



Standard Test Method for Chemical Analysis of Zinc-5 % Aluminum-Mischmetal Alloys by ICP Emission Spectrometry¹

This standard is issued under the fixed designation E 1277; 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 covers the chemical analysis of zinc alloys having chemical compositions within the following limits:

Element	Concentration 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 Included are procedures for elements in the following concentration ranges:

Element	Concentration 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

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific safety hazards statements are given in Section 7.*

2. Referenced Documents

2.1 ASTM Standards:

D 1193 Specification for Reagent Water²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications³

¹ This test method is under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.05 on Zn, Sn, Pb, Be, and Other Metals.

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² Annual Book of ASTM Standards, Vol 11.01.

³ Annual Book of ASTM Standards, Vol 14.02.

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals⁴

E 55 Practice for Sampling Wrought Nonferrous Metals for Determination of Chemical Composition⁴

E 88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition⁴

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals⁴

E 876 Practice for Use of Statistics in the Evaluation of Spectrometric Data⁵

3. Summary of Test Method

3.1 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.

4. Significance and Use

4.1 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.

5. Apparatus

5.1 *Inductively-Coupled Argon Plasma (ICP) Atomic Emission Spectrometer*—The instrument is equipped with an argon-plasma source, a sample transport system for introducing the test sample and calibration solutions into the plasma. The monochromator or polychromator must be capable of isolating the required wavelengths shown in Table 1 for measurement of their intensities by a linear photometer. Multielement programmed analysis including automatic data acquisition and computer-controlled calibration and analysis calculations 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.

⁴ Annual Book of ASTM Standards, Vol 3.05.

⁵ Annual Book of ASTM Standards, Vol 03.06.

TABLE 1 Wavelengths and Instrument Conditions^A

Element	Wavelength	Time, s	No. Integ.	B _{Cor1}	B _{Cor2}
Al	3092.7	1.0	3
Cd	2265.02	.5	3	2264.46	2265.58
Ce	4186.6	.5	2
Fe	2599.4	.5	2
La	3988.5	.5	2	3987.54	3989.06
Pb	2832.97	1.0	3	...	2833.36

^A The tabulated conditions were those found satisfactory on one instrument. Wavelengths are expressed in angstroms. Time = seconds for each integration, No. Integ. = number of integrations averaged for each reading, and B_{Cor1} and B_{Cor2} are off-peak background correction wavelengths.

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 must 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 concentrations as is consistent with stable instrument readings. If there is a question of linearity of the instrument's response over the range of solution concentrations given, a third standard, equidistant between the two listed standards, must be measured to verify linearity.

6. Reagents

6.1 *Purity of Reagents*—Unless otherwise indicated, all reagents used in this test method shall conform to the "Reagent Grade" Specifications of the American Chemical Society.⁶ Other chemicals may be used provided that it is first ascertained that the reagent used is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

6.3 *Aluminum, Standard Solution* (1 mL = 20.0 mg Al)—Transfer 2.0000 g of aluminum (purity: 99.999 % min) 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.

⁶ *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 *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

6.4 *Boric Acid Solution* (3 g/100 mL)—Dissolve 3.0 g of boric acid (H₃BO₃) in 100 mL of boiling water.

6.5 *Cadmium, Standard Solution* (1 mL = 1.00 mg Cd)—Transfer 1.000 g of cadmium (purity: 99.95 % min) to a 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 volumetric flask, add 240 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

6.6 *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 % min) 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 HNO₃, dilute to volume, and mix. Store in a polyethylene bottle.

6.7 *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.

6.8 *Dilution Solution*—Half fill a 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.

6.9 *Iron, Standard Solution A* (1 mL = 1.00 mg Fe)—Transfer 1.000 g of iron (purity: 99.95 % min) 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 HNO₃, dilute to volume, and mix. Store in the polyethylene bottle.

6.10 *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.

6.11 *Lanthanum, Standard Solution A* (1 mL = 0.010 mg La)—Ignite lanthanum oxide (La₂O₃) (purity: 99.9 % min) 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 HNO₃, dilute to volume, and mix. Store in a polyethylene bottle.

6.12 *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.

6.13 *Lead, Standard Solution* (1 mL = 1.00 mg Pb)—Transfer 1.000 g of lead (purity: 99.9 % min) to a 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 volumetric flask, add 250 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

6.14 *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 HNO₃. When all solids have dissolved, remove from the heat and allow to cool.