



# Standard Test Methods for Chemical Analysis of Zinc and Zinc Alloys<sup>1</sup>

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 ( $\epsilon$ ) 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:

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

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:*<sup>2</sup>

[D1193 Specification for Reagent Water](#)

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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 \(Withdrawn 1998\)](#)<sup>3</sup>
- [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](#).

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://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 NH<sub>4</sub>OH. 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<sub>2</sub>O<sub>2</sub> and boil the solution for at least 5 minutes to destroy excess H<sub>2</sub>O<sub>2</sub> 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<sub>4</sub>OH 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 <sub>2</sub> O–C <sub>2</sub> H <sub>2</sub>	"A"	9	3.5	2.0
			"B"	25	1.0	0.5
Cadmium	228.8	Air–C <sub>2</sub> H <sub>2</sub>	...	50	0.8	0.4
Copper	324.7	Air–C <sub>2</sub> H <sub>2</sub>	"A"	15	2.0	0.8
			"B"	50	0.8	0.4
Iron	248.3	Air–C <sub>2</sub> H <sub>2</sub>	"A"	15	2.0	1.0
			"B"	25	1.0	0.5
Lead	217.0	Air–C <sub>2</sub> H <sub>2</sub>	"A"	10	2.0	1.5
			"B"	30	1.0	0.4
Magnesium	285.2	N <sub>2</sub> O–C <sub>2</sub> H <sub>2</sub>	...	20	1.5	0.8

**TABLE 5 Dilute Standard Solutions**

Dilute Standard Solution	Composition Range, %	Volume, mL	Concentration of Dilute Standard Solution, mg/mL
Aluminum "A"	0.004 to 0.020	5	0.05
Aluminum "B"	0.020 to 0.50	20	0.20
Cadmium	0.001 to 0.50	2	0.020
Copper "A"	0.001 to 0.005	2	0.020
Copper "B"	0.005 to 2.5	10	0.10
Iron "A"	0.001 to 0.005	2	0.020
Iron "B"	0.005 to 0.10	10	0.10
Lead "A"	0.001 to 0.005	2	0.020
Lead "B"	0.005 to 2.5	10	0.10
Magnesium	0.001 to 0.10	4	0.004

the least significant digit of the scale reading of the most concentrated calibration solution.

22.1.4 *Curve Linearity*—Calculate the difference between the scale readings of the reference solution and the lowest of the five equally spaced calibration solutions. If necessary, convert this difference and the difference calculated in 22.1.3 to absorbance units. Divide the difference for the highest interval by that for the lowest interval. This ratio shall be equal to or greater than 0.70.

22.1.5 If the instrument meets or surpasses the minimum response and curve linearity criteria, the initial composition range may be considered suitable. In this case, proceed in accordance with 22.1.7; otherwise, proceed as follows:

22.1.6 If the minimum response is not achieved, prepare another dilute standard solution to provide a higher composition range, and repeat 22.1.1 – 22.1.4. If the calibration curve does not meet the linearity criterion, prepare another dilute standard solution to provide a lower composition range, and repeat 22.1.1 – 22.1.4. If a composition range cannot be found for which both criteria can be met, do not use this test method until the performance of the apparatus satisfies the requirements.

22.1.7 *Instrument Stability*—Calculate the calibration variability and reference variability as follows:

$$V_c = \frac{100}{\bar{c}} \left( \frac{\sum (c - \bar{c})^2}{n - 1} \right)^{\frac{1}{2}} \quad (2)$$

$$V_o = \frac{100}{\bar{o}} \left( \frac{\sum (o - \bar{o})^2}{n - 1} \right)^{\frac{1}{2}} \quad (3)$$

where:

- $V_c$  = calibration variability,
- $\bar{c}$  = average absorbance value for the highest calibration solution,
- $C$  = individual absorbance readings on the highest calibration solution,
- $\sum (c - \bar{c})^2$  = sum of the squares of the  $n$  differences between the absorbance readings on the highest calibration solution and their average,
- $V_o$  = reference variability relative to  $\bar{c}$ ,
- $\bar{o}$  = average absorbance value for the reference solution,
- $o$  = individual absorbance readings on the reference solution,

$\sum (o - \bar{o})^2$  = sum of the squares of the  $n$  differences between the absorbance readings on the reference solution and their average, and

$n$  = number of readings taken on each solution.

22.1.8 If the variability of the readings of the highest calibration and the reference solutions are not equal to or smaller than the values specified in Table 4, the stability of the instrument shall be improved before this test method may be used.

## 23. Reagents

23.1 *Aluminum, Standard Solution* (1 mL = 1.00 mg Al)—Transfer 1 g of aluminum (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 50 mL of HCl (1 + 1). After the reaction has subsided, add 1 mL of H<sub>2</sub>O<sub>2</sub> and boil to complete dissolution. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

23.2 *Cadmium, Standard Solution* (1 mL = 1 mg Cd)—Transfer 1 g of cadmium (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 25 mL of HNO<sub>3</sub> (1 + 1) and 5 mL of HCl. Boil gently to complete dissolution and to remove oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

23.3 *Copper, Standard Solution* (1 mL = 1 mg Cu)—Transfer 1 g of copper (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 25 mL of HNO<sub>3</sub> (1 + 1) and 5 mL of HCl. Boil gently to complete dissolution and to remove the oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

23.4 *Iron, Standard Solution* (1 mL = 1 mg Fe)—Transfer 1 g of iron (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 50 mL of HCl (1 + 1). Boil gently to complete dissolution. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

23.5 *Lead, Standard Solution* (1 mL = 1 mg Pb)—Transfer 1 g of lead (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 50 mL of HNO<sub>3</sub> (1 + 1). Boil gently to complete dissolution and to remove oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HNO<sub>3</sub>, dilute to volume, and mix. Store in a polyethylene bottle.

23.6 *Magnesium, Standard Solution* (1 mL = 0.1 mg Mg)—Transfer 0.1 g of magnesium (purity: 99.95 % minimum) to a 250-mL beaker, cover, and add 25 mL of HCl (1 + 1). Boil gently to complete dissolution. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

23.7 *Sodium Chloride Solution* (100 g/L)—Dissolve 100 g of NaCl in 1 L of water.

23.8 *Zinc, Purified Stock Solution* (1 mL = 0.2 g Zn)—Transfer 200 g of zinc (purity: 99.9 % minimum) to a 2-L beaker. Add 100 mL of water and 700 mL of HCl in small amounts to control the rate of reaction. Add 1 mL of H<sub>2</sub>O<sub>2</sub>, and

evaporate carefully to a syrupy consistency. Dilute to about 800 mL with water, add 30 g of zinc powder (purity: 99.9 % minimum), and let it react at least 30 min, stirring vigorously several times during the reaction period. Dilute to 1 L with water and filter into a polyethylene bottle. Discard the residue without washing.

NOTE 6—For the determination of aluminum and magnesium in the lowest 10 % of the ranges listed in 18.1, the zinc used shall contain less than 0.0002 % Al and 0.0001 % of Mg for samples that contain less than 0.05 % Al and 0.01 % Mg respectively.

23.9 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or II of Specification D1193. Type III or IV may be used if they effect no measurable change in the blank or sample.

## 24. Calibration

24.1 *Dilute Standard Solution*—Using pipets, transfer the volume of the appropriate standard solution to a 100-mL volumetric flask in order to obtain the desired concentration of dilute standard solution. Dilute to volume and mix. Use Table 5 as a guide, unless previous experience has shown a different concentration range to be optimum.

24.2 *Calibration Solutions*—Prepare five calibration solutions for each element to be determined in the range required. Using pipets, transfer (5, 10, 15, 20, and 25)-mL portions of the appropriate dilute standard solution to 100-mL volumetric flasks, and provide another 100-ml volumetric flask for the reference solution described in 24.3. Using pipets, add appropriate quantities of NaCl solution, zinc solution, and HCl solution to ensure that the calibration and reference solutions approximate the composition of the test solution. Dilute to volume and mix. Use Table 6 as a guide.

24.3 *Reference Solution*—Prepare a reference solution containing the same volumes of reagents and zinc solution as the calibration solutions but without addition of the dilute standard solution.

## 25. Procedure

### 25.1 *Test Solution:*

25.1.1 Transfer a 10-g sample, weighed to the nearest 10 mg, to a 400-mL beaker, except as follows: use a 2.5-g sample for determining copper and lead in compositions greater than 0.5 %. Cover and add 50 mL of HCl in small increments to control the rate of reaction. Heat gently to complete the dissolution. Add 0.5 mL of H<sub>2</sub>O<sub>2</sub> and boil gently for 5 min. Cool, transfer to a 100-mL volumetric flask, dilute to volume, and mix.

NOTE 7—For aluminum in the range from 0.002 % to 0.020 % only, add 5 mL of NaCl solution before diluting to volume and omit 25.1.2.

25.1.2 Using a pipet, transfer an appropriate volume of the sample solution listed in Table 7 to a 100-mL volumetric flask, dilute to volume, and mix.

## 26. Measurement

### 26.1 *Instrument Adjustment:*

**TABLE 6 Calibration Solutions**

Element	Compo- sition Range, <sup>A</sup> %	NaCl Solution, mL	Dilute Standard Solution <sup>B</sup>	Zinc Solution, mL	HCl, mL
Aluminum	0.002 to 0.020	5	"A"	25	10
	0.020 to 0.10	5	"B"	25	10
	0.10 to 0.50	5	"B"	5	...
Cadmium	0.001 to 0.005	...	Cadmium	50	20
	0.005 to 0.025	...	Cadmium	10	5
	0.025 to 0.125	...	Cadmium	2	...
	0.10 to 0.50	...	Cadmium	...	...
Copper, Iron, or Lead	0.001 to 0.005	...	"A"	50	20
	0.005 to 0.025	...	"B"	50	20
	0.025 to 0.125	...	"B"	10	5
	0.125 to 0.625	...	"B"	2	...
	0.50 to 2.5	...	"B"	...	...
Magnesium	0.001 to 0.005	5	Magnesium	10	5
	0.005 to 0.025	5	Magnesium	2	...
	0.020 to 0.10	5	Magnesium	...	...

<sup>A</sup> These values are based upon dilute standard solutions prepared in accordance with the values in 24.1. If higher or lower concentrations are used for the dilute standards, the concentration ranges covered by the calibration solution will be changed accordingly.

<sup>B</sup> The "A" or "B" designates the A or B dilute standard solution of the appropriate element.

**TABLE 7 Recommended Test Solutions**

Element	Composition Range, %	Sample Solution, mL/100 mL	NaCl Solution, mL
Aluminum	0.002 to 0.020	100 <sup>A</sup>	5
	0.020 to 0.10	50	5
	0.10 to 0.50	10	5
Cadmium	0.001 to 0.005	100 <sup>B</sup>	...
	0.005 to 0.025	20	...
	0.025 to 0.125	4	...
	0.10 to 0.50	1	...
Copper, Iron, and Lead	0.001 to 0.005	100 <sup>B</sup>	...
	0.005 to 0.025	100	...
	0.025 to 0.125	20	...
	0.125 to 0.625	4	...
	0.50 to 1.6 <sup>C</sup>	4	...
Magnesium	0.001 to 0.005	20	5
	0.005 to 0.025	4	5
	0.020 to 0.10	1	5

<sup>A</sup> See Note 6.

<sup>B</sup> The test solution is the undiluted sample solution.

<sup>C</sup> For lead and copper above 0.5 %, use a 2.5-g sample.

26.1.1 Set the instrument parameters approximately to those known to be optimum for the element to be determined. Light the burner.

**TABLE 8 Statistical Information**

Specimen		Average, %	Repeatability, ( $R_1$ , E173)	Reproducibility, ( $R_2$ , E173)
Al	G-1	0.0015	0.0006	0.0011
	G-3	0.401	0.035	0.066
Cd	H-2	0.0008	0.0003	0.0003
	G-5	0.545	0.025	0.038
Cu	G-3	0.0015	0.0003	0.0005
	H-2	1.154	0.09	0.12
Fe	G-1	0.0026	0.0006	0.0017
	H-3	0.113	0.012	0.039
Pb	H-2	0.0016	0.0003	0.0011
	G-5	1.368	0.12	0.15
Mg	G-3	0.0010	0.0003	0.0005
	H-2	0.055	0.009	0.010

26.1.2 Adjust the instrument to the approximate wavelength for the element to be determined and aspirate water until thermal equilibrium is established. Aspirate the highest calibration solution, and adjust the wavelength to obtain maximum response.

26.1.3 Optimize fuel, air, and burner adjustments while aspirating the highest calibration solution.

26.1.4 Aspirate water long enough to establish that the instrument reading is stable, and then set the initial reading (approximately zero absorbance or 100 % transmittance). Verify that the reference solution gives readings that are above zero absorbance (less than 100 % transmittance).

#### 26.2 Spectrometry:

26.2.1 Aspirate the test solution and note, but do not record the reading.

26.2.2 Aspirate water until the initial reading is again obtained. Aspirate the reference, calibration, and test solutions in order of increasing instrument response. Record the reading when a stable response is obtained from each solution.

26.2.3 Proceed as directed in 26.2.2 at least two more times to obtain a minimum of three readings for each solution.

### 27. Calculation

27.1 Calculate the variability of the readings for the highest calibration and reference solutions as directed in 22.1.7. If the calculated values are not equal to or less than the maximum values given in Table 4 for the element being determined,

disregard the data, readjust the instrument, and proceed again in accordance with 26.2.

27.2 If necessary, convert the average of the readings of each of the calibration solutions and the test solution to absorbance.

27.3 Prepare a calibration curve by plotting the absorbance values for the calibration solutions against milligrams of the element per 100 millilitres.

27.4 Convert the absorbance value of the test solution to milligrams by means of the calibration curve.

27.5 Calculate the percentage of the element as follows:

$$\text{Element, \%} = \frac{A}{B} \times 100 \quad (4)$$

where:

A = element in 100 mL of the test solution, mg, and  
B = sample in 100 mL of the test solution, mg.

### 28. Precision and Bias<sup>4</sup>

28.1 *Precision*—Six laboratories cooperated in testing this test method and obtained eight sets of data which are summarized in Table 8.

28.2 *Bias*—No information on the accuracy of this test method is available because no certified reference materials suitable for chemical test methods were available when the interlaboratory test was performed. The analyst is urged to use a certified reference material, if available, to determine that the accuracy of results is satisfactory.

28.3 Practice E173 has been replaced by Practice E1601. The Reproducibility Index  $R_2$  of Practice E173 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.

### 29. Keywords

29.1 aluminum; atomic absorption; cadmium; copper; iron; lead; magnesium; zinc; zinc alloys

<sup>4</sup> Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1069. Contact ASTM Customer Service at service@astm.org.

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