Designation: E1277 – 14

Standard Test Method for Analysis of Zinc-5 % Aluminum-Mischmetal Alloys by ICP Emission Spectrometry[1]

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 (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	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.

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E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E876 Practice for Use of Statistics in the Evaluation of Spectrometric Data (Withdrawn 2003)[3]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

[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.

Current edition approved Nov. 1, 2014. Published January 2015. Originally approved in 1991. Last previous edition approved in 2008 as E1277 – 08. DOI: 10.1520/E1277-14.

[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.

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.

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.[5] 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_3 (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 ($(NH_4)_2Ce(NO_3)_6$, also known as ammonium hexanitrato 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_3, 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_3, 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_3, 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_2O_3) (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_3, 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_3 (1 + 1). Boil gently to complete dissolution and to remove oxides of

[5] 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.