Designation: E34 – 11$^{\varepsilon 1}$

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 U.S. Department of Defense.

ε^1 NOTE—Editorial changes were made throughout in August 2012.

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 Spectometry 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 Spectometry 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). DOI: 10.1520/E0034-11E01.

[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.

[1e] Discontinued as of July 1, 2011.

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

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

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

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

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]

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

E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)[3]

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.

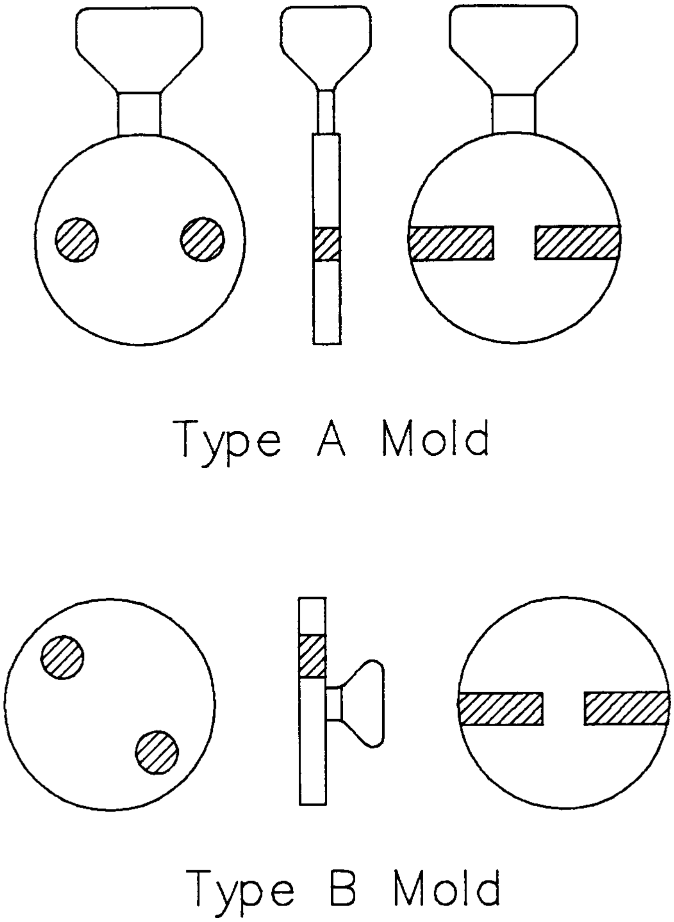
4. Significance and Use

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.



NOTE 1—Shaded areas are suitable for sampling.

FIG. 1 Type A and Type B Disks

---

[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.

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

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.[4]

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.[4]

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.[4]

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.[4]

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.)

---

[4] 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:

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

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:

$$\text{Cadmium, \%} = \frac{A}{B} \times 100 \qquad (2)$$

where:

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

177. Precision[5]

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

---

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

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

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.[6]

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.

---

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

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.

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:

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

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[7]

187.1 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 %.

---

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

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.

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. Calculation

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[8]

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.

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

---

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

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.

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.

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:

$$\text{Chromium, } \% = \frac{A}{B} \times 100 \qquad (5)$$

where:

A = chromium in the final test solution, mg, and

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

209. Precision and Bias[9]

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.

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

209.2 *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 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.

215.2 *Aluminum Solution B* (1 mL = 2.50 mg Al)—Pipet 25 mL of Aluminum Solution A into a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

215.3 *Copper Solution A* (1 mL = 1.00 mg Cu)—Transfer 1.000 g of copper (purity: 99.9 % min) to a 250-mL beaker. Add 5 mL of water, cover, and dissolve in 3 mL of HNO_3. After dissolution is complete, boil to remove oxides of nitrogen, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

215.4 *Zinc Solution A* (1 mL = 1.00 mg Zn)—Transfer 1.000 g of zinc (purity: 99.9 % min) to a 400-mL beaker containing 50 mL of water. Dissolve in 3 mL of HCl. Transfer the solution to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

215.5 *Copper and Zinc Standard Solution B* (1 mL = 0.04 mg Cu and 0.04 mg Zn)—Pipet 10 mL of Copper Solution A and 10 mL of Zinc Solution A into a 250-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

216. Calibration

216.1 *Calibration Solutions:*

216.1.1 *0.02 % to 0.1 % Cu and Zn*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Copper-Zinc Standard Solution B to 200-mL volumetric flasks. Add 20 mL of Aluminum Solution A and 13 mL of HCl (1+1). Dilute to volume and mix.

216.1.2 *0.1 % to 0.5 % Cu and Zn*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Copper-Zinc Standard Solution B to 200-mL volumetric flasks. Add 4 mL of Aluminum Solution A and 16 mL of HCl (1+1). Cool, dilute to volume, and mix.

216.1.3 *0.5 % to 2.5 % Cu and Zn*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Copper-Zinc Standard Solution B to 200-mL volumetric flasks. Add 16 mL of Aluminum Solution B and 16 mL of HCl (1+1). Cool, dilute to volume, and mix.

216.1.4 *2.0 % to 10 % Cu and Zn*—Using pipets, transfer 0, 5, 10, 15, 20, and 25-mL of Copper-Zinc Standard Solution B to 200-mL volumetric flasks. Add 4 mL of Aluminum Solution B and 17 mL of HCl (1+1). Cool, dilute to volume, and mix.

216.2 *Reference Solution*—The 0 calibration solution is used for the reference solution for each range of calibration.

216.3 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.

---

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

217. Procedure

217.1 *Test Solution:*

217.1.1 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). Warm, if necessary, to complete dissolution. When the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min.

217.1.2 Filter on a medium paper into a 100-mL volumetric flask. Wash the residue with hot water. Reserve the filtrate.

217.1.3 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 carefully to dryness, cool, and dissolve the residue in 5 drops of HCl (1+1) and minimum amount of water. Heat to dissolve the salts and add this solution to the filtrate reserved in 217.1.2.

217.1.4 Cool the solution from 217.1.2 or the combined filtrates obtained in 217.1.3. Dilute to volume and mix. This is Sample Solution A.

217.1.5 Pipet 10 mL of Sample Solution A into a 100-mL volumetric flask containing 8 mL of HCl (1+1), dilute to volume, and mix. This is Sample Solution B.

217.1.6 For copper or zinc concentrations less than 0.1 %, pipet 50 mL of Sample Solution A into a 100-mL volumetric flask containing 6.5 mL of HCl (1+1), dilute to volume, and mix. Use standards prepared in accordance with 216.1.1.

217.1.7 If the copper or zinc content is between 0.1 and 0.5 %, use Sample Solution B. Use standards prepared in 216.1.2.

217.1.8 If the copper or zinc content is between 0.5 and 2.5 %, pipet 20 mL of Sample Solution B into a 100-mL volumetric flask containing 6.5 mL of HCl (1+1), dilute to volume, and mix. Use standards prepared in accordance with 216.1.3.

217.1.9 If the copper or zinc content is between 2 and 10 %, pipet 10 mL of Sample Solution B into a 200-mL volumetric flask containing 16 mL of HCl (1+1), dilute to volume, and mix. Use standards prepared in accordance with 216.1.4.

218. Measurements

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

219. Calculation

219.1 Calculate the percentage of copper or zinc as follows:

$$\text{Copper or zinc,\%} = \frac{A}{B} \times 100 \tag{6}$$

where:

A = copper or zinc in the final test solution, mg, and

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

220. Precision and Bias[10]

220.1 *Precision*—Eight laboratories cooperated in testing this test method. The precision of this test method can be estimated by examining the data in Tables 7 and 8.

TABLE 7 Statistical Information

Test Specimen	Copper Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1. 5082 alloy	0.050	0.0019	0.0035
2. 7049 alloy	1.16	0.058	0.075
3. BCS No. 216/2 2014 alloy (4.56 % Cu)	4.52	0.054	0.24
4. 2219 alloy	6.18	0.093	0.26

TABLE 8 Statistical Information

Test Specimen	Zinc Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1. 5082 alloy	0.0028	0.0013	0.0016
2. 2219 alloy	0.036	0.0031[A]	0.0028
3. BCS No. 216/2 2014 alloy (0.20 % Zn)	0.20	0.0021	0.0094
4. 7049 alloy	7.60	0.25	0.25

[A] R_1 appears higher than R_2 because one of the eight laboratories that participated showed much poorer repeatability than the others for this sample.

220.2 *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.

IRON AND MANGANESE BY THE ATOMIC ABSORPTION TEST METHOD

221. Scope

221.1 This test method covers the determination of iron in concentrations from 0.02 % to 2.0 %, and manganese in concentrations from 0.01 % to 2.0 %.

222. Summary of Test Method

222.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption of the iron resonance line at 2483 Å and the manganese resonance line at 2795 Å is measured and compared with the absorption of calibration solutions containing known amounts of manganese or iron.

223. Concentration Range

223.1 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 for manganese and iron is widely obtained.

---

[10] Supporting data are available from ASTM Headquarters. Request RR:E01-1075.

224. Interferences

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

225. Apparatus

225.1 *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 %.

226. Reagents

226.1 *Aluminum Solution* (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 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.

226.2 *Manganese Standard Solution A* (1 mL = 0.40 mg Mn)—Transfer 0.400 g of manganese metal (purity: 99.9 % min) to a 400-mL beaker containing 50 mL 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.

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

226.4 *Iron Standard Solution A* (1 mL = 0.40 mg Fe)—Transfer 0.400 g of iron wire (purity: 99.9 % min) to a 400-mL beaker and proceed in accordance with 226.2.

226.5 *Iron Standard Solution B* (1 mL = 0.04 mg Fe)—Dilute Iron Standard Solution A in accordance with 226.3.

227. Calibration

227.1 *Calibration Solutions:*

227.1.1 *0.01 % to 0.10 % Mn or Fe*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of the appropriate Standard Solution B to 100-mL volumetric flasks. Add 20 mL of aluminum solution. Cool, dilute to volume, and mix.

227.1.2 *0.1 % to 1.0 % Mn or Fe*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of the appropriate Standard Solution B to 100-mL volumetric flasks. Add 2 mL of aluminum solution and 5 mL HCl (1+1). Cool, dilute to volume, and mix.

227.1.3 *1.0 % to 2.0 % Mn or Fe*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of the appropriate Standard Solution B to 100-mL volumetric flasks. Add 1 mL of aluminum solution and 5 mL HCl (1+1). Cool, dilute to volume, and mix.

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

227.3 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.

228. Procedure

228.1 *Test Solution:*

228.1.1 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, and cover with a ribbed cover glass. When the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min.

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

228.1.3 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 228.1.2.

228.1.4 Cool the solution obtained in 228.1.2 or the combined filtrates obtained in 228.1.3. Dilute to volume and mix. This is Sample Solution A.

228.1.5 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.

228.1.6 Pipet 5 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 C.

228.1.7 When the manganese or iron concentration is less than 0.10 %, aspirate Sample Solution A and use calibration solutions prepared in accordance with 227.1.1.

228.1.8 When the manganese or iron concentration is between 0.10 % and 1.0 %, aspirate Sample Solution B and use calibration solutions prepared in accordance with 227.1.2.

228.1.9 When the manganese or iron concentration is between 1.0 % and 2.0 %, aspirate Sample Solution C and use calibration solutions prepared in accordance with 227.1.3.

229. Measurements

229.1 Optimize the instrument response and take preliminary readings, then complete the analysis and determine the manganese or iron concentration using the graphical, ratio, or single-point procedure described in Guide E1024.

230. Calculation

230.1 Calculate the percentage of manganese or iron as follows:

$$\text{Manganese or iron,\%} = \frac{A}{B} \times 100 \tag{7}$$

where:

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

231. Precision and Bias[11]

231.1 *Precision*—Ten laboratories cooperated in testing this test method. The precision of this test method can be estimated by examining the data in Tables 9 and 10.

TABLE 9 Statistical Information

Test Specimen	Iron Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
7075 alloy	0.046	0.0036	0.009
BCS No. 181/2 2218 alloy (0.42 % Fe)	0.41	0.024	0.024
MD 184	1.60	0.039	0.051

TABLE 10 Statistical Information

Test Specimen	Manganese Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
MD 184	0.015	0.0009	0.003
NBS 85b 2024 alloy (0.61 % Mn)	0.60	0.015	0.023
3004 alloy	1.16	0.047	0.070

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

MAGNESIUM BY THE ATOMIC ABSORPTION TEST METHOD

232. Scope

232.1 This test method covers the determination of magnesium in concentrations from 0.002 % to 5.0 %.

233. Summary of Test Method

233.1 An acid solution of the sample is aspirated into the nitrous oxide-acetylene flame of an atomic absorption spectrophotometer. The absorption of the magnesium resonance line at 2852 Å is measured and compared with the absorption of calibration solutions containing known amounts of magnesium.

234. Concentration Range

234.1 If the optimum concentration range is not known, determine it as directed in Guide E1024. A sensitivity of 0.01 to 0.03 µg/mL for 0.0044 absorbance is widely obtained for magnesium.

235. Interferences

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

236. Apparatus

236.1 *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 %.

237. Reagents

237.1 *Aluminum Solution A* (1 mL = 50 mg Al)—Transfer 25 g of aluminum chips (purity: 99.999 % 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 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.

237.2 *Aluminum Solution B* (1 mL = 2.50 mg Al)—Pipet 25 mL of Aluminum Solution A into a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

237.3 *Aluminum Solution C* (1 mL = 1.00 mg Al)—Pipet 10 mL of Aluminum Solution A into a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

237.4 *Magnesium Standard Solution A* (1 mL = 1.00 mg Mg)—Transfer 1.000 g of magnesium (purity: 99.9 % min) to a 400-mL beaker. Dissolve by adding carefully, in small portions, 30 mL of HCl (1+1). Transfer the solution to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

237.5 *Magnesium Standard Solution B* (1 mL = 0.010 mg Mg)—Pipet 10 mL of Magnesium Solution A into a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

238. Calibration

238.1 *Calibration Solutions:*

238.1.1 *0.01 % to 0.05 % Mg*—Using pipets, transfer 0, 5, 10, 15, 20, and 25-mL portions of Magnesium Standard Solution B to 250-mL volumetric flasks. Add 10 mL of Aluminum Solution A and 20 mL of HCl (1+1). Cool, dilute to volume, and mix.

238.1.2 *0.05 % to 0.25 % Mg*—Using pipets, transfer 0, 5, 10, 15, 20, and 25-mL portions of Magnesium Standard Solution B to 250-mL volumetric flasks. Add 40 mL of Aluminum Solution B and 21 mL of HCl (1+1). Cool, dilute to volume, and mix.

238.1.3 *0.2 % to 1 % Mg*—Using pipets, transfer 0, 5, 10, 15, 20, and 25-mL portions of Magnesium Standard Solution B to 250-mL volumetric flasks. Add 10 mL of Aluminum Solution B and 21 mL of HCl (1+1). Cool, dilute to volume, and mix.

238.1.4 *1 % to 5 % Magnesium*—Using pipets, transfer 0, 5, 10, 15, 20, and 25-mL portions of Magnesium Standard Solution B to 250-mL volumetric flasks. Add 5 mL of Aluminum Solution C and 21 mL of HCl (1+1). Cool, dilute to volume, and mix.

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

---

[11] Supporting data are available from ASTM Headquarters. Request RR:E01-1076.

238.3 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.

239. Procedure

239.1 *Test Solution:*

239.1.1 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). Warm, if necessary, to complete dissolution. When the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min.

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

239.1.3 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 carefully to dryness, cool, and dissolve the residue in 5 drops of HCl (1+1) and a minimum amount of water. Heat to dissolve the salts and add this solution to the reserved filtrate obtained in 239.1.2.

239.1.4 Cool the solution obtained in 239.1.2 or the combined filtrates obtained in 239.1.3. Dilute to volume and mix. This is Sample Solution A.

239.1.5 Pipet 10 mL of Sample Solution A into a 100-mL volumetric flask containing 8 mL of HCl (1+1), dilute to volume, and mix. This is Sample Solution B.

239.1.6 For magnesium concentrations less than 0.05 %, pipet 20 mL of Sample Solution A into a 100-mL volumetric flask containing 8 mL of HCl (1+1), dilute to volume, and mix. Use the calibration solutions prepared in accordance with 238.1.1.

239.1.7 When the magnesium content is between 0.05 % and 0.25 %, pipet 10 mL of Sample Solution A into a 250-mL volumetric flask containing 21 mL of HCl (1+1), dilute to volume, and mix. Use the calibration solutions prepared in accordance with 238.1.2.

239.1.8 When the magnesium content is between 0.2 % and 1.0 %, pipet 25 mL of Sample Solution B into a 250-mL volumetric flask containing 19 mL of HCl (1+1), dilute to volume, and mix. Use the calibration solutions prepared in accordance with 238.1.3.

239.1.9 When the magnesium content is between 1 % and 5 %, pipet 5 mL of Sample Solution B into a 250-mL volumetric flask containing 20 mL of HCl (1+1), dilute to volume, and mix. Use the calibration solutions prepared in accordance with 238.1.4.

240. Measurements

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

241. Calculation

241.1 Calculate the percentage of magnesium as follows:

$$\text{Magnesium},\% = \frac{A}{B} \times 100 \tag{8}$$

where:

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

242. Precision and Bias[12]

242.1 *Precision*—Eight laboratories cooperated in testing this test method. The precision of this test method can be estimated by examining the data in Table 11.

TABLE 11 Statistical Information

Test Specimen	Magnesium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1. 2219 alloy	0.0066	0.0008	0.001
2. BCS No. 216/2 2014 alloy (0.74 % Mg)	0.75	0.013	0.030
3. 7049 alloy	2.78	0.042	0.15
4. 5082 alloy	4.25	0.14	0.18

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

NICKEL BY THE ATOMIC ABSORPTION TEST METHOD

243. Scope

243.1 This test method covers the determination of nickel in concentrations from 0.01 % to 4 %.

244. Summary of Test Method

244.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption of the nickel resonance line at 2320 Å is measured and compared with the absorption of calibration solutions containing known amounts of nickel.

245. Concentration Range

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

246. Interferences

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

---

[12] Supporting data are available from ASTM Headquarters. Request RR:E01-1077.

247. Apparatus

247.1 *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 %.

248. Reagents

248.1 *Aluminum Solution A* (1 mL = 50 mg Al)—Transfer 25.00 g of aluminum (purity: 99.99 % min) to a 1-L beaker. Add 100 mL of water, a small drop of mercury, and 275 mL of HCl in increments, heating moderately to accelerate the dissolution. When dissolution is complete, add 2 mL of H_2O_2 (30 %) and boil for 5 min. Cool, transfer to a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

248.2 *Aluminum Solution B* (1 mL = 2.5 mg Al)—Using a pipet, transfer 25 mL of Aluminum Solution A to a 500-mL volumetric flask, dilute to volume, and mix.

248.3 *Nickel, Standard Solution A* (1 mL = 1.00 mg Ni)—Transfer 1.000 g of nickel (purity: 99.9 % min) to a 400-mL beaker. Dissolve in 50 mL of HNO_3 (1+1), boil for 5 min, cool, and transfer to a 1-L volumetric flask. Dilute to volume and mix. Store in a polyethylene bottle.

248.4 *Nickel, Standard Solution B* (1 mL = 0.04 mg Ni)—Using a pipet, transfer 10 mL of Nickel Standard Solution A to a 250-mL volumetric flask, dilute to volume, and mix.

249. Calibration

249.1 *Calibration Solutions*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Nickel Standard Solution B to 100-mL volumetric flasks. Add Aluminum Solution A or B and HCl (1+1) as indicated as follows, dilute to volume, and mix.

Nickel Concentration, %	Aluminum Solution, mL	HCl (1+1), mL
0.01 to 0.20	10 Solution A	7
0.20 to 2.00	20 Solution B	8
2.00 to 4.00	10 Solution B	8

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

249.3 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.

250. Procedure

250.1 *Test Solution:*

250.1.1 Transfer a 1.0-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. When the reaction subsides, add 2 mL of H_2O_2 (30 %), heat until dissolution is complete, and boil gently for 5 min. Filter through a medium paper into a 100-mL volumetric flask, wash the residue with hot water, and reserve the filtrate.

250.1.2 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 HF, and then add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, and dissolve the salts in 5 drops of HCl (1+1) and a minimum amount of water. Add this solution to the reserved filtrate obtained in 250.1.1.

250.1.3 Cool the solution obtained in 250.1.1 or the combined filtrates obtained in 250.1.2. Dilute to volume and mix. This is Sample Solution A.

250.1.4 Pipet 10 mL of Sample Solution A into a 100-mL volumetric flask containing 8 mL of HCl (1+1), dilute to volume, and mix. This is Sample Solution B.

250.2 Prepare the test solution for aspiration according to the following:

250.2.1 When the nickel concentration is less than 0.2 %, pipet 50 mL of Sample Solution A into a 100-mL volumetric flask containing 7 mL of HCl (1+1), dilute to volume, and mix. Use the 0.01 % to 0.20 % nickel set of calibration solutions.

250.2.2 When the nickel concentration is between 0.20 % and 2.00 %, pipet 10 mL of Sample Solution A into a 200-mL volumetric flask containing 16 mL of HCl (1+1), dilute to volume, and mix. Use the 0.20 % to 2.00 % nickel set of calibration solutions.

250.2.3 When the nickel concentration is between 2.00 % and 4.00 %, pipet 25 mL of Sample Solution B into a 100-mL volumetric flask containing 6 mL of HCl (1+1), dilute to volume, and mix. Use the 2.00 % to 4.00 % nickel set of calibration solutions.

251. Measurements

251.1 Optimize the response of the instrument and take preliminary readings; complete the analysis and determine the concentration of nickel in the test solution by the graphical procedure, as described in Guide E1024.

NOTE 10—The graphical procedure is preferred because of the nonlinearity of nickel response at 232.0 nm.

252. Calculation

252.1 Calculate the percentage of nickel as follows:

$$\text{Nickel,\%} = \frac{A}{B} \times 100 \quad (9)$$

where:

A = nickel per 100 mL of final test solution, mg, and
B = sample represented in 100 mL of the final test solution taken for analysis, mg.

253. Precision and Bias[13]

253.1 *Precision*—Nine laboratories cooperated in testing this test method. The precision of this test method can be estimated by examining the data in Table 12.

253.2 *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.

---

[13] Supporting data are available from ASTM Headquarters. Request RR:E01-1078.

TABLE 12 Statistical Information

Test Specimen	Nickel Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
MD 184	0.011	0.0012	0.004
NBS 85b 2024 alloy (0.084 % Ni)	0.087	0.0053	0.009
BCS No. 181/2 2218 alloy (1.91 % Ni)	1.88	0.042	0.101

TITANIUM BY THE DIANTIPYRYLMETHANE PHOTOMETRIC TEST METHOD

254. Scope

254.1 This test method covers the determination of titanium in concentrations from 0.003 % to 0.3 %.

255. Summary of Test Method

255.1 The sample is dissolved in hydrochloric acid. Iron and vanadium are reduced with ascorbic acid in the presence of copper sulfate. The yellow titanium complex is formed with diantipyrylmethane. Photometric measurement is made at approximately 400 nm.

256. Concentration Range

256.1 The recommended concentration is from 0.015 to 0.15 mg of titanium per 100 mL, using a 1-cm cell.

NOTE 11—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.

257. Stability of Color

257.1 The color is developed within 1 h and is then stable for 8 h.

258. Interferences

258.1 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.

259. Reagents

259.1 *Aluminum Solution* (1 mL = 25 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 dissolution. When dissolution is complete, add 2 mL of H_2O_2 (1+1), and boil gently for 5 min. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene container.

259.2 *Ascorbic Acid Solution* (20 g/L)—Dissolve 2 g of ascorbic acid ($C_6H_8O_6$) in 100 mL of water. Do not use a solution that has stood for more than 1 h.

259.3 *Copper Sulfate Solution* (48 g/L)—Dissolve 7.5 g of copper sulfate ($CuSO_4 \cdot 5H_2O$) in water and dilute to 100 mL.

259.4 *Diantipyrylmethane Solution* (50 g/L)—Dissolve 10.0 g of diantipyrylmethane ($CH_2[C:(CH_3)N(CH_3)N(C_6H_5)CO]_2$), in 34 mL of HCl (1+1), and 150 mL of water. Dilute to 200 mL.

259.5 *Titanium, Standard Solution A* (1 mL = 0.5 mg Ti)—Dissolve 0.500 g of titanium (purity 99.5 % min) in 125 mL of H_2SO_4. When dissolution is complete, cool, add 10 drops of HNO_3, and boil gently for 5 min. Cool and dilute to about 800 mL. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

259.6 *Titanium, Standard Solution B* (1 mL = 0.015 mg Ti)—Using a pipet, transfer 15 mL of Titanium Solution A to a 500-mL volumetric flask, dilute to volume, and mix.

260. Preparation of Calibration Curve

260.1 *Calibration Solutions*—Using pipets, transfer 1, 2, 4, 6, 8, and 10 mL of Titanium Solution B to 100-mL volumetric flasks. Add 20 mL of aluminum solution. Proceed as directed in 260.3.

260.2 *Reference Solution*—Transfer 20 mL of aluminum solution to a 100-mL volumetric flask. Proceed as directed in 260.3.

260.3 *Color Development*—Using a pipet, add 25 mL of H_2SO_4 (1+1), dilute to 75 mL, and cool. Add 2 drops of copper sulfate solution and 2 mL of ascorbic acid solution, and mix. Using a pipet, add 10 mL of diantipyrylmethane solution, dilute to volume, and mix. Allow the color to develop for 1 h.

260.4 *Photometry:*

260.4.1 *Multiple–Cell Photometer*—Measure the cell correction using absorption cells with a 1-cm light path and a light band centered at approximately 400 nm. Using the test cell, take the photometric readings of the calibration solutions.

260.4.2 *Single–Cell Photometer*—Transfer a suitable portion of the reference solution to an absorption cell with a 1-cm light path and adjust the photometer to the initial setting, using a light path centered at approximately 400 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

260.5 *Calibration Curve*—Plot the net photometric readings of the calibration solutions against milligrams of titanium per 100 mL of solution.

261. Procedure

261.1 *Test Solution:*

261.1.1 Transfer a 1.00-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Cover with a ribbed cover glass.

261.1.2 Add 30 mL of water, and 30 mL of HCl (1+1) in small increments. Warm if necessary to complete dissolution. When the reaction subsides, add 2 mL of H_2O_2 (30 %), and boil for 5 min.

261.1.3 Filter, using a medium paper, into a 100-mL volumetric flask. Wash with hot water and reserve the filtrate.

261.1.4 When a visible silicon residue is present, transfer the filter paper and residue to a platinum crucible, dry, and ignite at 600°C until the carbon is removed, Cool, add 5 mL of HF, and add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, add 5 drops of HCl (1+1), and a minimum amount of water. Heat to dissolve the salts, and add the solution to the filtrate reserved in 261.1.3.

261.1.5 Cool the solution from 261.1.3 or 261.1.4, dilute to volume, and mix.

261.1.6 According to the expected titanium content, proceed with the volumes of test solution and aluminum solution listed in the following:

Expected Titanium, %	Volume of Test Solution, mL	Volume of Aluminum Solution, mL
0.003 to 0.030	50.0	...
0.020 to 0.070	20.0	12
0.060 to 0.30	5.0	18

261.1.7 Using a clean, dry pipet, transfer the appropriate volume of test solution to a 100-mL volumetric flask. Add the appropriate volume of aluminum solution. Reserve the remaining test solution for use in 261.4.

261.2 *Reference Solution*—Proceed as directed in 260.2.

261.3 *Color Development*—Proceed as directed in 260.3.

261.4 *Background Color Solution (Correction for elements in the test solution present as colored ions)*—Pipet an additional aliquot of the test solution to a 100-mL volumetric flask and add aluminum solution equal to that selected in 261.1.7. For a 50-mL aliquot, use the remaining test solution without pipetting. Proceed as in 260.3, but omit the diantipyrylmethane solution.

261.5 *Background Color Reference Solution*—Proceed as in 260.3, but omit the diantipyrylmethane solution.

261.6 *Photometry*—Take the photometric readings of the test solution and background color solution as directed in 260.4, each with its appropriate reference solution.

262. Calculation

262.1 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:

$$\text{Titanium,\%} = [(A - B)/C] \times 10 \qquad (10)$$

where:

A = titanium found in 100 mL of final test solution, mg,
B = background color correction, equivalent milligrams of titanium, and
C = sample represented in 100 mL of the final test solution, g.

263. Precision and Bias

263.1 *Precision*[14]—Ten laboratories cooperated in testing this test method on ISO-9 and ISO-22, and nine laboratories tested ISO-30. Seven of the laboratories analyzed each of the samples on five separate days while the other three laboratories analyzed their samples on either four or five separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 13.

263.2 *Bias*—No information on the accuracy of the test method is available.

TABLE 13 Statistical Information

Test Specimen	Titanium Found, %	Repeatability (R_1, E173; M = 1)	Reproducibility (R_2, E173; M = 1)
ISO-30 (5182 alloy)	0.0076	0.00085	0.00089
ISO-22 (7005 alloy)	0.0168	0.0013	0.0016
ISO-9 (10 Cu-1 Ni alloy)	0.155	0.0067	0.013

VANADIUM BY AN EXTRACTION-PHOTOMETRIC TEST METHOD USING *N*-BENZOYL-*N*-PHENYLHYDROXYLAMINE

264. Scope

264.1 This test method covers the determination of vanadium in concentrations from 0.002 % to 0.16 %.

265. Summary of Test Method

265.1 After dissolution of the sample in acids, the vanadium is oxidized with potassium permanganate. The vanadium (V) is complexed with *N*-benzoyl-*N*-phenylhydroxylamine, the complex is extracted with chloroform and photometric measurement is made at approximately 530 nm.

266. Concentration Range

266.1 The recommended concentration range is from 0.02 to 0.40 mg of vanadium per 50 mL of chloroform solution, using a 1-cm cell.

NOTE 12—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.

267. Stability of Color

267.1 The color is stable for at least 48 h.

268. Interferences

268.1 Other than titanium, the elements ordinarily present in aluminum alloys do not interfere when concentrations are less than the maximum limits shown in 1.1. Titanium, at concentrations above 2 mg in the sample solution will produce a positive interference. This becomes significant only when operating near the lower limit of the scope of this test method with samples having a high Ti-to-V ratio. It is evidenced by an off color in the test solution.

269. Reagents

269.1 *Chloroform* ($CHCl_3$), spectrophotometric grade.

269.2 *N-benzoyl-N-phenylhydroxylamine(BPHA) Solution* (1g/L)—Dissolve 0.250 g of BPHA ($C_6H_5CON(OH)C_6H_5$) in 100 mL of $CHCl_3$. Transfer to a dry 250-mL volumetric flask, dilute to volume with $CHCl_3$, and mix. When stored in a brown glass bottle in the dark, this reagent is stable for at least 2 months.

269.3 *Vanadium, Standard Solution A* (1 mL = 0.100 mg V)—Dissolve 0.1785 g of reagent grade V_2O_5, which has been ignited at 300°C for 1 to 2 h, in 20 mL of NaOH (5 g/100 mL) solution. Add 25 mL of H_2SO_4 (1+1) and cool to room temperature. Transfer to a 1-L volumetric flask, dilute to volume with water, and mix. Store in a polyethylene bottle.

---

[14] Supporting data are available from ASTM Headquarters. Request RR:E01-1087.

269.4 *Vanadium, Standard Solution B* (1 mL + 0.010 mg V)—Using a pipet, transfer 25 mL of Vanadium Solution A to a 250-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

269.5 *Potassium Permanganate Solution* (2 g/L)—Dissolve 0.20 g of $KMnO_4$ in water and dilute to 100 mL.

270. Preparation of Calibration Curve

270.1 *Calibration Solutions*—Using pipets, transfer 5, 10, 20, 30, and 40 mL of Vanadium Solution B to a series of 250-mL separatory funnels. Add to each 27 mL of H_2SO_4 (1+1), dilute to about 100 mL with water, and proceed as directed in 270.3.

270.2 *Reference Solution*—Transfer 27 mL of H_2SO_4 (1 + 1), to a 250-mL separatory funnel, dilute to about 100 mL with water, and proceed as directed in 270.3.

270.3 *Color Development:*

270.3.1 To the solutions in the separatory funnels, add $KMnO_4$ solution dropwise with mixing until a slight pink color persists for at least 10 min.

270.3.2 Add 30 mL BPHA solution and 34 mL of HCl. Immediately after addition of the HCl, shake for 1 min and allow the layers to separate. Collect the $CHCl_3$ layer in a dry 50-mL volumetric flask.

270.3.3 Re-extract with 10 mL of BPHA solution and combine with the extract obtained in 270.3.2. Dilute to volume with $CHCl_3$ and mix. Allow the extracts to stand for at least 1 h before taking photometric readings.

270.4 *Photometry:*

270.4.1 *Multiple Cell Photometer*—Measure the cell correction using stoppered absorption cells with a 1-cm light path and a light band centered at approximately 530 nm. Using the test cell, take the photometric readings of the calibration solutions.

270.4.2 *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 approximately 530 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.

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

271. Procedure

271.1 *Test Solution:*

271.1.1 Select a sample weight in accordance with the following table:

Vanadium, %	Sample Weight, g
0.002–0.025	1.0
0.025–0.070	0.5
0.07–0.16	0.25

271.1.2 Transfer the portion, weighed to the nearest 0.5 mg, to a 250-mL beaker. Add 25 mL of water, 7 mL of H_2SO_4 (1+1), and 2 mL HCl. Cover with a watch glass and, if necessary, heat gently to start reaction. When reaction slows, boil gently until reaction is completed and cool.

271.1.3 Filter, using a medium paper, into a 250-mL beaker. Wash with hot water and reserve the filtrate.

271.1.4 If a visible residue is present, transfer the filter paper and residue to a platinum crucible, dry, and ignite at 500°C (Note 13). Cool, add 5 mL of HF, and add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, and dissolve the residue in 5 drops of H_2SO_4 (1+1) and a minimum amount of water. Heat to dissolve the salts and add the solution to the filtrate reserved in 271.1.3.

NOTE 13—Experimental data on alloys containing up to 6 % silicon have shown that this test method yields the same results for vanadium even when this recovery step is omitted.

271.1.5 To filtrate from 271.1.3 or 271.1.4, add 20 mL of H_2SO_4 (1+1), cover with a ribbed cover glass, and carefully evaporate to fumes of H_2SO_4. Reduce the heat to avoid bumping and continue fuming for 15 min to remove chloride. After cooling, wash down with about 30 mL of water and heat to dissolve any salts. Dilute to 75 mL and boil the solution for 5 min. Cool and transfer to a 250-mL separatory funnel. Dilute to about 100 mL and proceed as directed in 270.3.

271.2 *Reference Solution*—Prepare a reference solution as described in 270.2.

271.3 *Color Development*—Proceed as directed in 270.3.

271.4 *Photometry*—Take the photometric reading of the test solution as described in 270.4.1 or 270.4.2.

272. Calculation

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

$$\text{Vanadium, \%} = (A / B \times 10) \quad (11)$$

where:

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

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

273. Precision and Bias

273.1 *Precision*—Nine laboratories cooperated in testing this test method. The precision of this test method can be estimated by examining the data in Table 14.

TABLE 14 Statistical Information

Test Specimen	Vanadium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
1070 Alloy	0.0032	0.00018	0.00095
7029 Alloy	0.056	0.0017	0.0072
2219 Alloy	0.140	0.0045	0.0085

273.2 *Bias*—No information on the accuracy of this test method is available.

ZINC BY THE ION EXCHANGE-EDTA TITRIMETRIC TEST METHOD

274. Scope

274.1 This test method covers the determination of zinc in concentrations from 0.1 % to 12 %.

275. Summary of Test Method

275.1 The sample is dissolved in acid, and excess acid is removed by evaporation. The residue is dissolved in dilute hydrochloric acid and passed through a strongly basic anion exchange resin. The adsorbed zinc is eluted from the column and titrated with disodium (ethylenedinitrilo) tetraacetate (EDTA), using dithizone as the indicator.

276. Interferences

276.1 Cadmium remains with zinc, and will be titrated. Cadmium is rarely encountered in other than negligible amounts in alloys containing zinc. Other elements ordinarily present do not interfere if their concentrations are less than the maximum limits shown in 1.1.

277. Apparatus

277.1 *Anion Exchange Column*—A glass column 20 mm in diameter and approximately 400-mm long, provided with a fritted disk and a stopcock. A modification of Apparatus No. 8 may be adapted to this test method. A reservoir for reagents may be added at the top of the column. However, reagents must be added according to the procedure described in 280.1.

278. Reagents

278.1 *Acetic Acid* (1 *M*)—Add 58 mL of glacial acetic acid (CH_3COOH) to water and make up to a volume of 1 L.

278.2 *Ammonium Acetate Solution* (500 g/L)—Dissolve 50 g of ammonium acetate (CH_3COONH_4) in water, and dilute to 100 mL.

278.3 *Anion Exchange Resin:*

278.3.1 Use a strongly basic anion exchange resin of the polystyrene-quaternary-ammonium type, chloride form, having a crosslinkage of 2 % to 3 % and a 50 to 100 nominal mesh size.[15] Wash the resin with successive portions of HCl (0.005 *M*), decanting until a clear solution is obtained. Allow to stand for 12 h in HCl (0.005 *M*).

278.3.2 *Preparation of the Ion Exchange Column*—Stir the resin, and add a sufficient amount of the suspension to obtain a column approximately 150-mm high after the resin has settled. Precautions should be taken to avoid air bubbles or channels (Note 14). Wash the column with 100 mL of HCl (0.005 *M*) at a flow rate of 5 to 7 mL/min.

NOTE 14—When not in use, the resin bed in the column should always be covered with HCl (0.005 *M*).

278.4 *Disodium Ethylenedinitrilo Tetraacetate (EDTA), Standard Solution:*

278.4.1 Dissolve 7.5 g of disodium (ethylenedinitrilo) tetraacetate dihydrate (EDTA) in water. Transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a plastic bottle.

278.4.2 Standardize as follows: Using a pipet, transfer 25 mL of zinc standard solution to a 400-mL beaker and dilute to 100 mL. Proceed as directed in 280.1.11 and 280.1.12. Calculate the zinc equivalent of the EDTA solution as follows:

$$\text{Zinc equivalent, g/mL} = A/(B - C) \qquad (12)$$

where:

A = zinc represented in 25 mL of zinc solution, g,
B = EDTA solution required for titration of the zinc solution, mL, and
C = EDTA solution required for titration of the blank, mL.

278.5 *Dithizone Solution (0.25 g/L)*—Dissolve 0.025 g of diphenyl thiocarbazone ($C_6H_5NHNHCSN{:}NC_6H_5$) in ethanol ($CH_3{\cdot}CH_2OH$) and dilute to 100 mL with ethanol.

278.6 *Hydrochloric Acid* (2 *M*)—Add 170 mL of HCl to water and dilute to 1 L.

278.7 *Hydrochloric Acid* (1 *M*)—Add 85 mL of HCl to water and dilute to 1 L.

278.8 *Hydrochloric Acid* (0.005 *M*)—Dilute 5 mL of HCl (1 M) with water and make up to a volume of 1 L.

278.9 *Zinc, Standard Solution* (1 mL = 2.00 mg Zn)—Dissolve 2.000 g of zinc (purity 99.95 % min) in 50 mL of HCl (1 + 1) diluted with 50 mL of water. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

279. Hazards

279.1 Dilute acid concentrations are expressed as molarities in the sections pertaining to the ion exchange steps to emphasize the need for careful dilution of concentrated acids. Standardization is not required.

280. Procedure

280.1 *Test Procedure:*

280.1.1 Transfer a 2.0-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Cover with a ribbed cover glass.

280.1.2 Carry a reagent blank through the entire procedure, using the same amounts of all reagents, but with the sample omitted.

280.1.3 Carefully add 50 mL of HCl (1+1) in small increments. When the reaction subsides, wash down the sides of the beaker and the cover glass, and add 2 mL of H_2O_2 (30 %) to dissolve copper. Warm gently to complete the dissolution, and carefully evaporate just to crystallization. Cool, and dissolve the salts with 100 mL HCl (2 *M*). Heat to complete the dissolution.

280.1.4 Filter, using a medium-porosity paper previously washed with hot HCl (6 *M*) and hot water, into a 250-mL beaker. Wash with 30 to 50 mL of hot HCl (2 *M*), and reserve the filtrate.

280.1.5 When a visible silicon residue is present, wash the paper and residue with hot water and discard the washings. Transfer the filter paper and residue to a platinum crucible, dry, and ignite at 600°C until the carbon is removed. Cool, add 5 mL of HF, and add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, and add 5 mL of HCl (2 *M*). Heat to dissolve the salts, add the solution to the filtrate reserved in 280.1.4, and cool.

280.1.6 For samples containing more than 1.5 % zinc, transfer the solution to a 200-mL volumetric flask, using HCl (2 *M*) as the transfer solution. Dilute to volume with HCl (2 *M*)

---

[15] Dowex 1 × 2, manufactured by the Dow Chemical Co., Midland, MI, has been found satisfactory for this purpose.

and mix. Using the following table as a guide, select the appropriate portion of the test solution for ion exchange.

Zn, %	Aliquot Volume, mL
0.1 to 1.5	Solution from 280.1.5
1.5 to 3	100
3 to 6	50
6 to 12	25

280.1.7 Transfer the aliquot selected into a beaker, dilute to approximately 150 mL with HCl (2 *M*), and add 0.5 mL of HNO_3.

280.1.8 Condition the ion exchange column by passing 200 mL of HCl (2 *M*), containing 0.5 mL of HNO_3, through the column at a rate of 5 to 7 mL/min.

280.1.9 Pass the test solution through the column at a rate of 5 to 7 mL/min. Wash the beaker and the column with five successive 25-mL portions of HCl (2 *M*), and then wash the column with 125 mL of HCl (1 *M*), maintaining the flow rate.

NOTE 15—If more than 6 mg of lead (Pb) are present, wash with 200 mL of HCl (1 *M*).

280.1.10 Elute the zinc from the column with 300 mL of HCl (0.005 *M*), at a flow rate of 5 to 7 mL/min, collecting the eluate in a 400-mL beaker. Cover with a ribbed cover glass, and evaporate carefully to approximately 100 mL.

NOTE 16—When bismuth (Bi) is present in the alloy, it will be retained on the column and will be seen as a light-colored band at the top of the column. Remove by washing the column with 200 mL of HNO_3 (7.5 *M*), followed by 200 mL of water. Condition the column for further samples as directed in 280.1.8.

280.1.11 Neutralize the evaporated eluate with ammonium hydroxide using a pH meter. Add 10 mL of acetic acid (1 *M*), and 10 mL of ammonium acetate solution. Adjust the pH to 5.3 ± 0.2 with ammonium hydroxide or acetic acid (1 *M*). Add 50 mL of acetone, and cool to approximately 5°C.

280.1.12 Add 2 mL of dithizone solution, and titrate immediately with the EDTA solution, until the color changes from red to yellow. The color should not vary after the addition of two additional drops of EDTA.

281. Calculation

281.1 Calculate the percentage of zinc as follows:

$$\text{Zinc,\%} = \left[(A - B)C/D\right] \times 100 \tag{13}$$

where:

A = EDTA solution required for titration of the test solution, mL,
B = EDTA solution required for titration of the blank, mL,
C = zinc equivalent of the EDTA solution, g/mL, and
D = sample represented in the final test solution, g.

282. Precision and Bias[16]

282.1 *Precision*—Sixteen laboratories cooperated in testing this test method on sample ISO-10 and fourteen laboratories tested sample ISO-15. All but one of the laboratories analyzed the samples on a minimum of five separate days. One laboratory analyzed ISO-15 on four separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 15.

TABLE 15 Statistical Information

Test Specimen	Zinc Found, %	Repeatability (R_1, E173, M = 1)	Reproducibility (R_2, E173, M = 1)
ISO–10	5.97	0.255	0.305
ISO–15	0.432	0.0155	0.0193

282.2 *Bias*—No information on the accuracy of this test method is available.

BERYLLIUM BY ARGON PLASMA OPTICAL EMISSION SPECTROSCOPY

283. Scope

283.1 This test method covers the analysis of aluminum and aluminum alloys having beryllium composition within the limits of 0.3 to 100 ppm.

284. Summary of Test Method

284.1 An acid solution of the sample is aspirated into the jet of an argon plasma optical emission spectrometer. The beryllium concentration is measured at 234.86 nm by comparing it with matched matrix calibration solutions.

285. Interferences

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

286. Apparatus

286.1 *Argon Plasma Optical Emission Spectrometer*—Follow manufacturer's instruction manual for installation and operation.

287. Reagents

287.1 *Argon*—Purity: 99.998 %, min.

287.2 *Aluminum Solution* (1 mL = 20 mg Al)—Transfer 20 g of aluminum (purity: 99.999 % min) into a 1-L beaker. Add 400 mL of HCl (1+1) in which 20 mg of $HgCl_2$ have been dissolved and heat moderately to accelerate dissolution (Note 17). When dissolution is complete, add 2 mL of H_2O_2 (30 %) and boil gently for 5 min. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

NOTE 17—High purity aluminum is difficult to dissolve. An alternative procedure is to allow the aluminum and acid mixture to stand overnight in the covered beaker. After 16 h, only a minimum amount of heating is usually required to complete the dissolution.

287.3 *Beryllium, Standard Solution A* (1 mL = 0.1 mg Be)—Transfer 100 mg beryllium metal (purity: 99.95 %) into a 100-mL beaker. Add 20 mL of HCl (1+1) and warm gently to just dissolve the metal. Transfer into a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

---

[16] Supporting data are available from ASTM Headquarters. Request RR:E01-1090.

287.4 *Beryllium, Standard Solution B* (1 mL = 0.01 mg Be)—Pipet 50 mL of Beryllium Standard Solution A into a 500-mL volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

287.5 *Beryllium, Standard Solution C* (1 mL = 0.001 mg Be)—Pipet 10 mL of Beryllium Standard Solution B into a 100-mL volumetric flask. Add 2 mL of HCl (1+1), dilute to volume with distilled water, and mix. Prepare fresh daily as needed.

287.6 *Beryllium, Standard Solution D* (1 mL = 0.0001 mg Be)—Pipet 10 mL of Beryllium Standard Solution C into a 100-mL volumetric flask. Add 2 mL of HCl (1+1), dilute to volume with distilled water, and mix. Prepare fresh daily as needed.

288. Hazards

288.1 Beryllium and its compounds are extremely toxic and care must be exercised in their handling. For precautions to be observed, refer to Practices E50. To prevent eye damage, the ultraviolet radiation from the argon plasma must be shielded at all times.

289. Calibration

289.1 *Calibration Solutions*—Using pipets, transfer into 100-mL volumetric flasks the volumes of the appropriate beryllium standard indicated as follows:

mL	Beryllium Standard	Be in Al, ppm
0	...	0
2.0	D	0.2
5.0	D	0.5
10.0	D	1.0
5.0	C	5.0
10.0	C	10.0
25.0	C	25.0
5.0	B	50.0
10.0	B	100.0

Add 50 mL of aluminum solution to each flask, dilute to volume, and mix.

289.2 *Instrument Optimization*—While aspirating the highest beryllium calibration solution, adjust all instrument parameters, argon flow rate, wavelength, photomultiplier tube voltage, viewing height, and sample uptake to optimize response according to the instrument manufacturer's recommended operating practice.

289.3 *Standardization*—Aspirate each of the calibration solutions in 289.1 and record the emission intensity as related to concentration. Aspirate 0.01 *M* HCl between each run. Prepare an analytical curve by plotting the emission intensity of each calibration solution versus its concentration or calculate a standard curve. If the instrument is equipped for direct reading of concentrations, this method of calibration may be used provided that the proper blank correction is applied.

290. Procedure

290.1 *Test Solution:*

290.1.1 Transfer a 1.0-g sample, weighed to the nearest 1 mg, into a 250-mL beaker. Add 20 mL of water and 20 mL of HCl (1+1) in small increments, and cover with a ribbed cover glass. When the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min.

290.1.2 Filter, using a 0.45-µ membrane filter, into a 100-mL volumetric flask. Wash with hot water and reserve the filtrate.

290.1.3 When a visible silicon residue is present, transfer the membrane filter and residue into a platinum crucible, dry, and ignite at 500°C until the carbon is removed. Cool, add 5 mL HF, and add HNO_3 dropwise until a clear solution is obtained. Add 4 drops of H_2SO_4 (1+1), evaporate to fumes, and gently fume for 2 min. Cool, add 5 drops of HCl (1+1) and a minimum amount of water. Heat to dissolve the salts and combine the solution with the filtrate reserved in 290.1.2.

290.1.4 Cool the solution from 290.1.2 or 290.1.3, dilute to volume, and mix.

290.1.5 Aspirate the test solution and record its emission intensity or concentration. Verify the calibration by analyzing a calibration standard with beryllium content similar to the test solution. If the value obtained is not within 5 % of the expected value, repeat the instrument calibration procedure. Wash with 0.01 *M* HCl between each run.

291. Calculation

291.1 Calculate the concentration of beryllium in the test solution in parts per million (µg/g) using the analytical curve prepared in 289.3. Direct reading instruments will provide the results in the calibrated concentration units.

292. Precision and Bias[17]

292.1 *Precision*—Ten laboratories cooperated in testing this test method and obtained twelve sets of data. Five laboratories used ICP spectrometers and seven laboratories used DCP. All samples were analyzed on three separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 16.

TABLE 16 Statistical Information

Test Specimen	Beryllium Found, µg/g (ppm)	Repeatability (R_1, E173, M = 1)	Reproducibility (R_2, E173, M = 1)
1145 Alloy	0.18	0.10	0.16
1145 Alloy	6.50	0.58	0.92
7075 Alloy (NBS 859) (26 ppm Be)	26.2	2.2	3.3
4032 Alloy	31.0	3.5	3.8

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

---

[17] Supporting data are available from ASTM Headquarters. Request RR:E01-1092.

MANGANESE BY THE PERIODATE (PHOTOMETRIC) TEST METHOD

293. Scope

293.1 This test method covers the determination of manganese in aluminum and aluminum alloys in concentrations from 0.005 % to 2.0 %.

294. Summary of Test Method

294.1 Manganese, in acid solution, is oxidized to permanganate ion by potassium periodate. Photometric measurement is made at approximately 525 nm.

295. Concentration Range

295.1 The recommended concentration range is from 0.1 to 2.0 mg Mn/100 mL of solution using a cell depth of 1 cm (see Note 12).

296. Stability of Color

296.1 The color develops within 30 min if the concentration is less than 0.2 mg Mn/100 mL of solution and within 15 min if the concentration is greater than this amount. The color is stable for several weeks if excess periodate is present.

297. Interferences

297.1 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.

298. Reagents

298.1 *Boric Acid Solution* (50 g/L)—Dissolve 50 g of boric acid (H_3BO_3) in hot water, cool, and dilute to 1 L.

298.2 *Fluoroboric Acid Solution*—Add 200 mL of HF to 800 mL of boric acid solution (298.1) in a plastic bottle, and mix.

298.3 *Manganese, Standard Solution A* (1 mL = 1.0 mg Mn)—Clean electrolytic manganese metal (purity 99.9 % min) in H_2SO_4 (1+9). Rinse thoroughly by decantation and dry. Transfer 1.000 g (weighed to within 1 mg) to a 400-mL beaker. Dissolve in 100 mL of H_2SO_4 (1+9). When dissolution is complete, boil the solution for 5 min and cool. Transfer to a 1-L volumetric flask, dilute to volume, and mix.

298.4 *Manganese, Standard Solution B* (1 mL = 0.1 mg Mn)—Using a pipet, transfer 100 mL of Manganese Standard Solution A to a 1-L volumetric flask. Dilute to volume and mix.

298.5 *Potassium Periodate Solution* (50 g/L)—Dissolve 50 g of potassium periodate (KIO_4) in water, add 200 mL HNO_3, and dilute to 1 L.

298.6 *Sodium Hydroxide Solution* (200 g/L)—In a nickel dish, dissolve 200 g of sodium hydroxide (NaOH) in water. Cool and dilute to 1 L. Store in a plastic container.

298.7 *Sodium Nitrite Solution* (20 g/L)—Dissolve 2 g of sodium nitrite ($NaNO_2$) in water and dilute to 100 mL. Prepare fresh daily.

298.8 *Sulfurous Acid Solution*—Pass a stream of sulfur dioxide gas (SO_2) through water until saturated.

298.9 *Water, Free of Reducing Agents*—To 1 L of water, and 10 mL of H_2SO_4 and heat to boiling. Add a few crystals of KIO_4 and continue to boil for 10 min.

299. Preparation of Calibration Curve

299.1 *Calibration Solutions*—Using pipets or a buret, transfer 1.0, 2.0, 5.0, 10.0, 15.0, and 20.0 mL of Manganese Standard Solution B (1 mL = 0.10 mg Mn) to 250-mL beakers. To each add 15 mL of H_2SO_4 (1+1), 10 mL of HNO_3, and 5 mL of H_3PO_4. Dilute to about 70 mL.

299.2 *Reagent Blank*—Transfer 15 mL of H_2SO_4, 10 mL of HNO_3, and 5 mL of H_3PO_4 to a 250-mL beaker and dilute to about 70 mL.

299.3 *Color Development*—Heat to boiling and add 10 mL of KIO_4 solution. Continue to boil until the characteristic color develops and for an additional 5 min (an additional 15 to 30 min if the manganese content is less than 0.2 mg). Cool and transfer to a 100-mL volumetric flask, previously rinsed with water treated to remove reducing agents (see 298.7). Dilute to volume with the treated water.

299.4 *Photometry:*

299.4.1 *Multiple Cell Photometer*—Measure the cell correction using absorption cells with a 1-cm light path and a light band centered at approximately 525 nm. Using the test cell, take photometric readings of the reagent blank and calibration solutions. After each reading, add 2 drops of $NaNO_2$ solution to the cell, mix, and repeat the measurement.

299.4.2 *Single Cell Photometer*—Transfer a suitable portion of water to an absorption cell with a 1-cm light path and adjust the photometer to the initial setting using a light band centered at approximately 525 nm. While maintaining this adjustment, take photometric readings of the reagent blank and calibration solutions. After each reading, add 2 drops of $NaNO_2$ solution to the cell, mix, and repeat the measurement.

299.5 For each solution, calculate the net absorbance as follows:

$$A = (B - C) - (D - E) \tag{14}$$

where:

A = net absorbance,
B = gross absorbance of the calibration or test solution,
C = absorbance of the solution after $NaNO_2$ solution addition,
D = gross absorbance of the reagent blank, and
E = absorbance of the reagent blank after $NaNO_2$ solution addition.

299.6 *Calibration Curve*—Plot the net absorbance readings of the calibration solutions against milligrams of manganese per 100 mL of solution.

300. Procedure

300.1 *Test Solution:*

300.1.1 *Manganese Content, 0.1 % to 2.0 %*—Transfer 1.0 g of the sample, weighed to the nearest 1 mg to a 100-mL platinum or tetrafluoroethylene (TFE) vessel and add, in small amounts, 40 mL of NaOH solution. Cover with a platinum or TFE lid and heat gently to facilitate the attack. Move the lid slightly and evaporate with care until a syrupy consistency is reached. Cool, wash the lid and walls of the vessel with about 25 mL of warm water, and warm gently. Transfer to a 250-mL beaker containing 30 mL of H_2SO_4 (1+1) and 10 mL of HNO_3. If manganese hydroxide adheres to the vessel, return some of the test solution and add several drops of sulfurous acid solution. Mix and transfer back to the beaker. Transfer the test solution to the volumetric flask indicated below. Dilute to volume, mix, and using a pipet, transfer the indicated aliquot into a 250-mL beaker. Add 5 mL of HNO_3, 5 mL of H_3PO_4, and the required volume of H_2SO_4 (1+1). Dilute to about 70 mL.

Mn, % (estimated)	Volumetric Flask, mL	Aliquot, mL	H_2SO_4 (1+1) Addition, mL
0.1 to 0.4	100	50	5
0.4 to 1.0	250	50	10
1.0 to 2.0	250	25	10

300.1.2 *Manganese Content, 0.005 % to 0.1 %, Silicon Content Less Than 10 %*—Weigh and dissolve 1.0 g of sample as directed in 300.1.1 but do not transfer to a volumetric flask. Concentrate the solution in the beaker to a volume of about 65 mL and add 5 mL of H_3PO_4.

300.1.3 *Manganese Content, 0.005 % to 0.1 %, Silicon Content More Than 10 %*—Transfer 1.0 g of sample, weighed to the nearest 1 mg, to a 250-mL beaker. Add 20 mL of H_2SO_4 (1+1), 10 mL of HNO_3, and 2 mL of fluoroboric acid solution and warm gently to dissolve. Heat to boiling to remove nitrous fumes. Dilute to about 50 mL, add a little filter pulp, and heat for several minutes. Filter through closetextured paper, collecting the filtrate and washings in a 250-mL beaker. Transfer the filter paper and pulp to a platinum crucible and ignite in a muffle furnace at 500°C to 600°C to complete destruction of the paper. Cool, add several drops of H_2SO_4, 2 to 5 mL of HF, and HNO_3 dropwise until the solution clears. Evaporate to dryness over a Meker burner, continue heating for several more minutes, and cool. Take up the residue in a little H_2SO_4 (1+1) and warm water with heating. Filter if necessary and combine the solutions. Concentrate to about 65 mL and add 5 mL of H_3PO_4.

NOTE 18—This alternative procedure may be used for silicon-aluminum alloys, containing more than 0.1 % Mn, by diluting the test solution to volume before the addition of the 5 mL of H_3PO_4, and taking a suitable aliquot as described in 300.1.1.

300.2 *Reagent Blank*—Carry a reagent blank through the entire procedure using the same amounts of all reagents.

300.3 *Color Development*—Proceed as directed in 299.3.

300.4 *Photometry*—Proceed as directed in 299.4.

301. Calculation

301.1 Calculate net absorbance as directed in 299.5. Convert the net absorbance of the test solution to milligrams of manganese by means of the calibration curve. Calculate the percentage of manganese as follows:

$$\text{Manganese, \%} = F/(G \times 10) \tag{15}$$

where:

F = manganese in 100 mL of the final test solution, mL, and

G = sample represented in 100 mL of the final test solution, g.

302. Precision and Bias[18]

302.1 *Precision*—Eleven laboratories cooperated in testing this test method using alkaline dissolution on sample ISO–17, ten laboratories on ISO–6, and eight on ISO–13. Each laboratory analyzed the samples on a minimum of six separate days. Six laboratories cooperated in testing the acid dissolution procedure on ISO–7 (12.9 % Si). Each laboratory analyzed the sample on a minimum of four separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 17.

TABLE 17 Statistical Information

Test Specimen	Manganese Found, %	Repeatability (R_1, E173, $M = 1$)	Reproducibility (R_2, E173, $M = 1$)
NaOH Dissolution:			
ISO–17	0.848	0.0200	0.0442
ISO–6	0.244	0.00886	0.0157
ISO–13	0.0209	0.00163	0.00362
Acid Dissolution:			
ISO–7 (12.9 % Si)	0.0096	0.00072	0.00124

302.2 *Bias*—No data relating to the magnitude of the bias of results obtained using this test method are available.

COPPER BY THE ELECTROLYTIC (GRAVIMETRIC) TEST METHOD

303. Scope

303.1 This test method covers the determination of copper in aluminum and aluminum alloys in concentrations from 0.5 % to 20 %.

304. Summary of Test Method

304.1 The sample is dissolved in acid, silicon is removed by filtration, and copper is recovered from the nonvolatile residue remaining after treatment with HF. The sample solution is electrolyzed and the cathodic deposit is weighed.

305. Interferences

305.1 Of the elements ordinarily present in aluminum and aluminum alloys, only bismuth, tin, and antimony interfere. This test method should not be applied when these elements are known to be present. This test method should not be applied to alloys containing greater than 2.0 % silicon.

306. Apparatus

306.1 *Electrodes for Electroanalysis*—Platinum electrodes of the stationary type are recommended as described in 306.1.1 and 306.1.2, but strict adherence to the exact size and shape of

---

[18] Supporting data are available from ASTM Headquarters. Request RR:E01-1094.

the electrodes is not mandatory. When agitation of the electrolyte is permissible in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed. The surface of the platinum electrodes should be smooth, clean, and bright to promote uniform deposition and good adherence. Sandblasting is not recommended.

306.1.1 *Cathodes*—Platinum cathodes may be formed either from plain or perforated sheets or from wire gauze, and may be either open or closed cylinders. Gauze cathodes are recommended, and shall be made preferably from 50-mesh gauze woven from wire approximately 0.21 mm (0.0085 in.) in diameter. The cathode should be stiffened by doubling the gauze for about 3 mm at the top and the bottom of the cylinder or by reinforcing the gauze at the top and bottom with a platinum band or ring. The cylinder should be approximately 30 mm in diameter and 50 mm in height. The stem should be made from a platinum alloy wire such as platinum-iridium, platinum-rhodium, or platinum-ruthenium, having a diameter of approximately 1.30 mm. It should be flattened and welded the entire length of the gauze. The over-all height of the cathode should be approximately 130 mm. A cathode of these dimensions will have a surface area of 135 cm^2 exclusive of the stem.

306.1.2 *Anodes*—Platinum anodes may be of the spiral type when anodic deposits are not being determined, or if the deposits are small (as in the electrolytic determination of lead when it is present in amounts not over 0.2 %). When used in analyses where both cathodic and anodic plates are to be determined, the anodes should be of wire gauze. Spiral anodes should be made from 1.00-mm or larger platinum wire formed into a spiral of seven turns having a height of approximately 50 mm and a diameter of 12 mm, the over-all height being approximately 130 mm. A spiral anode of this description will have a surface area of 9 cm^2. Platinum gauze anodes should be made of the same material and of the same general design as platinum gauze cathodes. The anode cylinder should be approximately 12 mm in diameter and 50 mm in height and the over-all height of the anode should be approximately 130 mm. A gauze anode of these dimensions will have a surface area of 54 cm^2. Both areas are exclusive of the stem.

307. Hazards

307.1 Perchloric acid can be safely used as specified in the procedure. It should not be brought into contact with finely divided aluminum, silicon, or organic matter if nitric acid is not present. Exhaust hoods should be in accordance with Practices E50.

308. Reagents

308.1 *Sulfamic Acid*—NH_2SO_3H.

309. Procedure

309.1 Select a sample weight in accordance with the following:

Copper, %	Sample Weight, g	$HClO_4$ (1+1), mL	HNO_3, mL
0.5 to 2.0	5	180	5
2.0 to 5.0	2	75	5
5.0 to 20.0	1	40	5

309.2 Transfer the sample, weighed to the nearest 1 mg, to a 400-mL beaker. Cover with a ribbed cover glass. Just before use, mix the quantities of $HClO_4$ (1+1) and HNO_3 as specified in 309.1, and add the mixture, in small increments. Moderate heat may be required to start the reaction. When dissolution is complete, evaporate to dense white fumes and fume for 15 min. Allow to cool, add 300 mL of hot water, stir, and boil for 5 min.

309.3 Allow the residue to settle, and filter while hot, using a medium-porosity paper and a little paper pulp. Wash well with hot water and collect the filtrate and washings in a 600-mL beaker.

309.4 Transfer the filter paper and residue to a platinum crucible, dry, and ignite at 550°C. Cool, add 1 mL of H_2SO_4 (1+3), 5 mL of HF, and then add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness and dissolve the residue in 1 mL of $HClO_4$ (1+1) and a minimum amount of water. Filter if necessary and add to the solution obtained in 309.3. Adjust the volume to approximately 200 mL or to a volume sufficient to cover the electrodes.

309.5 Add 10 mL of HNO_3 (1+1), and 1 g of sulfamic acid. Use weighed electrodes and a current density from 0.5 to 1 A/dm^2 of cathode surface, and electrolyze while stirring until the solution is colorless. Raise the level of the electrolyte 1 cm by adding water, add 1 g of sulfamic acid, and continue the electrolysis for an additional 30 min. If deposition occurs, add more water, and continue electrolysis until there is no further deposition.

309.6 Without interrupting the current, remove the beaker containing the electrolyte and quickly replace it with a beaker containing sufficient water to cover the electrodes completely. Remove the beaker, disconnect the cathode, and rinse in two successive beakers of ethanol. Dry in an oven at 110°C for 10 min, cool in a desiccator, and weigh as metallic copper. The electrolyte should not contain more than 0.2 µg/mL copper when tested with a sensitive method such as the neocuproine photometric or atomic absorption.

310. Calculation

310.1 Calculate the percentage of copper as follows:

$$\text{Copper, \%} = (A / B) \times 100 \tag{16}$$

where:

A = copper found, g, and
B = sample used, g.

311. Precision and Bias[19]

311.1 *Precision*—Eleven laboratories cooperated in testing sample ISO–9; nine laboratories tested ISO–12; and ten laboratories tested ISO–8. Each laboratory analyzed the samples on a minimum of six separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 18.

---

[19] Supporting data are available from ASTM Headquarters. Request RR:E01-1044.

TABLE 18 Statistical Information

Test Specimen	Copper Found, %	Repeatability (R_1, E173, M = 1)	Reproducibility (R_2, E173, M = 1)
ISO–9	10.03	0.107	0.262
ISO–12	5.44	0.100	0.183
ISO–8	1.03	0.048	0.082

311.2 *Bias*—The accuracy of this test method could not be evaluated because adequate certified standard reference materials were unavailable at the time of testing. The user is cautioned to verify by the use of certified reference materials, if available, that the accuracy of this test method is adequate for the contemplated use.

GALLIUM BY THE ION-EXCHANGE-ATOMIC ABSORPTION

312. Scope

312.1 This test method covers the determination of gallium in concentrations from 0.001 % to 0.05 %.

313. Summary of Test Method

313.1 Gallium is adsorbed on a column of strongly basic anion exchange resin from a 6 *M* hydrochloric acid solution of the sample. The adsorbed gallium is eluted from the column with 0.5 *M* hydrochloric acid and the concentrated eluate is aspirated into the nitrous oxide-acetylene flame of an atomic absorption spectrophotometer. The absorption of the gallium resonance line at 2874 Å is measured and compared with the absorption of calibration solutions containing known amounts of gallium.

314. Concentration Range

314.1 If the optimum concentration range is not known, determine it as directed in Guide E1024. A sensitivity of 0.5 to 0.8 µg/mL for 0.0044 absorbance is widely obtained.

315. Interferences

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

316. Apparatus

316.1 *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 %.

316.2 *Anion Exchange Column*—A glass column 20 mm in diameter and approximately 400 mm long, provided with a fritted disk and a stopcock. A reservoir for reagents may be added at the top of the column. However, reagents must be added in accordance with the procedure described in 320.1.

317. Reagents

317.1 *Anion Exchange Resin:*

317.1.1 Use a strongly basic anion exchange resin of the polystyrene-quaternary ammonium type, chloride form, having a cross linkage of 8 % and a 50 to 100 nominal mesh size.[20] Wash the resin with successive portions of water, decanting to remove "fines" until a clear supernatant solution is obtained.

317.1.2 *Preparation of the Ion Exchange Column*—Stir the resin slurry and transfer a sufficient amount of the suspension to the glass column to obtain a resin bed approximately 150 mm high after settling. Using a 19-mm outside diameter column, this is about 30 mL of wet resin. Precautions should be taken to avoid air bubbles or channels (Note 19). Wash the column with 150 mL of HCl (0.5 *M*), followed by 200 mL of water using a flow rate of 3 to 5 mL/min.

NOTE 19—**Precaution:** When not in use, the resin bed in the column should always be covered with water.

317.2 *Gallium Solution A (1 mL = 1.00 mg Ga)*—Transfer 0.500 g of gallium (purity: 99.9 % min) to a 250-mL beaker. Cover and add 10 mL of HCl (1+1). Cautiously add, in small amounts at a time, 2 mL of 30 % H_2O_2. When dissolution is complete, boil the solution for about 5 min. After cooling, add 33 mL of HCl (6 *M*) and transfer to a 500-mL volumetric flask. Dilute to volume and mix. Store in a polyethylene bottle.

317.3 *Gallium Solution B (1 mL = 0.1 mg Ga)*—Pipet 50 mL of Gallium Solution A into a 500-mL volumetric flask, add 33 mL of HCl (6 M), dilute to volume, and mix. Store in a polyethylene bottle.

317.4 *Hydrochloric Acid* (6 *M*)—Add 510 mL of HCl to water and dilute to 1 L.

317.5 *Hydrochloric Acid* (4 *M*)—Add 340 mL of HCl to water and dilute to 1 L.

317.6 *Hydrochloric Acid* (0.5 *M*)—Dilute 125 mL of HCl (4 *M*) with water and make up to 1 L.

318. Hazards

318.1 Dilute acid concentrations are expressed as molarities in the sections pertaining to the ion exchange steps to emphasize the need for careful dilution of concentrated acids. Standardization is not required.

319. Calibration

319.1 *Calibration Solutions*—Using pipets, transfer 10, 20, 30, 40, and 50 mL of Gallium Solution B to 100-mL volumetric flasks. Dilute to volume with HCl (0.5 *M*) and mix. Store in polyethylene bottles.

319.2 *Reference Solution*—Use HCl (0.5 *M*) as the reference solution.

319.3 Since sensitivity may vary among instruments, determine the suitability of the selected concentration range and apparatus in accordance with Guide E1024.

320. Procedure

320.1 *Test Solution:*

---

[20] Dowex 1-X8 manufactured by Dow Chemical Co., Midland, MI, and AG 1-X8 produced by Bio-Rad, Richmond, CA, have been found satisfactory for this purpose.

320.1.1 Transfer a 1.0-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Add 10 mL of water and cover with a ribbed cover glass. Carefully add 30 mL of HC1 (1+1) in small increments. When the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min. If no precipitate is observed, proceed as directed in 320.1.3.

320.1.2 When a visible silicon residue is present, filter through a 3.0-µm membrane filter into a 250-mL beaker. Wash any residue with hot water and reserve the filtrate. Transfer the membrane filter and residue to a platinum crucible. Moisten the filter with 1 mL HNO_3 (1+1) and heat on a hot plate until the membrane chars. Ignite in a muffle furnace at 500°C to 600°C until the carbon is removed. Cool, add 5 mL of HF, and add HNO_3 dropwise until a clear solution is obtained. Evaporate to dryness, cool, and dissolve the residue in 1 mL of HCl (1+1) and a minimum amount of water. Heat to dissolve the salts and add the solution to the reserved filtrate.

320.1.3 Evaporate the solution from 320.1.1 or 320.1.2, using a ribbed cover glass, to a volume of about 10 mL or until heavy crystallization occurs. Add 40 mL of HCl (6 *M*) and warm to dissolve the salts.

320.1.4 Condition the anion exchange resin by passing 50 mL of HCl (6 *M*) through the column at a flow rate of 3 to 5 mL/min.

320.1.5 Transfer the cooled solution from 320.1.3 to the reservoir of the column and rinse the beaker with small portions of HCl (6 *M*). Pass the test solution through the column at a flow rate of 3 to 5 mL/min. Wash the column reservoir three times with 10-mL portions of HCl (6 *M*). Allow each wash to drain before adding the next.

320.1.6 Wash the column with 200 mL of HCl (4 *M*) maintaining the same flow rate. This step separates all the aluminum and those metals not adsorbed by anion exchange, plus most of the copper, if present. Discard the eluate and washes.

320.1.7 Elute the gallium with 150 mL HCl (0.5 *M*) at a flow rate of 3 to 5 mL/min, collecting the eluate in a 250-mL beaker. Cover with a ribbed cover glass and evaporate just to dryness.

NOTE 20—The only elements present in this eluate are gallium, iron, and, for copper-containing alloys, a small amount of residual copper.

320.1.8 Dissolve the residue in 2 to 3 mL of HCl (0.5 *M*) with gentle heating. Cool and transfer to a 10-mL volumetric flask using HCl (0.5 *M*). Dilute to volume with HCl (0.5 *M*) and mix. Complete the analysis as described in 321.1.

320.1.9 To prepare for the next determination, wash the column with 200 mL of hot water (boiling) using a flow rate of 5 to 7 mL/min to elute any zinc. Under these conditions, the resin bed will maintain a temperature of about 50°C. If high-zinc alloys, such as the 7000 series, have been analyzed, wash with a second 200-mL portion of hot water (Note 21).

NOTE 21—For alloys containing bismuth, recondition the column in a manner similar to that described in Note 16. The final conditioning should be done in accordance with 320.1.4.

321. Measurements

321.1 Optimize the response of the instrument and take preliminary readings using the standards prepared in 319.1. Complete the analysis and calculate the gallium concentration using one of the procedures described in Guide E1024.

322. Calculation

322.1 Calculate the percentage of gallium as follows:

$$\text{Gallium},\% = \frac{A}{B} \times 100 \qquad (17)$$

where:

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

323. Precision and Bias[21]

323.1 *Precision*—Eight laboratories cooperated in testing this test method by analyzing each of the samples on three separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 19.

TABLE 19 Statistical Information

Test Specimen	Gallium Found, %	Repeatability (R_1, E173, M = 1)	Reproducibility (R_2, E173, M = 1)
Alcoa Standard GA-2-A, 1075 Alloy (0.005 % Ga)	0.0053	0.00049	0.00050
NBS 85b 2024 Alloy (0.019 % Ga)	0.0204	0.00108	0.00258

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

LITHIUM BY THE ATOMIC ABSORPTION TEST METHOD

324. Scope

324.1 This test method covers the determination of lithium in concentrations from 0.001 % to 4 %.

325. Summary of Test Method

325.1 An acid solution of the sample is aspirated into the air-acetylene flame of an atomic absorption spectrophotometer. The absorption of the lithium resonance line at 670.8 nm is measured and compared with the absorption of matched matrix calibration solutions containing known amounts of lithium.

326. Concentration Range

326.1 If the optimum concentration range is not known, determine it in accordance with Guide E1024. A sensitivity of 0.035 µg/mL for 0.0044 absorbance is widely obtained.

---

[21] Supporting data are available from ASTM Headquarters. Request RR:E01-1050.

327. Interferences

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

328. Apparatus

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

329. Reagents

329.1 *Aluminum Solution A* (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 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.

329.2 *Aluminum Solution B* (1 mL = 5.0 mg Al)—Pipet 10 mL of Aluminum Solution A into a 100-mL volumetric flask. Dilute to volume, and mix. Store in a polyethylene bottle.

329.3 *Lithium Standard Solution A* (1 mL = 1.00 mg Li)—Dry a quantity of lithium carbonate (Li_2CO_3) (purity: 99.5 % min), for 12 h at 140°C. Weigh 5.324 g into a beaker and cover with 20 mL of water. Dissolve by adding HCl (1+1), dropwise. Add an additional 10 mL of HCl (1+1) and heat gently to remove CO_2. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store in a polyethylene bottle.

329.4 *Lithium Standard Solution B* (1 mL = 0.040 mg Li)—Pipet 10 mL of Lithium Standard Solution A into a 250-mL volumetric flask, dilute to volume, and mix. Prepare fresh as needed.

329.5 *Lithium Standard Solution C* (1 mL = 0.020 mg Li)—Pipet 10 mL of Lithium Standard Solution A into a 500-mL volumetric flask, dilute to volume, and mix. Prepare fresh as needed.

329.6 *Lithium Standard Solution D* (1 mL = 0.008 mg Li)—Pipet 20 mL of Lithium Standard Solution B into a 100-mL volumetric flask, dilute to volume, and mix. Prepare fresh as needed.

330. Calibration

330.1 *Calibration Solutions:*

330.1.1 *0.002 % to 0.02 % Li*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Lithium Standard Solution D to 100-mL volumetric flasks containing 5 mL of HCl (1+1). Add 20 mL of Aluminum Solution A to each flask, cool, dilute to volume, and mix.

330.1.2 *0.02 % to 0.5 % Li*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Lithium Standard Solution C to 100-mL volumetric flasks containing 8.5 mL of HCl (1+1). Add 2 mL of Aluminum Solution A to each flask, dilute to volume, and mix.

330.1.3 *0.5 % to 4 % Li*—Using pipets, transfer 0, 5, 10, 15, 20, and 25 mL of Lithium Standard Solution B to 200-mL volumetric flasks containing 17 mL of HCl (1+1). Add 5 mL of Aluminum Solution B to each flask, dilute to volume, and mix.

330.2 *Reference Solution*—The zero lithium addition calibration solution is used for the reference solution for each range of calibration.

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

331. Procedure

331.1 *Test Solution:*

331.1.1 Transfer a 1.000-g sample, weighed to the nearest 1 mg, to a 400-mL beaker. Add 20 mL of water and cover with a ribbed cover glass. Add 27 mL of HCl (1+1) in small increments. Warm, if necessary, to complete dissolution. When the reaction subsides, add 2 mL of H_2O_2 (30 %) and boil for 5 min.

331.1.2 Filter through a medium-porosity paper into a 100-mL volumetric flask. Wash the filter with hot water, but be careful not to exceed about 85 mL total filtrate. Reserve the filtrate.

331.1.3 When a visible residue is present, transfer the filter paper and residue to a platinum crucible, dry, and ignite at 600°C. Cool, add 5 mL of HF, and then add HNO_3 dropwise until a clear solution is obtained. Evaporate carefully to dryness, cool, and dissolve the residue in 5 drops of HCl (1+1) and a minimum amount of water. Heat to dissolve the salts, and add this solution to the filtrate reserved in 331.1.2.

331.1.4 Cool the solution from 331.1.2 or the combined solutions from 331.1.3. Dilute to volume and mix.

331.1.5 For lithium concentrations less than 0.02 %, aspirate the solution from 331.1.4. Use calibration solutions prepared in accordance with 330.1.1.

331.1.6 For lithium concentrations between 0.02 % and 0.5 %, pipet 10 mL of the solution obtained in 331.1.4 into a 100-mL volumetric flask containing 8 mL of HCl (1+1), cool, dilute to volume, and mix. Use calibration solutions prepared in accordance with 330.1.2.

331.1.7 For lithium concentrations between 0.5 % and 4.0 %, prepare a solution as described in 331.1.6. Pipet 25 mL into a 200-mL volumetric flask containing 15 mL of HCl (1+1), cool, dilute to volume, and mix. Use calibration solutions prepared in accordance with 330.1.3.

332. Measurements

332.1 Optimize the instrument response and take preliminary readings; then complete the analysis and determine the lithium concentration using the graphical or ratio procedure in accordance with Guide E1024. The graphical procedure is preferred above 0.02 % lithium.

333. Calculation

333.1 Calculate the percentage of lithium as follows:

$$\text{Lithium,\%} = \frac{A}{B} \times 100 \quad (18)$$

where:

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

334. Precision and Bias

334.1 *Precision*—Eight laboratories cooperated in testing this test method by analyzing each of the samples on three separate days. Repeatability (R_1) and reproducibility (R_2) were calculated by analysis of variance (Practice E173) using M = 1. The data obtained are summarized in Table 20.

TABLE 20 Statistical Information

Test Specimen	Lithium Found, %	Repeatability (R_1, E173, M = 1)	Reproducibility (R_2, E173, M = 1)
1075 Alloy	0.00627	0.000563	0.000673
2020 Alloy	0.9567	0.02615	0.05251
1.5 Cu, 1.1 Mg, 3.5 Li Alloy	3.457	0.0431	0.1340

334.2 *Bias*—No certified reference materials suitable for testing this test method were available when the interlaboratory testing program was conducted. The user of this test method is encouraged to employ accepted reference materials, if available, to determine the accuracy of this test method as applied in a specific laboratory.

335. Keywords

335.1 aluminum and aluminum alloys; analysis; chemical analysis

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the ASTM website (www.astm.org/COPYRIGHT/).