



# Standard Test Method for Analysis of Zinc-5 % Aluminum-Mischmetal Alloys by ICP Emission Spectrometry<sup>1</sup>

This standard is issued under the fixed designation E1277; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This test method covers the chemical analysis of zinc alloys having chemical compositions within the following limits:

Element	Composition Range, %
Aluminum	3.0–8.0
Antimony	0.002 max
Cadmium	0.025 max
Cerium	0.03–0.10
Copper	0.10 max
Iron	0.10 max
Lanthanum	0.03–0.10
Lead	0.026 max
Magnesium	0.05 max
Silicon	0.015 max
Tin	0.002 max
Titanium	0.02 max
Zirconium	0.02 max

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

1.3 Included are procedures for elements in the following composition ranges:

Element	Composition Range, %
Aluminum	3.0–8.0
Cadmium	0.0016–0.025
Cerium	0.005–0.10
Iron	0.0015–0.10
Lanthanum	0.009–0.10
Lead	0.002–0.026

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.* Specific safety hazards statements are given in Section 8, 11.2, and 13.1.

<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 E01.05 on Cu, Pb, Zn, Cd, Sn, Be, Precious Metals, their Alloys, and Related Metals.

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## 2. Referenced Documents

2.1 *ASTM Standards*:<sup>2</sup>

D1193 Specification for Reagent Water

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)<sup>3</sup>

E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data (Withdrawn 2003)<sup>3</sup>

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

2.2 *NIST Standard Reference Materials*:<sup>4</sup>

SRM 728 Zinc, Intermediate Purity

## 3. Terminology

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

## 4. Summary of Test Method

4.1 The sample is dissolved in mixed acids. The sample solution is introduced into the plasma source of an ICP spectrometer and the intensities at selected wavelengths from

<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

<sup>4</sup> Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

**TABLE 1 Wavelengths and Instrument Conditions<sup>A</sup>**

Element	Wavelength, nm	Time, s	No. Integ.	BCor1	BCor2
Aluminum	309.27	1.0	3	...	...
Cadmium	226.502	.5	3	226.446	226.558
Cerium	418.66	.5	2	...	...
Iron	259.94	.5	2	...	...
Lanthanum	398.85	.5	2	398.754	398.906
Lead	283.297	1.0	3	...	283.336

<sup>A</sup> The tabulated conditions were those found satisfactory on one instrument. Wavelengths are expressed in nanometres (nm). Time = seconds for each integration, No. Integ. = number of integrations averaged for each reading, and BCor1 and BCor2 are off-peak background correction wavelengths.

the plasma emission spectrum are compared to the intensities at the same wavelengths measured with calibration solutions.

## 5. Significance and Use

5.1 This test method for the chemical analysis of metals and alloys is primarily intended to test such materials for compliance with compositional specifications. It is assumed that all those who use this test method will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

## 6. Apparatus

6.1 *Inductively-Coupled Argon Plasma (ICP) Atomic Emission Spectrometer*—The instrument may be either sequential or simultaneous, axial or radial, and shall be capable of isolating the required wavelengths shown in **Table 1** for measurement of their intensities. Multielement programmed analysis including automatic data acquisition and computer-controlled calibration and determinations may be used if available, provided that, in addition to calculated results, the instrument records intensity readings each time a test sample or calibration solution is presented to the instrument.

## 7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>5</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification **D1193**.

7.3 *Aluminum, Standard Solution* (1 mL = 20.0 mg Al)—Transfer 2.0000 g of aluminum (purity: 99.999 % minimum) to a 250-mL beaker. Cover, add 50 mL of HCl (1 + 1) and a small crystal of mercuric nitrate. Heat gently to accelerate the

reaction, but avoid temperatures high enough to cause a noticeable volume loss. If the reaction slows, add more mercuric salt as needed. A number of hours may be required to complete the dissolution (only a small droplet of mercury will remain undissolved). Transfer the solution to a 100-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

7.4 *Cadmium, Standard Solution* (1 mL = 1.00 mg Cd)—Transfer 1.000 g of cadmium (purity: 99.95 % minimum) to a 250-mL beaker. Cover and add 40 mL of HNO<sub>3</sub> (1 + 1) and 10 mL of HCl. After dissolution is complete, heat to boiling to remove oxides of nitrogen. Cool, transfer to a 1-L volumetric flask, add 240 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

7.5 *Cerium, Standard Solution A* (1 mL = 1.00 mg Ce)—Dry ceric ammonium nitrate ((NH<sub>4</sub>)<sub>2</sub>Ce(NO<sub>3</sub>)<sub>6</sub>, also known as ammonium hexanitrate cerate) (purity: 99.95 % minimum) for 4 h at 85 °C and cool to room temperature in a desiccator. Dissolve 3.913 g of dry ceric ammonium nitrate in 100 mL of HCl (1 + 9). Transfer to a 1-L volumetric flask, add 240 mL of HCl and 20 mL of HNO<sub>3</sub>, dilute to volume, and mix. Store in a polyethylene bottle.

7.6 *Cerium, Standard Solution B* (1 mL = 0.010 mg Ce)—Using a pipet, transfer 1.00 mL of Cerium Standard Solution A to a 100-mL volumetric flask. Dilute to volume with dilution solution and mix.

7.7 *Dilution Solution*—Half fill a 2-L volumetric flask with water. Add 500 mL of HCl and 40 mL of HNO<sub>3</sub>, swirl to mix, dilute to the mark, and mix.

7.8 *Iron, Standard Solution A* (1 mL = 1.00 mg Fe)—Transfer 1.000 g of iron (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 100 mL of HCl (1 + 1). Boil gently to complete dissolution. Cool and transfer to a 1-L volumetric flask, add 200 mL of HCl and 20 mL of HNO<sub>3</sub>, dilute to volume, and mix. Store in the polyethylene bottle.

7.9 *Iron, Standard Solution B* (1 mL = 0.010 mg Fe)—Using a pipet, transfer 1.00 mL of Iron Standard Solution A to a 100-mL volumetric flask. Dilute to volume with dilution solution and mix.

7.10 *Lanthanum, Standard Solution A* (1 mL = 0.010 mg La)—Ignite lanthanum oxide (La<sub>2</sub>O<sub>3</sub>) (purity: 99.9 % minimum) for 1 h at 1000 °C and cool to room temperature in a desiccator. Dissolve 1.173 g of dry lanthanum oxide in 100 mL of HCl (1 + 9) and transfer to a 1-L volumetric flask. Add 240 mL of HCl and 20 mL of HNO<sub>3</sub>, dilute to volume, and mix. Store in a polyethylene bottle.

7.11 *Lanthanum, Standard Solution B* (1 mL = 0.010 mg La)—Using a pipet, transfer 1.00 mL of Lanthanum Standard Solution A to a 100-mL volumetric flask. Dilute to volume with dilution solution and mix.

7.12 *Lead, Standard Solution* (1 mL = 1.00 mg Pb)—Transfer 1.000 g of lead (purity: 99.9 % minimum) to a 250-mL beaker, cover, and add 40 mL of HNO<sub>3</sub> (1 + 1). Boil gently to complete dissolution and to remove oxides of

<sup>5</sup> 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 Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

nitrogen. Cool, transfer to a 1-L volumetric flask, add 250 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

7.13 *Zinc Matrix Solution* (50 mL = 3.75 g Zinc Matrix Standard)—Transfer 18.75 g ± 0.10 g of Zinc Matrix Standard to a 250-mL plastic beaker. Cover and add about 50 mL of water. Add 62.5 mL of HCl and heat enough to maintain the reaction but not enough to evaporate the solution. When most of the material has dissolved, add 5.0 mL of HNO<sub>3</sub>. When all solids have dissolved, remove from the heat and allow to cool. Transfer to a 250-mL plastic volumetric flask, dilute to the mark, and mix.

7.14 *Zinc Matrix Standard*—Use a zinc reference material of known composition (SRM 728 has been found suitable) with respect to the elements listed in the scope of this test method.

## 8. Hazards

8.1 For precautions to be observed in the use of certain reagents in this test method, refer to Practices E50.

## 9. Sampling

9.1 For procedures for sampling the material, refer to Practices E55 and E88.

## 10. Calibration

10.1 Prepare calibration and test sample solutions before calibration measurements are started.

10.2 *Calibration Solutions*—All calibration solutions contain the same composition of zinc as the test sample solutions. The aluminum content of calibration solutions No. 2 and No. 3 shall be equal to the midpoint of the calibrated aluminum range. Using a pipet, transfer 50.0 mL of the Zinc Matrix Solution into each of four 100-mL plastic volumetric flasks marked Cal No. 1 through Cal No. 4. Add the volumes of standard solutions specified in Table 2 (also see Table 3), dilute to volume with dilution solution, and mix.

NOTE 1—All elements (including aluminum) are calibrated as linear functions of intensity. If the instrument cannot be set to measure aluminum and ignore other elements in calibration solutions No. 1 and No. 4, then a separate determination of aluminum shall be made using calibration solutions No. 1, No. 2, and No. 4. The other elements can then be determined together in another run using only calibration solutions No. 2 and No. 3. Use the calibration solutions prepared in 10.1 in determining the instrument settings for the elements in this matrix. Follow the manufacturer's instructions to set the wavelengths and parameters to provide as large a difference between the intensity readings for the high and low calibration compositions as is consistent with stable instrument readings. If there is a question of linearity of the instrument's response

**TABLE 2 Standard Solution Volumes Added, mL<sup>A,B</sup>**

Element	No. 1	No. 2	No. 3	No. 4
Aluminum	6.00	11.0	11.0 <sup>B</sup>	16.0
Cadmium	...	...	1.00	...
Cerium	...	2.00(B)	4.00(A)	...
Iron	...	1.00(B)	4.00(A)	...
Lanthanum	...	2.00(B)	4.00(A)	...
Lead	...	...	1.00	...

<sup>A</sup> Use standard solution A or B as indicated in parentheses.

<sup>B</sup> Added to match solution No. 2, not for calibration purposes.

**TABLE 3 Solution Compositions Added, mg/L<sup>A</sup>**

Element	No. 1	No. 2	No. 3	No. 4
Aluminum	1200	2200	...	3200
Cadmium	...	...	10.0	...
Cerium	...	0.2	40.0	...
Iron	...	0.1	40.0	...
Lanthanum	...	0.2	40.0	...
Lead	...	...	10.0	...

<sup>A</sup> Table 4 is derived from this table by adding the trace element contributions from the zinc matrix solution to the compositions shown in this table and converting the resulting sum to weight percent.

over the range of solution compositions given, a third standard, equidistant between the two listed standards, shall be measured to verify linearity.

10.3 *Test Sample Solution*—Transfer a 3.8-g to 4.2-g portion of the test sample weighed to the nearest 0.02 g to a 250-mL polytetrafluoroethylene beaker. Add about 30 mL of water, cover, and cautiously add 25 mL of HCl in increments. Heat gently to maintain the reaction, if necessary, but do not boil. When most of the material has dissolved, add 2.0 mL of HNO<sub>3</sub>, let the solution cool for about 20 min, transfer to a 100-mL plastic volumetric flask, dilute to volume, and mix.

10.4 *Automatic Calibration Mode*—(If the instrument does not have the capability to take data from calibration solutions and calculate and store the equations needed to convert instrument readings from test samples directly into composition values automatically, or if that capability is not to be used, proceed in accordance with 10.5.) Set up the instrument parameters as directed in Section 6. If one of the parameters is a “lower limit” (used to establish a printed “less than” value), set it to 0 for each element. Enter the compositions of the elements to be found in each calibration solution. Table 4 gives the composition table for solutions based upon SRM 728 as Zinc Matrix Standard. If a different Zinc Matrix Standard is used, Table 4 shall be revised to reflect the different composition of that material. Using the calibration solutions, follow the manufacturer's procedure to perform the instrument calibration at the wavelengths specified in Table 1. Without undue delay, proceed in accordance with 11.2.

10.5 *Nonautomatic Mode*—No separate calibration run is required if intensity readings only are recorded. Set up the instrument to measure intensities at the wavelengths specified in Table 1 according to the manufacturer's instructions and proceed to 11.3.

**TABLE 4 Composition Table for Calibration Solutions<sup>A,B</sup>**

Element	No. 1	No. 2	No. 3	No. 4
Aluminum	3.00	5.50	...	8.00
Cadmium	...	.00011	.0251	...
Cerium	...	.00050	.1000	...
Iron	...	.00052	.1003	...
Lanthanum	...	.00050	.1000	...
Lead	...	.00104	.0260	...

<sup>A</sup> The values in this table assume SRM 728 as zinc matrix, a sample weight of 4.00 g, and results reported in %.

<sup>B</sup> To calculate the composition table for a different zinc matrix material, add the parts per million contributed from 3.75 g of that material in a volume of 100 mL to the parts per million shown in Table 3. Calculate the percent element by dividing the parts per million by 400.

## 11. Procedure

11.1 *Measurement Sequences*—To reduce the distortion of data if instrument drift occurs while measurements are taken, solutions are presented to the instrument in a specified order and only a single reading (or, if desired, the average of several integrations) is recorded each time a solution is presented to the instrument. Repeat the following sequence of solution presentations four times to obtain the required four replicate readings: calibration solution No. 1, calibration solution No. 2, test sample solution, calibration solution No. 3, and calibration solution No. 4. More than one test sample solution may be presented to the instrument between calibration solutions No. 2 and No. 3. Many instruments do not require a rinse between each solution presentation, but it is advisable to rinse the system periodically. A rinse with dilution solution after each completed sequence is the minimum recommended frequency.

11.2 *Automatic Mode*—Calibrate the instrument in accordance with 10.4. Without undue delay, proceed to analyze the solutions as directed in 11.1. (**Warning** —Attempting to shorten the measurement time by substituting four readings taken during a single solution presentation instead of the prescribed sequences may lead to an improper calibration even though the precision of the measurements appears satisfactory. Be sure that the instrument has been set to record intensities as well as compositions for both calibration and test solutions. Proceed in accordance with 12.1.)

11.3 *Nonautomatic Mode*—With the instrument set up in accordance with Section 6, measure the calibration and test sample solutions as directed in 11.1, recording the intensity readings for each solution presentation. Solution presentation may be performed manually, or, if the instrument is provided with the necessary equipment, by automatic solution presentation. The proper sequencing of the replicate readings shall be followed in either case. Proceed in accordance with 12.2.

## 12. Calculation

12.1 *Automatic Mode*—The instrument record includes calibration and test solutions results in both intensity and composition units. To test the accuracy of the recorded compositions, calculate the average compositions of the sample and the appropriate calibration solutions. For each calibration solution, determine the difference between the average recorded composition and the value listed in the composition table. The automatically calculated composition values are acceptable if the differences are less than 5 % (relative) for the high and 10 % (relative) for the low calibration solution. The requirement for aluminum is that the average error for the three calibration results shall be within 2 % (relative). If the results for an element meet these criteria, record the average test sample composition ( $C_s$ ), and proceed in accordance with Section 13. Otherwise, use the intensity readings for the element to calculate results as directed in 12.2.

12.2 *Nonautomatic Mode*—The measurement data consists of four intensity readings for each calibration and test sample solution. Calculate the average intensity values for the high calibration solution ( $R_h$ ), the low calibration solution ( $R_l$ ), and the test sample solution ( $R_s$ ). For aluminum, also determine the

average intensity ( $R_m$ ) for the middle calibration solution. Determine the test sample composition as directed in either 12.3 or 12.4.

12.3 *Graphical Calibration*—Plot  $R_h$  and  $R_l$  (also  $R_m$  for aluminum) as the  $y$  variable (measured along the vertical axis) against the corresponding compositions from the composition table as the  $x$  variable (horizontal axis). Draw the calibration curve, a line which (except for aluminum) extends through both points back to intersect the vertical axis. For aluminum, the calibration curve is a straight line plotted from the composition value for the low calibration solution to the composition value of the high calibration solution in such a manner that the positive and negative differences between the line and all points is minimized. Note that no calibration curve can be extended above the composition of its high end. In addition, the aluminum curve cannot be used for compositions below its low end. The composition of the test sample ( $C_s$ ) is read from the  $x$  axis corresponding to the intersection of the average sample reading ( $R_s$ ) with the calibration curve. In the same manner, read the compositions corresponding to all of the individual intensity readings for the low calibration solution. Proceed in accordance with Section 13.

12.4 *Calculator Calibration*—Use a calculator or computer least-squares curve fit program that also calculates predicted values for  $x$  and  $y$  from given values of the other variable. Enter the intensity readings for calibration solutions as the  $y$  variable with compositions from the composition table as the associated  $x$  values. Enter the average intensity reading of the test sample solution and use the program to predict its composition ( $C_s$ ). In the same manner, calculate the compositions corresponding to all of the individual intensity readings for the low calibration solution. Proceed as directed in Section 13.

12.5 *Determination Limit*, is calculated in accordance with Practice E876. For this application, it is defined as the composition below which the relative error of the calculated composition is predicted to be greater than 15 % at the 95 % confidence level. The determination limit concept does not apply to the aluminum determination because the calibration curve for that element does not extend lower than approximately 3 % aluminum. For all other elements, the determination limit establishes the lowest practical composition that can be reported by the use of this test method. This test method specifies four replicate readings for both calibration and sample solutions. Use the standard deviation of the low calibration solution readings to calculate the determination limit:

$$DL = 10.6 \times s \text{ where } s = \text{the standard deviation} \quad (1)$$

12.6 *Test Sample Composition, (C)*—Calculate by correcting for the sample weight if different from 4.00 g:

$$C = C_s \times (4/A) \quad (2)$$

where:

$C_s$  = the average recorded test sample composition, and  
 $A$  = the test sample weight, g.

## 13. Report

13.1 Report the aluminum content as  $C$  % Al if the calculated value falls within the range of 3.0 % to 8.0 %. Otherwise,

do not report the results for aluminum *or any other element* because the test sample is not within the scope of this test method. For each other element, calculate the determination limit as directed in 12.5. Compare this value with the lower scope limit and use the greater of the two as the lower reporting limit (LRL). If the calculated composition, *C*, of the element is less than the value of LRL, report the composition of the element as “less than” LRL. If *C* is greater than the value of LRL, report the element composition as *C* % Element. (**Warning**—Do not report any element whose composition is more than 10 % (relative) higher than the high calibration composition for that element.)

13.2 Calculated values shall be rounded to the desired number of places as directed in 6.4 to 6.6 of Practice E29.

#### 14. Precision and Bias

14.1 *Precision*—Only four laboratories were available to test this method, therefore the interlaboratory study does not comply with the protocol for Practice E173. However, the statistics were calculated in accordance with Practice E173. The results are summarized in Table 5.

14.2 *Bias*—The bias of this test method could not be evaluated because adequate certified standard reference materials were unavailable at the time of testing. The user is cautioned to verify by the use of certified reference materials, if available, that the accuracy of this test method is adequate for the contemplated use.

14.3 Practice E173 has been replaced by Practice E1601. The Reproducibility Index ( $R_2$ ) corresponds to the Reproduc-

**TABLE 5 Statistical Information**

NOTE 1—Results reported in weight percent.

Element	Sample	Number of Laboratories	Mean	$R_1$	$R_2$
Aluminum	Gal-5	4	4.88	0.139	0.273
	Gal-6	4	5.31	0.097	0.319
	Gal-7	4	6.93	0.384	0.582
Cadmium	Gal-5	4	0.00020	ND <sup>A</sup>	ND <sup>A</sup>
	Gal-6	4	0.00008	ND <sup>A</sup>	ND <sup>A</sup>
	Gal-7	4	0.0109	0.00067	0.00082
Cerium	Gal-5	4	0.0235	0.0015	0.0026
	Gal-6	4	0.0399	0.0022	0.0031
	Gal-7	4	0.0306	0.0021	0.0020
Iron	Gal-5	4	0.0040	0.00054	0.00076
	Gal-6	4	0.0274	0.0057	0.0114
	Gal-7	4	0.0291	0.0047	0.0074
Lanthanum	Gal-5	4	0.0299	0.0018	0.0044
	Gal-6	4	0.0664	0.0039	0.0050
	Gal-7	4	0.0339	0.0026	0.0072
Lead	Gal-5	3	0.0047	0.00095	0.00090
	Gal-6	3	0.0013	0.0013	0.0012
	Gal-7	3	0.0062	0.00089	0.0011

<sup>A</sup> Not determinable. The data was not suitable for  $R_1$  and  $R_2$  calculations.

ibility Index ( $R$ ) of Practice E1601. Likewise, the Repeatability Index ( $R_1$ ) of Practice E173 corresponds to the Repeatability Index ( $r$ ) of Practice E1601.

#### 15. Keywords

15.1 inductively-coupled argon plasma atomic emission spectrometer; mischmetal; rare earths; spectrometry; zinc alloys

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