Designation: E34 – 11

Standard Test Methods for
Chemical Analysis of Aluminum and Aluminum-Base Alloys[1]

This standard is issued under the fixed designation E34; 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 Department of Defense.

1. Scope

1.1 These test methods cover the chemical analysis of aluminum and aluminum-base alloys having compositions within the following limits:

Beryllium, ppm	0.3 to 100
Bismuth, %	0.02 to 1.0
Boron, %	0.005 to 0.060
Cadmium, %	0.001 to 0.50
Chromium, %	0.01 to 1.0
Copper, %	0.01 to 20.0
Gallium, %	0.001 to 0.05
Iron, %	0.01 to 3.0
Lead, %	0.01 to 1.0
Lithium, %	0.001 to 4.0
Magnesium, %	0.002 to 12.0
Manganese, %	0.005 to 2.0
Nickel, %	0.01 to 4.0
Silicon, %	0.05 to 20.0
Tin, %	0.03 to 1.0
Titanium, %	0.002 to 0.30
Vanadium, %	0.002 to 0.16
Zinc, %	0.003 to 12.0
Zirconium, %	0.01 to 0.30

1.2 The analytical procedures appear in the following sections:

Procedure	Sections
Beryllium:	
Beryllium by Argon Plasma Optical Emission Spectroscopy	283 to 292
Beryllium by the Morin (Fluorometric) Test Method	[1e]
Bismuth:	
Bismuth by the Thiourea (Photometric) Method	[1a]
Bismuth and Lead by the Atomic Absorption Test Method	188 to 198
Boron:	
Boron by the Carmine (Photometric) Test Method	[1e]
Cadmium:	
Cadmium by the Atomic Absorption Test Method	167 to 177
Chromium:	
Chromium by the Diphenylcarbazide (Photometric) Test Method	[1e]
Chromium by the Persulfate Oxidation (Titrimetric) Test Method	[1b]
Chromium by the Atomic Absorption Test Method	199 to 209
Copper:	
Copper and Lead by the Electrolytic (Gravimetric) Test Method	[1c]
Copper and Zinc by the Atomic Absorption Spectrometry Test Method	210 to 220
Copper by the Electrolytic (Gravimetric) Test Method	303 to 311
Copper by the Neocuproine (Photometric) Test Method	[1a]
Gallium:	
Gallium by the Ion Exchange-Atomic Absorption Test Method	312 to 323
Iron:	
Iron by the 1,10-Phenanthroline (Photometric) Method	73 to 81
Iron and Manganese by the Atomic Absorption Spectrometry Method	221 to 231
Lead:	
Copper and Lead by the Electrolytic (Gravimetric) Test Method	[1c]
Bismuth and Lead by the Atomic Absorption Spectrometry Test Method	188 to 198
Lithium:	
Lithium by the Atomic Absorption Test Method	324 to 334
Magnesium:	
Magnesium by the Pyrophosphate (Gravimetric) Method	[1b]
Magnesium by the Ethylenediamine Tetraacetate (Titrimetric) Test Method	[1e]
Magnesium by the Atomic Absorption Spectrometry Test Method	232 to 242
Manganese:	
Iron and Manganese by the Atomic Absorption Spectrometry Test Method	221 to 231
Manganese by the Periodate (Photometric) Test Method	293 to 302
Nickel:	
Nickel by the Dimethylglyoxime (Photometric) Test Method	[1a]
Nickel by the Dimethylglyoxime (Gravimetric) Test Method	[1b]
Nickel by the Atomic Absorption Spectrometry Test Method	243 to 253
Silicon:	

---

[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 E01.04 on Aluminum and Magnesium.

Current edition approved July 1, 2011. Published August 2011. Originally published as E34 – 60 T. Last previous edition E34 – 94 (Reapproved 2002).

[1a] Discontinued as of Feb. 25, 1983.

[1b] Discontinued as of May 29, 1981.

[1c] Discontinued as of Oct. 25, 1985.

[1d] Discontinued as of March 25, 1983. DOI: 10.1520/E0034-11.

[1e] Discontinued as of July 1, 2011. DOI: 10.1520/E0034-11.

Silicon by the Molybdisilicic Acid (Photometric) Test Method	[1e]
Silicon by the Sodium Hydroxide-Perchloric Acid (Gravimetric) Method	[1e]
Tin:	
Tin by the Iodate (Titrimetric) Test Method	[1e]
Titanium:	
Titanium by the Chromotropic Acid (Photometric) Test Method	141 to 150
Titanium by the Diantipyrylmethane Photometric Test Method	254 to 263
Vanadium:	
Vanadium by an Extraction-Photometric Test Method using *N*-Benzoyl-*N*-Phenylhydroxylamine	264 to 273
Zinc:	
Zinc by the Ammonium Mercuric Thiocyanate or the Zinc Oxide (Gravimetric) Test Method	[1b]
Zinc by the Ethylenediamine Tetraacetate (Titrimetric) Test Method	[1d]
Copper and Zinc by the Atomic Absorption Spectrometry Test Method	210 to 220
Zinc by the Ion Exchange-EDTA Titrimetric Test Method	274 to 282
Zirconium:	
Zirconium by the Arsenazo III (Photometric) Method	178 to 187

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety problems, 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 hazard statements are given throughout these test methods.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications [3]

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials [3]

E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition [3]

E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry [3]

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[3] [4]

E716 Practices for Sampling and Sample Preparation of Aluminum and Aluminum Alloys for Determination of Chemical Composition by Spectrochemical Analysis [4]

E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry[5]

E1479 Practice for Describing and Specifying Inductively-Coupled Plasma Atomic Emission Spectrometers

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

3. Terminology

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

4. Significance and Use

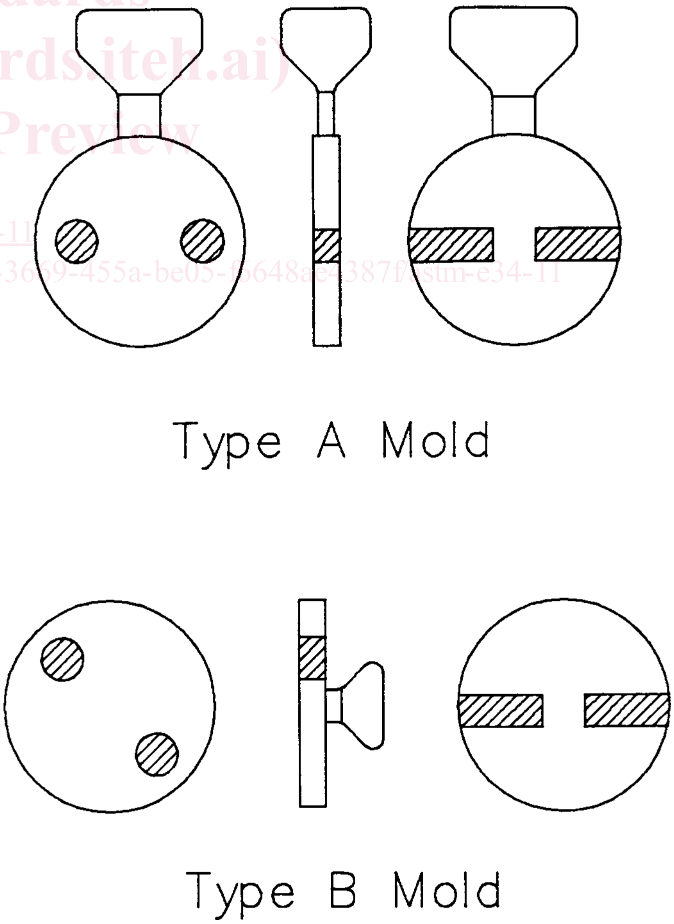
4.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

5. Apparatus, Reagents, and Photometric Practice

5.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure.

5.2 Photometric practice prescribed in these test methods shall conform to Practice E60.

5.3 Calculated values shall be rounded to the desired number of places in accordance with the rounding method of Practice E29.



NOTE 1—Shaded areas are suitable for sampling.

FIG. 1 Type A and Type B Disks

---

[2] *Annual Book of ASTM Standards*, Vol 14.02.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

6. Precautions

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

7. Sampling

7.1 Wrought products shall be sampled in accordance with Practice E55. Cast products shall be sampled in accordance with Practice E88.

7.2 Chill cast disks produced for analysis by spectrochemical methods (see Practices E716) shall be sampled by drilling or milling through the entire thickness. Drill bits or milling cutters should be carbide to avoid iron contamination.

NOTE 1—The use of a machined disk may result in the exclusion of an element-rich portion of the sample. This practice should be avoided wherever possible, especially for analyses affecting product acceptance.

7.2.1 If samples are produced by drilling, use a minimum of two positions approximately opposite each other and combine the drillings.

7.2.2 The outer edges of the holes shall be approximately 0.48 cm (3⁄16 in.) from the edge of the disk. Drill bits shall be not less than 0.95 cm (3⁄8 in.) in diameter and not larger than 1.27 cm (1⁄2 in.) in diameter.[6]

7.2.3 If samples are produced by milling, mill disks at similar points to a distance of 40 % of the sample diameter or other methods that provide a representative sample such as quarter of half milling. A 0.95-cm (3⁄8 in.) milling cutter has been shown to provide acceptable chips.[6]

7.2.4 Center pour (Type B, Practices E716) and vacuum cast disks may be sampled around the entire circumference. Fig. 1 illustrates the areas suitable for sampling Type B disks. Vacuum cast disks are sampled in the same manner as Type B disks.[6]

7.2.5 Drilling or milling techniques ideally should produce uniformly small chips. Break large continuous pieces into smaller pieces 0.64 cm (1⁄4 in.) to 0.95 cm (3⁄8 in.) long. Drilling or milling techniques should minimize production of fine, dust-like material.[6]

BERYLLIUM BY THE MORIN (FLUOROMETRIC) TEST METHOD

(This test method, which consisted of Sections 8 through 19 of this standard, was discontinued in 2008.)

BISMUTH BY THE THIOUREA (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 20 through 29 of this standard, was discontinued in 1983.)

BORON BY THE CARMINE (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 30 through 38 of this standard, was discontinued in 2008.)

CHROMIUM BY THE DIPHENYLCARBAZIDE (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 39 through 47 of this standard, was discontinued in 2008.)

CHROMIUM BY THE PERSULFATE OXIDATION (TITRIMETRIC) TEST METHOD

(This test method, which consisted of Sections 48 through 53 of this standard, was discontinued in 1981.)

COPPER BY THE NEOCUPROINE (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 54 through 63 of this standard, was discontinued in 1983.)

COPPER AND LEAD BY THE ELECTROLYTIC (GRAVIMETRIC) TEST METHOD

(This test method, which consisted of Sections 64 through 72 of this standard, was discontinued in 1985.)

IRON BY THE 1,10-PHENANTHROLINE (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 73 through 81 of this standard, was discontinued in 2008.)

MAGNESIUM BY THE PYROPHOSPHATE (GRAVIMETRIC) TEST METHOD

(This test method, which consisted of Sections 82 through 87 of this standard, was discontinued in 1981.)

MAGNESIUM BY THE ETHYLENEDIAMINE TETRAACETATE (TITRIMETRIC) TEST METHOD

(This test method, which consisted of Sections 88 through 93 of this standard, was discontinued in 2008.)

MANGANESE BY THE PERIODATE (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 94 through 102 of this standard, was replaced in 1984 by Sections 293 through 302.)

NICKEL BY THE DIMETHYLGLYOXIME (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 103 through 111 of this standard, was discontinued in 1983.)

NICKEL BY THE DIMETHYLGLYOXIME (GRAVIMETRIC) TEST METHOD

(This test method, which consisted of Sections 112 through 117 of this standard, was discontinued in 1981.)

SILICON BY THE MOLYBDISILICIC ACID (PHOTOMETRIC) TEST METHOD

(This test method, which consisted of Sections 118 through 127 of this standard, was discontinued in 2008.)

---

[6] Olson, H. A., and Macy, D. W., "Metallurgical Approach to Evaluating Chemical Sample Disks," *Light Metals*, Vol 2, 1978, pp. 301–311.

SILICON BY THE SODIUM HYDROXIDE-PERCHLORIC ACID (GRAVIMETRIC) TEST METHOD

(This test method, which consisted of Sections 128 through 133 of this standard, was discontinued in 2008.)

TIN BY THE IODATE (TITRIMETRIC) TEST METHOD

(This test method, which consisted of Sections 134 through 140 of this standard, was discontinued in 2008.)

TITANIUM BY THE CHROMOTROPIC ACID (PHOTOMETRIC) TEST METHOD

141. Scope

141.1 This test method covers the determination of titanium in concentrations from 0.002 to 0.3 %.

142. Summary of Test Method

142.1 The sample is dissolved in a sodium hydroxide solution and acidified with nitric and sulfuric acids. Iron is reduced with ascorbic acid. The yellow complex of titanium with chromotropic acid is formed at a pH between 3.1 and 3.2. Photometric measurement is made at approximately 470 nm.

143. Concentration Range

143.1 The recommended concentration range is from 0.002 to 0.10 mg of titanium per 50 mL of solution, using a 2-cm cell.

NOTE 2—This test method has been written for cells having a 2-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

144. Stability of Color

144.1 The color develops within 5 min and is stable for 40 min.

145. Interferences

145.1 Chromium, if present, interferes because of the background color of the solution. Provision is made to correct for this interference.

146. Reagents

146.1 *Ascorbic Acid Solution* (40 g/L)—Dissolve 1 g of ascorbic acid in 25 mL of water. Do not use a solution that has stood more than 1 h.

146.2 *Chromotrophic Acid Solution (Disodium Salt)* (20 g/L)—Dissolve 2 g of chromotropic acid (4,5-dihydroxy-2,7-naphthalenedisulfonic acid, disodium salt) in 70 mL of water containing 0.75 mL of acetic acid. Add 0.2 g of sodium metadisulfite ($Na_2S_2O_5$) and stir until completely dissolved. Filter through a fine paper into a 100-mL volumetric flask. Wash with water, dilute to volume, and mix. Select a lot of reagent that meets the following criteria: The solution must be light, clear yellow and have an absorbance reading of 0.3 or less when measured at 470 nm in a 2-cm cell, using distilled water as the reference. Do not use a solution that has stood more than 3 weeks.

146.3 *Potassium Permanganate Solution* (1 g/L)—Dissolve 0.1 g of potassium permanganate ($KMnO_4$) in water and dilute to 100 mL.

146.4 *Reagent Mixture*—Transfer 300 mL of water to a 1-L volumetric flask, add in order 250 mL of NaOH Solution A, 250 mL of H_2SO_4 (1+4), and 18 mL of HNO_3 and mix. Cool, dilute to volume, and mix. (The pH should be about 0.50.)

146.5 *Sodium Hydroxide Solution A* (200 g/L)—Dissolve 200 g of sodium hydroxide (NaOH) in about 500 mL of water, dilute to about 900 mL, and cool. Transfer to a 1-L volumetric flask, dilute to volume, and mix. Immediately transfer to a plastic bottle.

146.6 *Sodium Hydroxide Solution B* (80 g/L)—Dissolve 80 g of sodium hydroxide (NaOH) in about 200 mL of water, dilute to about 900 mL, and cool. Transfer to a 1-L volumetric flask, dilute to volume, and mix. Immediately transfer to a plastic bottle.

146.7 *Sodium Metadisulfite* ($Na_2S_2O_5$).

146.8 *Sodium Monochloroacetic Acid Buffer Solution*—Dissolve 189 g of monochloroacetic acid in 150 mL of water. Dissolve 40 g of sodium hydroxide (NaOH) in about 100 mL of water, and cool. Add the NaOH solution to the monochloroacetic acid solution, mix thoroughly, and cool. If turbid, filter through a fine paper and wash the filter with water. Transfer to a 500-mL volumetric flask, dilute to volume, and mix. (The pH should be about 2.9.)

146.9 *Sodium Sulfite Solution* (20 g/L)—Dissolve 2 g of sodium sulfite (Na_2SO_3) in water and dilute to 100 mL. Do not use a solution that has stood more than 8 h.

146.10 *Sulfurous Acid Solution (saturated).*

146.11 *Titanium, Standard Solution A* (1 mL = 0.4 mg Ti)—Dissolve 0.400 g of titanium (purity: 99.5 % minimum) in 125 mL of H_2SO_4(1+4). When dissolution is complete, oxidize with 10 drops of HNO_3, and boil gently to expel fumes of nitrous oxide. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

146.12 *Titanium, Standard Solution B* (1 mL = 0.02 mg Ti)—Using a pipet, transfer 50 mL of Titanium Solution A to a 1-L volumetric flask, dilute to volume, and mix.

146.13 *Titanium, Standard Solution C* (1 mL = 0.002 mg Ti)—Using a pipet, transfer 100 mL of Titanium Solution B to a 1-L volumetric flask. Add 2.5 mL of H_2SO_4 (1+4), cool, dilute to volume, and mix. Do not use a solution that has stood more than 1 day.

147. Preparation of Calibration Curve

147.1 *Calibration Solutions:*

147.1.1 Using pipets, transfer 1, 2, 5, 10, and 15 mL of Titanium Solution C to 100-mL beakers containing 10 mL of the reagent mixture.

147.1.2 Using pipets, transfer 1, 2, 3, 4, and 5 mL of Titanium Solution B to 100-mL beakers containing 10 mL of the reagent mixture.

147.1.3 Add $KMnO_4$ solution dropwise until a permanent red color is developed. Add Na_2SO_3 solution dropwise, while mixing the solution thoroughly, until the permanganate is decomposed, and then add 1 drop in excess. Add 10 mL of monochloroacetic acid buffer solution and mix. Add 1.0 mL of ascorbic acid solution and mix. Adjust the volume to about 35 mL. Using a pH meter, adjust the pH from 2.1 to 2.2 with H_2SO_4 (1+4) or NaOH Solution B, as required. Proceed as directed in 147.3.

147.2 *Reference Solution*—Transfer 10 mL of reagent mixture to a 100-mL beaker and proceed as directed in 147.1.3.

147.3 *Color Development*—Using a pipet, add 5 mL of chromotropic acid solution, transfer to a 50-mL volumetric flask, dilute to volume, and mix.

147.4 *Photometry:*

147.4.1 *Multiple–Cell Photometer*—Measure the cell correction using absorption cells with a 2-cm light path and a light band centered at approximately 470 nm. Using the test cell, take the photometric readings of the calibration solutions.

147.4.2 *Single–Cell Photometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 2-cm light path and adjust the photometer to the initial setting, using a light band centered at approximately 470 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

147.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of titanium per 50 mL of solution.

148. Procedure

148.1 *Test Solution:*

148.1.1 Select and weigh a sample in accordance with the following table and transfer it to a 250-mL beaker.

Titanium, %	Sample Weight, g	Tolerance in Sample Weight, mg
0.001 to 0.03	1.000	0.5
0.02 to 0.30	0.500	0.2

148.1.2 Add 25 mL of NaOH Solution A, cover, and, if necessary, heat gently to start reaction. When reaction slows, wash the cover and sides of the beaker with hot water. Boil gently for a few minutes to complete the dissolution, and cool.

NOTE 3—For alloys containing more than 3 % silicon, proceed as follows: Transfer the sample to a platinum dish and cover with a platinum cover. Add 25 mL of NaOH solution A. When the major reaction ceases, wash down the sides of the dish and the cover with hot water, and evaporate the solution to a syrupy paste. Proceed as directed in 148.1.3.

148.1.3 Dilute to about 50 mL. Add 2 mL of HNO_3 and 40 mL of H_2SO_4(1+4). Mix and boil gently until the salts dissolve. If manganese dioxide has separated, add a few drops of H_2SO_3 solution and boil for 3 to 5 min. Cool, transfer to a 100-mL volumetric flask, dilute to volume, and mix.

148.1.4 Filter through a fine, dry paper, discard the first 10 to 20 mL, and collect about 50 mL. Using a pipet, transfer 10 mL if the expected titanium concentration is less than 0.15 %, or 5 mL if the expected titanium concentration is greater than 0.15 %, to a 100-mL beaker. Proceed as directed in 147.1.3.

148.2 *Reference Solution*—Carry a reagent blank through the entire procedure, using the same amounts of all reagents with the sample omitted.

148.3 *Color Development*—Proceed as directed in 147.3.

148.4 *Background Color Solution*—If the test solution contains chromium or other elements which form colored ions, transfer a second aliquot of the filtered solution obtained in 148.1.4 and proceed as directed in 147.1.3. After the pH adjustment, transfer to a 50-mL volumetric flask, dilute to volume, and mix.

148.5 *Background Color Reference Solution*—Use a portion of the reagent blank to which no chromotropic acid has been added.

148.6 *Photometry*—Take the photometric reading of the test solution and background color solution, if necessary, as directed in 147.4.

149. Calculation

149.1 Convert the net photometric readings of the test solution and the background color solution to milligrams of titanium by means of the calibration curve. Calculate the percentage of titanium as follows:

$$\text{Titanium, \%} = (A - B)/(C \times 10) \quad (1)$$

where:

A = titanium found in 50 mL of the final test solution, mg,
B = background color correction, mg of titanium, and
C = sample represented in 50 mL of the final test solution, g.

150. Precision

150.1 Six laboratories cooperated in testing this test method and obtained eight sets of data summarized in Table 1.

TABLE 1 Statistical Information

Test Specimen	Titanium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1. 1075 alloy	0.003	0.001	0.001
2. 356 alloy	0.112	0.006	0.006

ZINC BY THE AMMONIUM MERCURIC THIOCYANATE OR THE ZINC OXIDE (GRAVIMETRIC) TEST METHOD

(This test method, which consisted of Sections 151 through 159 of this standard, was discontinued in 1981.)

ZINC BY THE ETHYLENEDIAMINE TETRAACETATE (TITRIMETRIC) TEST METHOD

(This test method, which consisted of Sections 160 through 166 of this standard, was discontinued in 1983.)

CADMIUM BY THE ATOMIC ABSORPTION TEST METHOD

167. Scope

167.1 This test method covers the determination of cadmium in concentrations from 0.001 to 0.5 %.

168. Summary of Test Method

168.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption by the sample of the cadmium resonance line at 2288 Å is measured and compared with that of calibration solutions containing known amounts of cadmium and aluminum.

169. Concentration Range

169.1 If the optimum concentration range is not known, determine it as directed in Guide E1024. A sensitivity of 0.02 µg/mL at 0.0044 absorbance is frequently obtained.

170. Interferences

170.1 Elements normally present do not interfere if their concentrations are less than the maximum limits shown in 1.1.

171. Apparatus

171.1 *Atomic Absorption Spectrophotometer*—Determine that the instrument is suitable for use as prescribed in Guide E1024. The percent variability for the highest calibration solution (V_c) should not exceed 2 %.

171.1.1 *Operation Parameters:*

Wavelength	2288Å
Bandpass	about 6 Å
Gas mixture	air-acetylene
Flame type	lean

172. Reagents

172.1 *Aluminum Solution* (1 mL = 50 mg Al)—Transfer 10 g of aluminum (purity: 99.999 % min) to a 400-mL beaker. Add 50 mL of water and a small drop of mercury. Add 110 mL of HCl in small increments, heating moderately to accelerate the dissolution. When dissolution is complete, add 2 mL of HNO_3 and boil gently for 5 min. Cool, transfer to a 200-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

NOTE 4—The high purity aluminum is necessary when determining cadmium in concentrations less than 0.01 %.

172.2 *Cadmium, Standard Solution A* (1 mL = 1.00 mg Cd)—Transfer 1.00 g of cadmium (purity: 99.9 % min) to a 400-mL beaker. Add 5 mL of water, 10 mL of HCl, and 2 mL of HNO_3. Cover, heat gently until dissolution is complete, cool, and add 50 mL of water. Transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

172.3 *Cadmium, Standard Solution B* (1 mL = 0.08 mg Cd)—Using a pipet, transfer 20 mL of Cadmium Solution A to a 250-mL volumetric flask. Add 10 mL HCl, dilute to volume, and mix. Store in a polyethylene bottle.

172.4 *Cadmium, Standard Solution C* (1 mL = 0.02 mg Cd)—Using a pipet, transfer 20 mL of Cadmium Solution A to a 250-mL volumetric flask. Add 10 mL HCl, dilute to volume, and mix. Store in a polyethylene bottle.

173. Calibration

173.1 *Calibration Solutions:*

173.1.1 *0.001 to 0.05 % Cadmium*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Cadmium Solution C to 100-mL volumetric flasks. Add 20 mL of aluminum solution (171.1) to each flask, dilute to volume, and mix.

173.1.2 *0.05 to 0.50 % Cadmium*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Cadmium Solution B to 200-mL volumetric flasks. Add 8 mL of aluminum solution (171.1) to each flask, dilute to volume, and mix.

173.2 Since sensitivity may vary among instruments, determine the suitability of the selected concentration range and apparatus as directed in Guide E1024. Scale expansion may be required to meet the minimum response criteria for some ranges. Sample and calibration solutions always must contain the same quantity of aluminum per millilitre.

174. Procedure

174.1 *Test Solution:*

174.1.1 Transfer a 1.00-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Add 22 mL of HCl (1+1) in small increments. After the reaction has subsided, heat to hasten dissolution. Cool for 5 min, add 2 mL of HNO_3, and boil gently for 3 to 5 min.

NOTE 5—If insoluble silicon is present, dilute to 50 mL with hot water, filter using a medium paper into a 250-mL beaker, and wash the residue with hot water. Reserve the filtrate. Transfer the paper and residue to a platinum crucible, dry, and ignite at 600°C. Cool, add 5 drops of HNO_3 and 5 mL of HF, and evaporate carefully to dryness. Cool, add 1 mL of HCl (1+1) and 5 mL of hot water. Heat to dissolve the salts and add the solution to the reserved filtrate.

174.1.2 For 0.001 to 0.05 % cadmium, transfer the solution to a 100-mL volumetric flask, dilute to volume, and mix. Use a 500-mL volumetric flask for 0.05 and 0.5 % cadmium.

175. Measurements

175.1 Optimize the response of the instrument and take preliminary readings; complete the analysis and calculate the cadmium concentration as in the graphical, ratio, or single-point procedures, as described in Guide E1024.

NOTE 6—A three-slot burner is recommended for the lower range, and a 5-cm single slot burner for the higher range.

176. Calculation

176.1 Calculate the percentage of cadmium as follows:

$$\text{Cadmium, \%} = \frac{A}{B} \times 100 \tag{2}$$

where:

A = cadmium in the final test solution, mg, and
B = sample represented in the test solution, mg.

177. Precision[7]

177.1 Eight laboratories cooperated in testing this test method. The data are summarized in Table 2.

TABLE 2 Statistical Information

Test Specimen	Cadmium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
Pure aluminum (Aluminum Association 1080 alloy, 99.80 % Al)	0.0018	0.00008	0.0005
Pure aluminum (Aluminum Association 1075 alloy, 99.75 % Al)	0.011	[A]	0.002
Aluminum-copper alloy (Aluminum Association X2020 Alloy, 4 Cu-1 Li-0.6 Mn-0.2 Cd)	0.191	0.007	0.025

[A] R_1 is indeterminate because no deviations were observed in the pairs of determinations, which were carried to only three decimal places.

ZIRCONIUM BY THE ARSENAZO III PHOTOMETRIC TEST METHOD

178. Scope

178.1 This test method covers the determination of zirconium in concentrations from 0.01 to 0.3 %.

179. Summary of Test Method

---

[7] Supporting data are available from ASTM Headquarters. Request RR:E01-1066.

179.1 Zirconium in hydrochloric acid reacts with Arsenazo III to form a complex suitable for photometric measurement at approximately 665 nm.

180. Concentration Range

180.1 The recommended concentration range is from 0.002 to 0.030 mg of zirconium per 50 mL of solution, using a 1-cm cell.

NOTE 7—This test method has been written for cells having a 1-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

181. Stability of Color

181.1 The color develops within 5 min and is stable for 3 h; however, because of the possible loss of hydrochloric acid, it is advisable to take photometric readings promptly and to use covered absorption cells.

182. Interferences

182.1 Strong oxidants, reductants, sulfates, and fluorides interfere. Concentrations of fluoride and sulfate in the final solution must be less than 2 µg/mL and 1 mg/mL, respectively. The elements ordinarily present in aluminum and aluminum-base alloys do not interfere if their concentrations are under the maximum limits shown in 1.1.

183. Reagents

183.1 *Aluminum Solution* (1 mL = 25 mg Al)—Dissolve 45 g of aluminum chloride hexahydrate ($AlCl_3{\cdot}6H_2O$) in about 150 mL of HCl (1+1). Transfer to a 200-mL volumetric flask, dilute to volume with HCl (1+1), and mix.

183.2 *Ammonium Nitrate Wash Solution* (50 g/L)—Dissolve 25 g of ammonium nitrate (NH_4NO_3) in about 400 mL of water and dilute to 500 mL.

183.3 *Arsenazo III Solution* (2.5 g/L)—Dissolve 0.250 g of Arsenazo III [2,2′-(1,8-dihydroxy-3,6-disulfonaphthylene2,7-diazodibenzenearsonic acid)] in 90 mL of water containing 300 mg of sodium carbonate (Na_2CO_3), and heat gently. Using a pH meter, adjust the pH to 4.0 ± 0.1 with HCl (1+1), and cool. Transfer to a 100-mL volumetric flask, dilute to volume, and mix. This solution is stable at least 6 months.

NOTE 8—Some lots of reagent have been found to be completely unsatisfactory. Therefore, the reagent should be checked with a standard zirconium solution before use in this test method. A satisfactory reagent should give an absorbance of about 0.8 for the high standard (0.6 µg/mL Zr) at 665 nm using 1-cm cells.[8]

183.4 *Diammonium Phosphate Solution* (120 g/L)—Dissolve 60 g of diammonium phosphate ($(NH_4)_2HPO_4$) in about 400 mL of water and dilute to 500 mL.

183.5 *Zirconium, Standard Solution A* (1 mL = 0.100 mg Zr)—Prepare as described in 183.5.1 or 183.5.2. Store in a polyethylene bottle.

183.5.1 Transfer 0.100 g of zirconium (purity: 99.5 % min) to a 250-mL beaker. Add 30 mL of methanol (CH_3OH) and, while cooling, 5 mL of bromine (Br_2). When the reaction has ceased, heat gently to complete the attack. Add 20 mL of HCl and evaporate to moist salts but do not bake. Add 75 mL of HCl (1+3) and heat gently until dissolution of the salts is complete. Cool, transfer to a 1-L volumetric flask, dilute to volume with HCl (1+3), and mix.

183.5.2 Transfer 0.354 g of zirconyl chloride octahydrate ($ZrOCl_2{\cdot}8H_2O$) to a 250-mL beaker and add 100 mL of HCl (1+3). Boil for 5 min. Cool, transfer to a 1-L volumetric flask, dilute to volume with HCl (1+3), and mix. Standardize as follows: Using a pipet, transfer 200 mL to a 400-mL beaker. Add 2 mL of H_2O_2 and 25 mL of the $(NH_4)_2HPO_4$ solution. An excess of H_2O_2 must be present at all times. Filter using a 9-cm medium paper containing ashless paper pulp and wash thoroughly with cold NH_4NO_3 solution. Transfer the paper to a platinum crucible, dry, and ignite carefully so that the paper chars but does not flame. When the paper is charred, gradually increase the temperature until all the carbon is gone, and then heat at 1050°C for 15 min. Cool in a desiccator and weigh as zirconium pyrophosphate (ZrP_2O_7).

183.6 *Zirconium, Standard Solution B* (1 mL = 0.005 mg Zr)—Using a pipet, transfer 5 mL of Zirconium Solution A to a 100-mL volumetric flask. Add 2.5 mL of HCl, cool, dilute to volume with HCl (1+1), and mix. Do not use a solution which has stood for more than 8 h.

184. Preparation of Calibration Curve

184.1 *Calibration Solutions*—Using pipets, transfer 1, 2, 3, 4, 5, and 6 mL of Zirconium Solution B to six 50-mL volumetric flasks containing 10 mL of HCl (1+1). Add 2 mL of aluminum solution (1 mL = 25 mg Al). Proceed as directed in 184.3.

184.2 *Reference Solution*—Transfer 2 mL of aluminum solution (1 mL = 25 mg Al) to a 50-mL volumetric flask containing 10 mL of HCl (1+1). Proceed as directed in 184.3.

184.3 *Color Development*—Using a pipet, add 1 mL of Arsenazo III solution, dilute to volume with HCl (1+1), and mix.

184.4 *Photometry:*

184.4.1 Determine the wavelength of maximum absorbance (Note 8) by taking photometric readings of the calibration solution containing 0.020 mg of zirconium over the range from 600 to 700 nm. Between 630 and 670 nm, take 5-nm increments. Using the reference solution, adjust the photometer to the initial setting before each reading.

NOTE 9—The maximum absorbance of the zirconium-Arsenazo III complex normally occurs at 665 nm. It is advisable to verify this absorption maximum for each new lot of Arsenazo III.

184.4.2 *Multiple Cell Photometer*—Measure the cell correction using stoppered absorption cells with a 1-cm light path and a light band centered at the wavelength determined in 184.4.1. Using the test cell, take the photometric readings of the calibration solutions.

184.4.3 *Single Cell Photometer*—Transfer a suitable portion of the reference solution to a stoppered absorption cell having a 1-cm light path and adjust the photometer to the initial setting using a light band centered at the wavelength determined in 184.4.1. While maintaining this adjustment, take the photometric readings of the calibration solutions.

---

[8] Aldrich Chemical Co. Reagent No. A9277-5 and G. Frederick Smith Chemical Co. Reagent No. 594 have been found to be suitable for this purpose.

184.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of zirconium per 50 mL of solution.

185. Procedure

185.1 *Test Solution:*

185.1.1 Transfer a 0.200-g sample, weighed to the nearest 0.5 mg, to a 250-mL beaker.

185.1.2 Add 20 mL of HCl (1+1), heat until dissolution is complete, and evaporate carefully to moist salts. Cool, add about 180 mL of HCl (1+1), and heat gently to dissolve salts.

185.1.3 Cool and transfer to a 200-mL volumetric flask, ignoring any remaining residue. Dilute to volume with HCl (1+1), and mix. Allow any residue to settle.

185.1.4 Using a pipet, transfer to a 50-mL volumetric flask, 20 mL if the expected zirconium concentration is less than 0.10 %, 10 mL if the expected zirconium concentration is between 0.10 and 0.20 %, or 5 mL if the expected zirconium concentration is between 0.20 and 0.30 %. Add 2 mL of aluminum solution (1 mL = 25 mg Al).

185.2 *Reference Solution*—Proceed as directed in 184.2.

185.3 *Color Development*—Proceed as directed in 184.3.

185.4 *Photometry*—Take the photometric reading of the test solution as directed in 184.4.2 or 184.4.3.

186. Calculation

186.1 Convert the net photometric reading of the test solution to milligrams of zirconium by means of the calibration curve. Calculate the percentage of zirconium as follows:

$$\text{Zirconium, \%} = \frac{A}{B \times 10} \qquad (3)$$

where:

A = zirconium found in 50 mL of the final test solution, mg, and

B = sample represented in 50 mL of the final test solution, g.

187. Precision[9]

185.1 Seven laboratories cooperated in testing this test method and obtained eight sets of data summarized in Table 3.

TABLE 3 Statistical Information

Test Specimen	Zirconium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1. 6151 alloy	0.023	0.0027	0.0033
2. 2219 alloy	0.152	0.0097	0.019
3. 7046 alloy	0.282	0.0278	0.060

BISMUTH AND LEAD BY THE ATOMIC ABSORPTION TEST METHOD

188. Scope

188.1 This test method covers the determination of bismuth in concentrations from 0.02 to 1.0 %, and lead in concentrations from 0.01 to 1.0 %.

189. Summary of Test Method

189.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption by the sample solution of the bismuth resonance line at 2230Å and the lead resonance line at 2833 Å is measured and compared with the absorption of calibration solutions containing known amounts of bismuth and lead. The 2170-Å lead resonance line may be used successfully on some instruments, especially if an electrodeless discharge lamp is employed.

190. Concentration Range

190.1 If the optimum concentration range is not known, determine it as directed in Guide E1024. A sensitivity of 0.4 to 0.8 µg/mL for 0.0044 absorbance for bismuth, and 0.4 to 0.8 µg/mL for 0.0044 absorbance for lead using the 2833-Å line is widely obtained. At 2170Å, the sensitivity for lead is 0.2 µg/mL for 0.0044 absorbance.

191. Interferences

191.1 Elements normally present do not interfere if their concentrations are less than the maximum limits shown in 1.1.

192. Apparatus

192.1 *Atomic Absorption Spectrophotometer*—Determine that the instrument is suitable for use as prescribed in Guide E1024. The percent variability for the highest calibration solution (V_c) should not exceed 1 %.

193. Reagents

193.1 *Aluminum Solution* (1 mL = 50 mg Al)—Transfer 25 g of aluminum (purity: 99.99 % min) to a 1-L beaker. Add 100 mL of water and a small drop of mercury. Add 315 mL of HCl in small increments, heating moderately to accelerate the dissolution. When dissolution is complete, add 2 mL of H_2O_2 (30 %) and boil gently for 5 min. Cool, transfer to a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

193.2 *Bismuth, Standard Solution A* (1 mL = 0.40 mg Bi)—Transfer 0.400 g of bismuth (purity: 99.9 % min) to a 400-mL beaker and dissolve in 50 mL of HNO_3 (1+1), heating gently if necessary. When dissolution is complete, boil for 5 min, cool, and transfer to a 1-L volumetric flask. Add 100 mL of HNO_3 (1+1), dilute to volume, and mix. Store in a polyethylene bottle.

193.3 *Bismuth, Standard Solution B* (1 mL = 0.04 mg Bi)—Using a pipet, transfer 25 mL of Bismuth Standard Solution A to a 250-mL volumetric flask. Dilute to volume and mix. Do not use a solution that has stood for more than 24 h.

193.4 *Lead, Standard Solution A* (1 mL = 0.40 mg Pb)—Transfer 0.400 g of lead (purity: 99.9 % min) to a 400-mL beaker and proceed in accordance with 193.2.

193.5 *Lead, Standard Solution B* (1 mL = 0.04 mg Pb)—Dilute Lead Standard Solution A as directed in 193.3.

194. Calibration

194.1 *Calibration Solutions:*

194.1.1 *0.01 to 0.10 % Bi or Pb*—Using pipets, transfer 5, 10, 15, 20, and 25-mL portions of the appropriate Standard Solution B to 100-mL volumetric flasks. Add 20 mL of aluminum solution and 10 mL of HNO_3 (1+1). Cool, dilute to volume, and mix.

---

[9] Supporting data are available from ASTM Headquarters. Request RR:E01-1070.

194.1.2 *0.10 to 1.0 % Bi or Pb*—Using pipets, transfer 5, 10, 15, 20, and 25-mL portions of the appropriate Standard Solution A to 250-mL volumetric flasks. Add 20 mL of aluminum solution and 10 mL of HNO_3 (1+1). Cool, dilute to volume, and mix.

194.2 *Reference Solution*—Prepare a reference solution by adding the appropriate amount of aluminum solution and 10 mL of HNO_3 (1+1) to the appropriate size volumetric flask. Dilute to volume and mix.

194.3 Since sensitivity may vary among instruments, determine the suitability of the selected concentration range and apparatus as directed in Guide E1024. Scale expansion may be required to meet the minimum response criteria for some ranges. Sample and calibration solutions always must contain the same quantity of aluminum per millilitre.

195. Procedure

195.1 *Test Solution:*

195.1.1 Transfer a 1.000-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Add 20 mL of water and 25 mL of HCl (1+1) in small increments, and cover with a borosilicate cover glass. When the reaction subsides, add 10 mL of HNO_3 (1+1) and boil for 5 min.

195.1.2 Filter using a medium paper into a 100-mL volumetric flask when the bismuth or lead content is expected to be 0.10 % or less, or into a 250-mL volumetric flask when the bismuth or lead content is expected to be greater than 0.10 %. Wash the residue with hot water and reserve the filtrate.

195.1.3 When the silicon content is 0.5 % or greater, transfer the filter paper and residue to a platinum crucible, dry, and ignite at 550°C. Cool, add 5 mL of HF, and then add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, and dissolve the residue in 5 drops of HCl (1+1) and a minimum amount of water. Add this solution to the reserved filtrate obtained in 195.1.2.

195.1.4 Cool the solution obtained in 195.1.2 or the combined filtrates obtained in 195.1.3. Dilute to volume and mix.

196. Measurements

196.1 Optimize the response of the instrument and take preliminary readings; then complete the analysis and determine the concentration of bismuth or lead using the graphical, ratio, or single-point procedure, as described in Guide E1024.

197. Calculations

197.1 Calculate the percentage of bismuth or lead as follows:

$$\text{Bismuth or lead, \%} = \frac{A}{B} \times 100 \tag{4}$$

where:

A = bismuth or lead in the final test solution, mg, and

B = sample represented in the test solution taken for analysis, mg.

198. Precision and Bias[10]

198.1 *Precision*—Eight laboratories cooperated in testing this test method. The precision of this test method can be estimated by examining the data in Tables 4 and 5.

198.2 *Bias*—No information on the accuracy of this test method is available. The accuracy may be judged, however, by comparing accepted reference values with the corresponding arithmetic averages obtained by interlaboratory testing.

TABLE 4 Statistical Information

Test Specimen	Bismuth Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1000	0.033	0.0046	0.008
KS-0010-12 6262 alloy	0.60	0.0089	0.024

TABLE 5 Statistical Information

Test Specimen	Lead Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
NBS 85b 2024 alloy (0.021 % Pb)	0.021	0.0014	0.003
BCS No. 181/2 2218 alloy (0.04 % Pb)	0.041	0.0029	0.005
KS-0010-12 6262 alloy	0.55	0.015	0.044

CHROMIUM BY THE ATOMIC ABSORPTION TEST METHOD

199. Scope

199.1 This test method covers the determination of chromium in concentrations from 0.01 to 1.0 %.

200. Summary of Test Method

200.1 An acid solution of the sample is aspirated into the nitrous oxide-acetylene flame of an atomic absorption spectrophotometer. The absorption of the chromium resonance line at 3579 Å is measured and compared with the absorption of calibration solutions containing known amounts of chromium.

201. Concentration Range

201.1 If the optimum concentration range is not known, determine it as directed in Guide E1024. A sensitivity of 0.1 to 0.2 µg/mL for 0.0044 absorbance is widely obtained.

202. Interferences

202.1 Elements normally present do not interfere if their concentrations are less than the maximum limits shown in 1.1.

203. Apparatus

203.1 *Atomic Absorption Spectrophotometer*—Determine that the instrument is suitable for use as prescribed in Guide E1024. The percent variability for the highest calibration solution (V_c) should not exceed 1 %.

204. Reagents

204.1 *Aluminum Solution* (1 mL = 50 mg Al)—*Transfer 25 g of aluminum (purity: 99.99 % min) to a 1-L beaker. Add 100 mL of water and a small drop of mercury. Add 275 mL of HCl in small increments, heating moderately to accelerate the dissolution. When dissolution is complete, add 2 mL of* H_2O_2 *(30 %) and boil gently for 5 min. Cool, transfer to a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.*

204.2 *Chromium Standard Solution A* (1 mL = 0.40 mg Cr)—*Transfer 0.400 g of chromium (purity: 99.9 % min) to a 400-mL beaker containing 50 mL of water. Dissolve the metal with 15 mL of HCl. Transfer the solution to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.*

204.3 *Chromium Standard Solution B* (1 mL = 0.04 mg Cr)— *Using a pipet, transfer 25 mL of Chromium Solution A to a 250-mL volumetric flask. Dilute to volume and mix.*

---

[10] Supporting data are available from ASTM Headquarters. Request RR:E01-1073.

205. Calibration

205.1 *Calibration Solutions:*

205.1.1 *0.01 to 0.10 % Cr*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of the Chromium Standard Solution B to 100-mL volumetric flasks. Add 20 mL of aluminum solution, dilute to volume, and mix.

205.1.2 *0.1 to 1.0 % Cr*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Chromium Standard Solution B to 100-mL volumetric flasks. Add 2 mL of aluminum solution and 5 mL of HCl (1+1). Cool, dilute to volume, and mix.

205.2 *Reference Solution*—The 0 calibration solution is used as the reference solution.

205.3 Since sensitivity may vary among instruments, determine the suitability of the selected concentration range and apparatus as directed in Guide E1024. Scale expansion may be required to meet the minimum response criteria for some ranges. Sample and calibration solutions always must contain the same quantity of aluminum per millilitre.

206. Procedure

206.1 *Test Solution:*

206.1.1 Transfer a 1.000-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Add 20 mL of water and 22 mL of HCl (1+1) in small increments. Cover with a ribbed cover glass and when the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min.

206.1.2 Filter through a medium paper into a 100-mL volumetric flask. Wash with hot water and reserve the filtrate.

206.1.3 When the silicon content is 0.5 % or greater, transfer the filter paper and residue to a platinum crucible, dry, and ignite at 500°C. Cool, add 5 mL of HF, and then add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, and dissolve the residue in 5 drops of HCl (1+1) and a minimum amount of water. Add this solution to the reserved filtrate obtained in 206.1.2.

206.1.4 Cool the solution obtained in 206.1.2 or the combined filtrates obtained in 206.1.3. Dilute to volume and mix. This is Sample Solution A.

206.1.5 Pipet 10 mL of Sample Solution A into a 100-mL volumetric flask containing 5 mL of HCl (1+1). Dilute to volume and mix. This is Sample Solution B.

206.1.6 When the chromium concentration is less than 0.10 %, aspirate Sample Solution A into the flame using the standards from 205.1.1

206.1.7 When the chromium content is between 0.10 and 1.0 %, aspirate Sample Solution B into the flame using standards from 205.1.2.

207. Measurements

207.1 Optimize the response of the instrument and take preliminary readings; then complete the analysis and determine the chromium concentration using the graphical, ratio, or single-point procedure, as described in Guide E1024.

208. Calculation

208.1 Calculate the percentage of chromium as follows:

$$\text{Chromium, } \% = \frac{A}{B} \times 100 \tag{5}$$

where:

A = chromium in the final test solution, mg, and
B = sample represented in the test solution taken for analysis, mg.

TABLE 6 Statistical Information

Test Specimen	Chromium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
MD 184	0.010	0.0009	0.002
NBS 85b 2024 alloy (0.211 % Cr)	0.21	0.008	0.014
KNB 793-96	0.80	0.015	0.032

209. Precision and Bias[11]

209.1 *Precision*—Nine laboratories cooperated in testing this test method. The precision of the test method can be estimated by examining the data in Table 6.

209.2 *Bias*—No information on the accuracy of this test method is available. The accuracy may be judged, however, by comparing the accepted reference values with the corresponding arithmetic averages obtained by interlaboratory testing.

COPPER AND ZINC BY THE ATOMIC ABSORPTION TEST METHOD

210. Scope

210.1 This test method covers the determination of copper in concentrations from 0.01 to 10 %, and zinc in concentrations from 0.003 to 10 %.

211. Summary of Test Method

211.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption by the sample of the copper resonance line at 3247 Å and the zinc resonance line at 2139 Å is measured and compared with the absorption of calibration solutions containing known amounts of copper or zinc.

212. Concentration Range

212.1 If the optimum concentration range is not known, determine it as directed in Guide E1024. A sensitivity of 0.05 to 0.10 µg/mL for 0.0044 absorbance is widely obtained for copper and 0.02 to 0.06 µg/mL for zinc.

213. Interferences

213.1 Elements normally present do not interfere when their concentrations are less than the maximum limits shown in 1.1.

214. Apparatus

214.1 *Atomic Absorption Spectrophotometer*—Determine that the instrument is suitable for use as prescribed in Guide E1024. The percent variability for the highest calibration solution (V_c) should not exceed 1 %.

215. Reagents

215.1 *Aluminum Solution A* (1 mL = 50 mg Al)—Transfer 25 g of aluminum chips (purity: 99.99 % min) to a 1-L beaker. Add 100 mL of water and a small drop of mercury. Add 275 mL of HCl in small increments, heating moderately to accelerate dissolution. When dissolution is complete, add 2 mL of

---

[11] Supporting data are available from ASTM Headquarters. Request RR:E01-1074.