Designation: E536 – 16

Standard Test Methods for Chemical Analysis of Zinc and Zinc Alloys[1]

This standard is issued under the fixed designation E536; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the chemical analysis of zinc and zinc alloys having chemical compositions within the limits of Table 1.

TABLE 1 Scope of Mass Fraction Ranges for Zinc and Zinc Alloys

Element	Composition Range, %
Aluminum	0.005 to 4.5
Cadmium	0.001 to 0.5
Copper	0.001 to 1.3
Iron	0.001 to 0.1
Lead	0.001 to 1.6
Magnesium	0.001 to 0.1
Tin	0.001 to 0.1

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 These test methods appear as follows:

	Sections
Aluminum by the EDTA Titrimetric Method (0.5 to 4.5 %)	10 – 17
Aluminum, Cadmium, Copper, Iron, Lead, and Magnesium by the Atomic Absorption Method	18 – 28

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 precautionary statements are given in Section 6.

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

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry

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E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

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

3. Terminology

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

4. Significance and Use

4.1 These test methods for the chemical analysis of zinc metals and alloys are primarily intended as referee methods to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods 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 and Reagents

5.1 Apparatus and reagents required for each determination are listed in separate sections of each test method. The apparatus, standard solutions, and reagents shall conform to the requirements prescribed in Practices E50. Spectrometers shall conform to the requirements prescribed in Practice E60.

[1] These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are 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] 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.

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6. Safety Hazards

6.1 For precautions to be observed in the use of certain reagents in these test methods, refer to Practices E50.

7. Sampling

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

8. Rounding Calculated Values

8.1 Calculated values shall be rounded to the desired number of places as directed in Practice E29, Rounding Method.

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E173, unless otherwise noted in the precision section.

ALUMINUM BY THE EDTA TITRIMETRIC METHOD

10. Scope

10.1 This test method covers the determination of aluminum in compositions from 0.5 % to 4.5 %.

11. Summary of Test Method

11.1 After dissolution of the sample in HCl, the solution is buffered and disodium (ethylenedinitrilo) tetraacetate (EDTA) is added. The excess EDTA is titrated with standard zinc solution. Sodium fluoride is added to decompose the aluminum-EDTA complex, and the released EDTA is titrated with standard zinc solution.

12. Interferences

12.1 The elements ordinarily present do not interfere if their compositions are under the maximum limits shown in 1.1.

13. Apparatus

13.1 *Magnetic Stirrer,* with stirring bar covered with tetrafluoroethylene polymer (TFE-fluorocarbon).

14. Reagents

14.1 *Bromcresol Green Indicator Solution* (0.4 g/L)—Dissolve 0.04 g of bromcresol green in 6 mL of 0.01 *N* sodium hydroxide (NaOH) solution and dilute to 100 mL.

14.2 *EDTA Solution* (90 g/L)—Dissolve 90.0 g of disodium (ethylenedinitrilo) tetraacetate dihydrate in about 800 mL of warm water. Cool and dilute to 1 L.

NOTE 1—Although it is not critical that this solution be prepared with a 1 L volumetric, doing so makes it more consistent and easier for the analyst run to run.

14.3 *Methyl Red Indicator Solution* (0.4 g/L)—Dissolve 0.1 g of methyl red in 3.72 mL of 0.1 N NaOH solution and dilute to 250 mL with water. Filter if necessary.

14.4 *Sodium Acetate Buffer Solution* (320 g/L)—Dissolve 320 g of sodium acetate trihydrate in about 800 mL of water and filter. Using a pH meter, adjust the pH of the solution to 5.5 ± 0.1 with NaOH solution or acetic acid and dilute to 1 L.

NOTE 2—The analyst is not restricted to using the 0.1 N solution of NaOH

14.5 *Sodium Fluoride Solution (Saturated)*—Dissolve 60 g of sodium fluoride (NaF) in 1 L of boiling water. Cool and filter through a coarse paper. Store in a polyethylene bottle.

14.6 *Xylenol Orange Indicator Solution* (10 g/L)—Dissolve 0.250 g of xylenol orange in 25 mL of water. Do not use a solution that has stood more than 1 month.

14.7 *Zinc Standard Solution* (1 mL = 1.00 mg Al)—Dissolve 2.423 g of zinc metal (purity: 99.99 % minimum) in 20 mL of HCl. Dilute to 100 mL. Add 3 drops of methyl red solution and neutralize with NH_4OH. Add HCl until the color changes to red. Transfer to a 1-L volumetric flask, dilute to volume, and mix.

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

15. Procedure

15.1 Select and weigh a sample to the nearest 1 mg, in accordance with Table 2.

Transfer the sample to a 400-mL beaker, and cover.

15.2 Add 100 mL of HCl (1 + 1). Heat until dissolution is complete and boil for 2 minutes to 3 minutes. If a residue remains, add 1 mL of H_2O_2 and boil the solution for at least 5 minutes to destroy excess H_2O_2 and expel free chlorine.

NOTE 3—Excess peroxide and free chlorine shall be removed to prevent fading of the indicators.

15.3 Transfer the solution to a 200-mL volumetric flask, dilute to volume, and mix.

15.4 Using a pipet, transfer the aliquot specified in 15.1 to a 500-mL wide-mouth Erlenmeyer flask.

15.5 Add the volume of EDTA solution specified in 15.1 and dilute to 200 mL.

NOTE 4—The amount of EDTA added shall be sufficient to complex the zinc and aluminum with some excess. The amount of EDTA required is 5.7 mg for each milligram of zinc and 14.0 mg for each milligram of aluminum.

15.6 Add five drops or six drops of methyl red solution. Add NH_4OH until the color changes to orange.

15.7 Add 25 mL of sodium acetate buffer solution and boil for 3 minutes to 5 minutes. Cool in a water bath.

15.8 Add four drops of xylenol orange solution and five drops or six drops of bromcresol green solution.

15.9 Using a TFE-fluorocarbon-covered stirring bar and a magnetic stirrer, stir the solution while adding standard zinc solution from a 50-mL buret to complex the excess EDTA. Add the solution dropwise as the end point is approached. Continue the titration until the color changes from green to red. Refill the buret.

TABLE 2 Recommended Sample Weight

Aluminum, %	Sample Weight, g	Aliquot, mL	EDTA Addition, mL
0.5 to 1.5	10.0	50	165 to 168
1.5 to 2.5	6.0	50	103 to 106
2.5 to 4.5	5.0	40	72 to 75