certified Value	Xbar, $\bar{X}$	Repeatability Standard Deviation, S_r	Reproducibility Standard Deviation, S_R	Repeatability Range, r	Reproducibility Range, R	Percent Relative Bias
Ag	675	0.4	0.3869	0.0021	0.0281	0.0058	0.0786	–3.3
As	6063	0.012	0.00929	0.00097	0.00187	0.0027	0.00523	–22.6
B	BN5	0.027	0.028329	0.00064	0.003621	0.001792	0.01014	4.9
Ba	PROF	0.03	0.0299	0.00064	0.002	0.0018	0.00561	–0.3
Be	PROF	0.11	0.1121	0.0017	0.0143	0.0048	0.0399	1.9
Bi	6262	0.587	0.5677	0.0056	0.0377	0.0156	0.1055	–3.3
Ca	CA4	0.048	0.0517	0.0010	0.0096	0.0029	0.0268	7.8
Cd	CD4	0.055	0.05907	0.00087	0.00813	0.00244	0.02277	7.4
Co	PROF	0.034	0.03196	0.00058	0.00562	0.00163	0.01573	–6
Cr	712	0.52	0.5196	0.0039	0.0309	0.0110	0.0864	–0.1
Cu	213	7.03	7.054	0.065	0.321	0.182	0.899	0.3
Fe	2800	3.06	3.008	0.047	0.228	0.132	0.640	–1.7
Ga	3003	0.019	0.0200	0.0011	0.003	0.0030	0.0087	5.2
Li	8090	2.48	2.448	0.012	0.160	0.033	0.449	–1.3
Mg	518	8.15	8.286	0.044	0.314	0.124	0.880	1.7
Mn	3003	1.41	1.455	0.023	0.095	0.063	0.266	3.2
Mo	PROF	0.15	0.1441	0.0017	0.0121	0.0049	0.0338	–3.9
Na	NA5	0.026	0.024804	0.00078	0.006602	0.002184	0.018487	–4.6
Ni	332	2.71	2.746	0.033	0.158	0.093	0.441	1.3
P	390	0.025	0.01832	0.0011	0.00112	0.00313	0.00313	–26.7
Pb	2011	0.51	0.5171	0.0028	0.0262	0.0077	0.0733	1.4
Sb	PROF	0.28	0.2549	0.0021	0.0117	0.0060	0.0327	–9
Sc	495	0.065	0.0654	0.0016	0.0055	0.0045	0.0154	0.6
Si	390	16.8	17.16	0.12	0.42	0.34	1.19	2.1
Sn	850	6.28	6.059	0.066	0.271	0.185	0.760	–3.5
Sr	332	0.028	0.02539	0.00031	0.00327	0.00088	0.00915	–9.3
Ti	206	0.202	0.20	0.0020	0.0108	0.0057	0.0301	–0.3
Tl	PROF	0.13	0.1143	0.0024	0.0138	0.0067	0.0386	–12.1
V	2219	0.12	0.12586	0.00098	0.01543	0.00274	0.04321	4.9
Zn	637	9.65	9.87	0.12	0.39	0.34	1.09	2.2
Zr	ZR15	0.25	0.23482	0.0042	0.02779	0.01176	0.07783	–6.1