



Designation: **E1277–14** **E1277 – 22**

Standard Test Method for Analysis of Zinc-5 % Aluminum-Mischmetal Alloys by ICP Inductively Coupled Plasma Atomic Emission Spectrometry¹

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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

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 Units—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, health, and environmental 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.*

1.5 *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.*

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 E01.05 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:²

- 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 1997)³
- E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data (Withdrawn 2003)³
- E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

2.2 NIST Standard Reference Materials:⁴

- 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 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.

TABLE 1 Wavelengths and Instrument Conditions^A

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

^A 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.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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

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.⁴ 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~~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~~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~~250 mL beaker. Cover and add 40 mL of HNO₃ (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~~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 ((NH₄)₂Ce(NO₃)₆, 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~~1 L volumetric flask, add 240 mL of HCl and 20 mL of HNO₃, 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~~100 mL volumetric flask. Dilute to volume with dilution solution and mix.

7.7 *Dilution Solution*—Half fill a ~~2-L~~2 L volumetric flask with water. Add 500 mL of HCl and 40 mL of HNO₃, 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~~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 HNO₃, 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~~100 mL volumetric flask. Dilute to volume with dilution solution and mix.

7.10 *Lanthanum, Standard Solution A* (1 ~~mL = 0.010 mg~~ mL = 1.00 mg La)—Ignite lanthanum oxide (La₂O₃) (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~~1 L volumetric flask. Add 240 mL of HCl and 20 mL of HNO₃, 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~~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~~250 mL beaker, cover, and add 40 mL of HNO₃ (1 + 1). Boil gently to complete dissolution and to remove oxides of nitrogen. Cool, transfer to a ~~1-L~~1 L volumetric flask, add 250 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

⁴ Reagent Chemicals, *American Chemical Society Specifications* (www.acs.org), 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* (www.usp.org), U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.13 *Zinc Matrix Solution* (50 mL = 3.75 g Zinc Matrix Standard)—Transfer 18.75 g \pm 0.10 g of Zinc Matrix Standard to a ~~250-mL~~ 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 HNO₃. When all solids have dissolved, remove from the heat and allow to cool. Transfer to a ~~250-mL~~ 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~~ (NIST SRM 728⁵) 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 Sampling the material is described in 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~~ 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~~ 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~~ 10.2 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 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~~ 3.8 g to ~~4.2-g~~ 4.2 g portion of the test sample weighed to the nearest 0.02 g to a ~~250-mL~~ 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 HNO₃, let the solution cool for about 20 min, transfer to a ~~100-mL~~ 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

TABLE 2 Standard Solution Volumes Added, mL^{A,B}

Element	No. 1	No. 2	No. 3	No. 4
Aluminum	6.00	11.0	11.0 ^B	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	...

^A Use standard solution A or B as indicated in parentheses.

^B Added to match solution No. 2, not for calibration purposes.

TABLE 3 Solution Compositions Added, mg/L^A

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	...

^A 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.

TABLE 4 Composition Table for Calibration Solutions^{A,B}

Element	No. 1	No. 2	No. 3	No. 4
Aluminum	3.00	5.50	...	8.00
Cadmium00011	.0251	...
Cerium00050	.1000	...
Iron00052	.1003	...
Lanthanum00050	.1000	...
Lead00104	.0260	...

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

^BTo 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.

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 only 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.

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~~ Ensure 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~~ Regardless, 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