



Designation: E536 – 16

Standard Test Methods for Chemical Analysis of Zinc and Zinc Alloys¹

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](#)

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.

Current edition approved Feb. 1, 2016. Published March 2016. Originally approved in 1975. Last previous edition approved in 2015 as E536 – 15. DOI: 10.1520/E0536-16.

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.

- [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](#).

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

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 NH₄OH. 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 H₂O₂ and boil the solution for at least 5 minutes to destroy excess H₂O₂ 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 NH₄OH 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

15.10 Add 25 mL of NaF solution and boil for 3 minutes to 5 minutes. Cool in a water bath.

15.11 Titrate with standard zinc solution as directed in 15.9 and record the volume to the nearest 0.01 mL.

16. Calculation

16.1 Calculate the percentage of aluminum as follows:

$$\text{Aluminum, \%} = (AB/C) \times 100 \quad (1)$$

where:

- A = standard zinc solution used in 15.11, mL;
- B = aluminum equivalent of the standard zinc solution, g/mL (noted in 14.7); and
- C = sample represented in the aliquot taken in 15.1, g.

17. Precision and Bias

17.1 *Precision*—Eight laboratories cooperated in testing this test method and obtained the results summarized in Table 3.

17.2 *Bias*—No information concerning the accuracy of this test method is available because certified reference materials suitable for chemical test methods were not available when the interlaboratory test was performed. The analyst is urged to use an accepted reference material, if available, to determine that the accuracy of results is satisfactory.

17.3 Practice E173 has been replaced by Practice E1601. The reproducibility Index R_2 , corresponds to the Reproducibility Index R of Practice E1601. Likewise the Repeatability Index R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

ALUMINUM, CADMIUM, COPPER, IRON, LEAD, AND MAGNESIUM BY THE ATOMIC ABSORPTION METHOD

18. Scope

18.1 This test method covers the determination of aluminum in compositions from 0.002 % to 0.5 %, cadmium from 0.001 % to 0.5 %, copper from 0.001 % to 1.3 %, iron from 0.003 % to 0.1 %, lead from 0.002 % to 1.6 %, and magnesium from 0.001 % to 0.1 %.

19. Summary of Test Method

19.1 HCl solution of the sample is aspirated into the flame of an atomic absorption spectrometer. The absorption of the resonance line energy from the spectrum of each element is measured and compared with that of calibration solutions of the same element. The wavelengths of the spectral lines and other method parameters are tabulated in 22.1 for each element.

TABLE 3 Statistical Information

Test Specimen	Aluminum Found, %	Repeatability (R_1 , E173)	Reproducibility (R_2 , E173)
G-3	0.3998	0.0190	0.0254
H-2	4.04	0.052	0.074

20. Composition Range

20.1 The composition range for each element shall be determined experimentally, because the optimum range will depend upon the individual instrument. If the optimum composition range and instrument parameters have been determined, proceed in accordance with Section 26; otherwise, determine the composition range in accordance with Section 22.

NOTE 5—The composition range will depend on the instrument. Section 24.1 lists the typical calibration composition ranges that most often will be acceptable, however this can be determined by using the criteria in Section 22. The analyst may find that different calibration composition ranges are required and must be adjusted accordingly to suit their instrument.

21. Interferences

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

22. Apparatus

22.1 *Atomic Absorption Spectrometer*, equipped with a pre-mix burner, with facilities for using the oxidizer-fuel combinations listed in Table 4. Use hollow-cathode lamps operated in accordance with manufacturer's recommendations as sources for the spectral lines. The instrument may be considered suitable for this test method if a composition range can be found for which the minimum response, calibration variability, and reference variability tabulated in Table 4 can be met.

22.1.1 Prepare the dilute standard solution, reference, and calibration solutions in accordance with Section 24. Refer to Table 5 for suggested initial compositions.

22.1.2 Prepare the instrument for use in accordance with in 26.1. Measure the instrument response while aspirating the reference solution, the lowest, and the two highest calibration solutions, performing the measurements in accordance with 26.2.2 and 26.2.3.

22.1.3 *Minimum Response*—Calculate the difference between the readings of the two highest of the five equally spaced calibration solutions. This difference shall be equal to or greater than the number of scale units specified in Table 4. For purposes of this test method, the scale unit is defined as one in

TABLE 4 Minimum Response, Calibration, and Reference Variability

Element	Spectral Line, nm	Oxidizer-Fuel	Standard Solution	Minimum Response, Units	Calibration Variability, %	Reference Variability, %
Aluminum	309.2	N ₂ O–C ₂ H ₂	"A"	9	3.5	2.0
			"B"	25	1.0	0.5
Cadmium	228.8	Air–C ₂ H ₂	...	50	0.8	0.4
Copper	324.7	Air–C ₂ H ₂	"A"	15	2.0	0.8
			"B"	50	0.8	0.4
Iron	248.3	Air–C ₂ H ₂	"A"	15	2.0	1.0
			"B"	25	1.0	0.5
Lead	217.0	Air–C ₂ H ₂	"A"	10	2.0	1.5
			"B"	30	1.0	0.4
Magnesium	285.2	N ₂ O–C ₂ H ₂	...	20	1.5	0.8