~~Designation:E34–94(Reapproved2002)~~ Designation: E34 – 11

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

This standard is issued under the fixed designation E34; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

1. Scope

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

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

1.2 The analytical procedures appear in the following ~~order:~~ sections:

~~Sections~~

~~Beryllium by Argon Plasma Optical Emission Spectroscopy~~

Procedure	Sections
Beryllium:	
Beryllium by Argon Plasma Optical Emission Spectroscopy	283 to 292 ~~Beryllium by the Morin (Fluorometric) Test Method~~
~~Bismuth by the Thiourea (Photometric) Method~~ 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~~

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[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.

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

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

Procedure	Sections
Bismuth and Lead by the Atomic Absorption Test Method	188 to 198 ~~Boron by the Carmine (Photometric) Test Method~~
~~Cadmium by the Atomic Absorption Test Method~~	
Boron:	
Boron by the Carmine (Photometric) Test Method	[1e]
Cadmium:	
Cadmium by the Atomic Absorption Test Method	167 to 177 ~~Chromium:~~
~~Diphenylcarbazide (Photometric) Test Method~~	~~39 to 47~~
~~Persulfate Oxidation (Titrimetric) Test Method~~	
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 and Lead by the Electrolytic (Gravimetric) Test Method~~
Chromium by the Atomic Absorption Test Method	199 to 209 ~~Copper and Lead by the Electrolytic (Gravimetric) Test Method~~
Copper:	
Copper and Lead by the Electrolytic (Gravimetric) Test Method	[1c]
~~Copper and Zinc by the Atomic Absorption Test Method~~	~~210 to 220~~
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 Electrolytic (Gravimetric) Test Method	303 to 311
~~Copper by the Neocuproine (Photometric) Test Method~~	[1a] ~~Gallium by the Ion Exchange-Atomic Absorption Test Method~~
Copper by the Neocuproine (Photometric) Test Method	[1a] ~~Gallium by the Ion Exchange-Atomic Absorption Test Method~~
Gallium:	
Gallium by the Ion Exchange-Atomic Absorption Test Method	312 to 323
~~Iron~~ ~~**Iron:**~~	
Iron:	
Iron by the 1,10-Phenanthroline (Photometric) Method	73 to 81
~~Iron and Manganese by the Atomic Absorption Method~~	~~221 to 231~~ ~~Lithium by the Atomic Absorption Test Method~~
Iron and Manganese by the Atomic Absorption Spectometry Method	221 to 231 ~~Lithium by the Atomic Absorption Test Method~~
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:~~

~~Pyrophosphate (Gravimetric) Method~~	
Magnesium:	
Magnesium by the Pyrophosphate (Gravimetric) Method	[1b] ~~Ethylenediamine Tetraacetate (Titrimetric) Test Method~~
~~Magnesium by the Atomic Absorption Test Method~~	
Magnesium by the Ethylenediamine Tetraacetate (Titrimetric) Test Method	[1e]
Magnesium by the Atomic Absorption Spectrometry Test Method	232 to 242 ~~Manganese by the Periodate (Photometric) Test Method~~
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:~~
~~Dimethylglyoxime (Photometric) Test Method~~	
Nickel:	
Nickel by the Dimethylglyoxime (Photometric) Test Method	[1a] ~~Dimethylglyoxime (Gravimetric) Test Method~~
Nickel by the Dimethylglyoxime (Gravimetric) Test Method	[1b] ~~Nickel by the Atomic Absorption Test Method~~
Nickel by the Atomic Absorption Spectrometry Test Method	243 to 253 ~~Silicon:~~
~~Molybdisilicic Acid (Photometric) Test Method~~	~~118 to 127~~
~~Sodium Hydroxide-Perchloric Acid (Gravimetric) Method~~	~~128 to 133~~
~~Tin by the Iodate (Titrimetric) Test Method~~	~~134 to 140~~
~~Titanium by the Chromotropic Acid (Photometric) Test Method~~	
Silicon:	
Silicon by the Molybdisilicic Acid (Photometric) Test Method	[1e]
Silicon by the Sodium Hydroxide-Perchloric Acid (Gravimetric) Method	[1e]
Tin:	
Tin by the Iodate (Titrimetric) Test Method	[1e]
Titanium:	
Titanium by the Chromotropic Acid (Photometric) Test Method	141 to 150 ~~Titanium by the Diantipyrylmethane Photometric Test Method~~
~~Titanium by the Diantipyrylmethane Photometric Test Method~~	~~254 to 263 Vanadium by an Extraction-Photometric Test Method using~~
Titanium by the Diantipyrylmethane Photometric Test Method	254 to 263
Vanadium:	
Vanadium by an Extraction-Photometric Test Method using *N*-Benzoyl-*N*-Phenylhydroxylamine	264 to 273 ~~Zinc:~~
~~Ammonium Mercuric Thiocyanate or the Zinc Oxide (Gravimetric) Test Method~~	
Zinc:	
Zinc by the Ammonium Mercuric Thiocyanate or the Zinc Oxide (Gravimetric) Test Method	[1b] ~~Ethylenediamine Tetraacetate (Titrimetric) Test Method~~
Zinc by the Ethylenediamine Tetraacetate (Titrimetric) Test Method	[1d] ~~Ion Exchange-EDTA Titrimetric Test Method~~

Copper and Zinc by the Atomic Absorption Spectrometry Test Method	210 to 220
Zinc by the Ion Exchange-EDTA Titrimetric Test Method	274 to 282
Zirconium:	
Zirconium by the Arsenazo III (Photometric) Method	178 to 187

~~1.3The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.~~

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

1.4 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given throughout these test methods.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

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

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

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

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

E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals[34]

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

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

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

~~3.1These~~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.

~~4.~~

5. Apparatus, Reagents, and Photometric Practice

~~4.1Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E50, except that photometers shall conform to the requirements prescribed in 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.

~~4.2Photometric practice prescribed in these test methods shall conform to Practice E60~~

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

~~5.~~

6. Precautions

~~5.1For~~6.1 For precautions to be observed in the use of certain reagents in these test methods, reference shall be made to Practices E50.

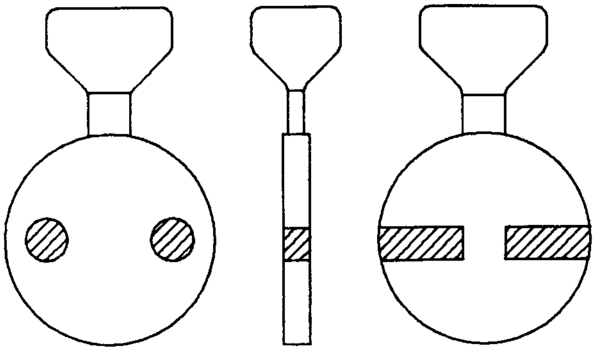
~~[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For~~

[2] *Annual Book of ASTM Standards*~~volume information, refer to the standard's Document Summary page on the ASTM website.~~, Vol 14.02.

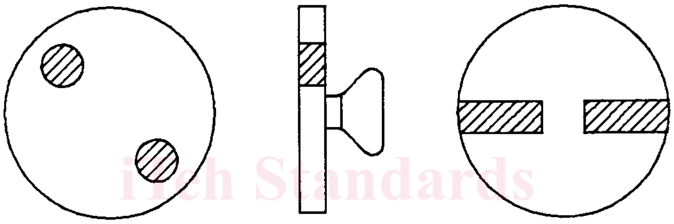
[3] *Annual Book of ASTM Standards*, Vol 03.05.

~~[4] Olson, H. A., and Macy, D. W., "Metallurgical Approach to Evaluating Chemical Sample Disks," *Light Metals*, Vol 2, 1978, pp. 301–311.~~

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



Type A Mold



Type B Mold

NOTE 1—Shaded areas are suitable for sampling.

FIG. 1 Type A and Type B Disks

~~6.~~

7. Sampling

~~6~~7.1 Wrought products shall be sampled in accordance with Practice E55. Cast products shall be sampled in accordance with Practice E88.

~~6.2Chill~~7.2 Chill cast disks produced for analysis by spectrochemical methods (see Practices E716~~) shall be considered cast products. The principles of Practice E88 shall apply.~~

~~6.2.1Prepare such disks for chemical analysis by drilling or milling through the entire thickness of an unmachined disk (Note 1). Use a minimum of two positions approximately opposite each other and combine the drillings or millings.~~) 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.

~~6.2.2The outer edges of the holes shall be approximately 0.48 cm (3⁄16 in.) from the edge of the disk. Drills shall be not less than 0.95 cm (3⁄8~~

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.[2]~~

~~6.2.3Mill disks at similar points to a distance of 40% of the sample diameter. Use a 0.95-cm (3⁄8 in.) milling cutter.[2]~~

~~6.2.4Sample book mold disks (Type A, Practices~~ in.) in diameter.[5]

~~[5] National Bureau of Standards Sample No. 44c (melting point aluminum) has been found satisfactory for this purpose.~~

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

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 (⅜ in.) milling cutter has been shown to provide acceptable chips.[5]

7.2.4 Center pour (Type B, Practices E716~~) at approximately the 9 o'clock and the 3 o'clock positions when the sprue is at the 12 o'clock position. 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 ~~A and Type~~ B disks. Vacuum cast disks are sampled in the same manner as Type B disks.[5]

~~6~~7.2.5 Drilling or milling techniques ideally should produce uniformly small chips. Break large continuous pieces into smaller pieces 0.64 cm (¼ in.) to 0.95 cm (⅜ ~~in.) long. Drilling or milling techniques should minimize production of fine, dust-like material.[2]~~

~~7.Rounding Calculated Values~~

~~7.1Calculated values shall be rounded to the desired number of places in accordance with the rounding method given in 3.4 and 3.5 of Practice E29.~~ in.) long. Drilling or milling techniques should minimize production of fine, dust-like material.[5]

BERYLLIUM BY THE MORIN (FLUOROMETRIC) TEST METHOD

~~8.Scope~~

~~8.1This test method covers the determination of beryllium in concentrations from 1 to 100 ppm.~~

~~9.Summary of Test Method~~

~~9.1In an alkaline solution, beryllium and morin form a compound which produces a yellow-green fluorescence at approximately 520 nm when excited by light at approximately 430 nm.~~

~~10.Concentration Range~~

~~10.1The recommended concentration range is from 0.05 to 0.5 µg of beryllium in 25 mL of solution.~~

~~11.Stability of Fluorescence~~

~~11.1The fluorescence is stable for at least 1 h if the test solutions are kept at constant temperature.~~

~~12.Interferences~~

~~12.1Silicon must be removed by dehydration or volatilization. Beryllium is separated from interfering ions by an acetylacetone extraction. Fluoride and pyrophosphate interfere in the extraction.~~

~~13.Apparatus~~

~~13.1*Bottles*, plastic, 500-mL capacity.~~

~~13.2*Constant-Temperature Bath*, 20 or 25°C.~~

~~13.3*Fluorescence-Measuring Instrument*— A suitable filter fluorometer or spectrofluorometer with primary excitation at approximately 430 nm and a means of isolating and measuring the secondary fluorescent emission at approximately 520 nm.~~

~~14.Reagents~~

~~14.1*Acetylacetone (2,4-pentanedione)*, practical.~~

~~14.2*Aluminum Sulfate Solution (49 g/L)*—Dissolve 4.9 g of aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$) in 70 mL of water, add 1 mL of $HClO_4$, transfer to a 100-mL volumetric flask, dilute to volume, and mix.~~

~~14.3*Ammonium Nitrate Wash Solution (10 g/L)*—Dissolve 5 g of ammonium nitrate (NH_4NO_3) in water and dilute to 500 mL.~~

~~14.4*Beryllium, Standard Solution A (1 mL=100 µg Be)*—Dissolve 1.964 g of beryllium sulfate ($BeSO_4 \cdot 4H_2O$) in water, add 10 mL of $HClO_4$, transfer to a 1-L volumetric flask, dilute to volume, and mix. Standardize as follows: transfer a 100-mL aliquot of this solution to a 250-mL beaker, add NH_4OH (1+1) until a permanent turbidity forms, and then add 10 mL in excess. Allow to stand for 2 to 3 h or overnight. Filter using a low-ash, medium paper and wash well with NH_4NO_3 wash solution. Transfer the precipitate and paper to a weighed, covered platinum crucible and char. Finally ignite to constant weight at 1000°C and weigh as beryllium oxide (BeO).~~

~~14.5*Beryllium, Standard Solution B (1 mL=0.10 µg Be)*—Transfer a 10-mL aliquot of Beryllium Solution A to a 100-mL volumetric flask. Add 1 mL of $HClO_4$, dilute to volume, and mix. Transfer a 10-mL aliquot of this solution to a 1-L volumetric flask. Add 10 mL of $HClO_4$, dilute to volume, and mix.~~

~~14.6*Chloroform*($CHCl_3$).~~

~~14.7*Diethylenetriaminepentaacetic Acid* (*DTPA*), *Recrystallized* (Note 2)—Add 100 g of DTPA to 800 mL of boiling water and heat to boiling. Add 8 to 10 g of high-quality decolorizing carbon and mix thoroughly. Filter with suction through a hardened~~

~~retentive paper. Transfer the clear filtrate to a 1-L beaker and stir vigorously to induce crystallization throughout the solution. Cool thoroughly in a water bath. Filter with suction using a hardened paper and remove as much water as possible. Dry the salts at 110°C.~~

~~NOTE 2—It is essential that the DTPA salts be recrystallized since commercial DTPA contains impurities that absorb ultraviolet and visible light. These impurities produce bright blue fluorescence under ultraviolet light, and react under certain conditions to produce a bright yellow fluorescence.~~

~~14.8 *Disodium Ethylenediaminetetraacetate (EDTA) Solution* (100 g/L)—Dissolve 100 g of EDTA in water and dilute to 1 L.~~

~~14.9 *EDTA Wash Solution*—Add 5 mL of H_2SO_4 and 10 mL of the EDTA solution to 300 mL of water. Add 2 drops of phenol red indicator solution and NH_4OH until the solution turns red. Cool and dilute to 500 mL.~~

~~14.10 *Morin Solution (0.075 g/L)*—Dissolve 0.0075 g of anhydrous morin (3,5,7,2′,4′-penta-hydroxyflavone), reagent grade, in 40 mL of ethanol. Transfer to a 100-mL volumetric flask with water, dilute to volume, and mix.~~

~~14.11 *Phenol Red Indicator Solution (1 g/L)*—Dissolve 0.1 g of phenol red in 50 mL of methanol and dilute to 100 mL with water.~~

~~14.12 *Piperidine Buffer Solution*—Dissolve 15 g of the recrystallized DTPA in 200 mL of water. Add 75 mL of redistilled piperidine and cool. Add 20 g of anhydrous sodium sulfite (Na_2SO_3) and dilute to 500 mL. Store in a plastic bottle. This solution slowly decomposes and should be discarded after 6 months.~~

~~14.13 *Potassium Iodide-Starch Paper*.~~

~~14.14 *Quinine Sulfate Solution (0.1 g/L)*—Dissolve 0.1 g of quinine sulfate in 300 mL of water. Add 10 mL of $HClO_4$ and dilute to 1 L.~~

~~14.15 *Sodium Hydroxide-Sodium Perchlorate-DTPA-Triethanolamine (TEA) Solution*—Dissolve 60 g of sodium hydroxide (NaOH) and 320 g of anhydrous sodium perchlorate ($NaClO_4$) in 250 mL of water. Filter through a double 7-cm glass-fiber filter. Add 10 mL of TEA solution (20 volume%) to 13.0 g of the recrystallized DTPA and dissolve in 50 mL of water and about 20 mL of the NaOH-$NaClO_4$ solution. When dissolution is complete, add the remainder of the NaOH-$NaClO_4$ solution and dilute to 500 mL. Store in a plastic bottle. Acidify a small portion of the solution and test for oxidizing agents with the potassium iodide-starch paper. If the presence of an oxidizing agent is indicated, add sodium sulfite (Na_2SO_3) in small portions until the oxidizing agent is destroyed.~~

~~14.16 *Sodium Hydroxide Solution (40 g/L)*—Dissolve 20 g of NaOH in water and dilute to 500 mL. Store in a plastic bottle.~~

~~15. Hazards~~

~~15.1 Beryllium and its compounds are extremely toxic and care must be exercised in the handling of these compounds. Hot perchloric acid and organic compounds present the possibility of an explosion if not handled carefully.~~

~~16. Preparation of Calibration Curve~~

~~16.1 *Calibration Solutions*—Transfer 0.5, 1.0, 3.0, 5.0, 7.0, and 9.0 mL of Beryllium Solution B (1 mL=0.10 µg Be) to six 25-mL volumetric flasks. Proceed as directed in 16.3.~~

~~16.2 *Reference Solution*—Transfer 5 mL of water to a 25-mL volumetric flask and proceed as directed in 16.3.~~

~~16.3 *Fluorescence Development*:~~

~~16.3.1 Add 1 mL of $Al_2(SO_4)_3$ solution, 3 mL of NaOH-$NaClO_4$-DTPA-TEA solution, and 3 drops of quinine sulfate solution.~~

~~16.3.2 Add $HClO_4$ dropwise until the solution fluoresces under an ultraviolet light, then add 1 drop in excess. Swirl the contents of the flask so that any beryllium that may have been deposited as the hydroxide on the wall of the flask is redissolved.~~

~~16.3.3 Add NaOH solution dropwise until the fluorescence is destroyed and then add 2 drops in excess. Rinse down the neck of the flask with water.~~

~~16.3.4 Add 5 mL of piperidine buffer solution and mix.~~

~~16.3.5 Add 1.00 mL of morin solution, dilute to volume, and mix. Place the flask in the constant-temperature bath for 20 min.~~

~~16.4 *Fluorometry*:~~

~~16.4.1 Following the manufacturer's operating instructions, adjust the fluorometer so that the calibration solution is excited by light at approximately 430 nm, and its fluorescence is detected at approximately 520 nm.~~

~~16.4.2 Transfer a suitable portion of the reference solution to the cuvette and adjust the fluorometer to the initial setting. While maintaining this adjustment, take the fluorometric readings of the calibration solutions.~~

~~16.5 *Calibration Curve*—Plot the fluorometric readings of the calibration solutions against micrograms of beryllium per 25 mL of solution.~~

~~17. Procedure~~

~~17.1 *Test Solution*:~~

~~17.1.1 Transfer 1.00 g of the sample, weighed to the nearest 1 mg, to a 250-mL beaker. Add 25 mL of water and 10 mL of HCl. Warm the solution, if necessary, to hasten dissolution. When dissolution is complete, boil for a few minutes.~~

~~17.1.2 Add 5 mL of HNO_3 and 20 mL of $HClO_4$ and evaporate to the appearance of $HClO_4$ fumes. Reduce the heat to avoid bumping and continue fuming for 10 min. Cool to room temperature. Add 100 mL of water and heat to dissolve the salts. Filter through a medium paper into a 500-mL volumetric flask. Wash the paper and precipitate with 10 mL of H_2SO_4(1+4) followed by several washings with hot water. Cool to room temperature, dilute to volume, and mix.~~

~~17.1.3Transfer an aliquot containing an amount of beryllium that is in the linear portion of the calibration curve, but not containing more than 70 mg of aluminum, to a 150-mL beaker. Add 10 mL of EDTA solution and adjust the volume to approximately 40 mL. Add 2 drops of phenol red indicator solution and NH_4OH until the solution turns red. Add 5 drops of $HClO_4$, cover the beaker with a watch glass, and boil for 5 min (Note 3). Cool to room temperature.~~

~~NOTE3—The aluminum-EDTA complex is slow in forming and, therefore, requires the 5-min boiling period. If the complex is not formed, an emulsion will result in the extraction procedure causing erroneous results. Ten millilitres of EDTA solution will chelate approximately 70 mg of .~~

~~17.1.4Transfer the solution to a 125-mL separatory funnel. A standard containing an amount of beryllium that is in the linear portion of the calibration curve must be carried through the extraction for calibration purposes. Add 10 drops of acetylacetone, stopper the funnel, and mix by shaking. Add NH_4OH dropwise until the solution turns red and add 5 drops in excess. Add 10 mL of $CHCl_3$ and shake vigorously for 2 min; allow the phases to separate. Transfer the $CHCl_3$ (lower) layer to a clean 125-mL separatory funnel and reserve. Repeat the extraction from the aqueous layer with 4 drops of acetylacetone and 10 mL of $CHCl_3$ and add the $CHCl_3$ layer to the reserved one. Add 20 mL of EDTA wash solution to the reserved $CHCl_3$ extracts, shake for 30 s, and allow the phases to separate. Drain the $CHCl_3$ layer into a 100-mL beaker. Add 2 drops of acetylacetone and 10 mL of $CHCl_3$ to the EDTA wash solution layer, shake for 30 s, and add the $CHCl_3$ extracts to those in the 100-mL beaker.~~

~~17.1.5Add 3 mL of HNO_3 and 5 mL of $HClO_4$. Cover the beaker with a watchglass and volatilize the $CHCl_3$ at a temperature just sufficient to boil the $CHCl_3$. When volatilization of the $CHCl_3$ is complete, increase the heat and allow the solution to boil until $HClO_4$ fumes appear. This refluxing action will dissolve materials adhering to the walls of the beaker. Cool, remove the watchglass after rinsing with water, and evaporate the solution to a volume of 0.5 mL. Do not bake or allow the beaker to go dry during the evaporation. Cool, add a few drops of water, warm to dissolve salts, and cool again. Transfer the solution to a 25-mL volumetric flask, keeping the volume in the flask to approximately 10 mL.~~

~~17.2*Reference Solution*—Carry a reagent blank through the entire procedure, using the same amounts of all reagents, for use as a reference solution.~~

~~17.3*Fluorescence Development*—Proceed as directed in 16.3.~~

~~NOTE4—If the solution fluoresces after the addition of the reagents in 16.3.1, neutralize the excess acid with NaOH solution (50%) dropwise until the fluorescence disappears. This neutralization step will keep the solution within the 25-mL volume.~~

~~17.4*Fluorometry*—Take the fluorometric readings of the standard beryllium and test solutions as directed in 16.4.~~

~~NOTE5—If the beryllium standard differs significantly from the calibration curve, the test solution and the beryllium standard must be rerun.~~

~~18.Calculation~~

~~18.1Convert the fluorometric reading of the test solution to micrograms of beryllium by means of the calibration curve. Calculate the parts per million of beryllium as follows:~~

~~(1) $\text{Beryllium, ppm} = A/B$~~

~~where:~~
~~*A* = beryllium found in 25 mL of the final solution, µg, and~~
~~*B* = sample represented in 25 mL of the final solution, g.~~

~~19.Precision~~

~~19.1Due to the limited number of laboratories participating in the cooperative testing of this test method, a precision statement conforming to the requirements of Practice E173 cannot be furnished. However, in a cooperative program conducted by three laboratories, the between-laboratory range was from 8.4 to 8.5 ppm beryllium on a sample averaging 8.5 ppm, and from 13.5 to 14.2 ppm on a sample averaging 13.8 ppm.~~

~~(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~~

~~where:~~
~~*A* = boron found in 22 mL of the final solution, mg, and~~
~~*B* = sample represented in 22 mL of the final solution, g.~~

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

CHROMIUM BY THE DIPHENYLCARBAZIDE ~~(PHOTOMETRIC) TEST METHOD~~

~~39. Scope~~

~~39.1 This test method covers the determination of chromium in concentrations from 0.01 to 0.30%.~~

~~40. Summary of Test Method~~

~~40.1 After dissolution of the sample in acids, chromium is oxidized to the hexavalent state in fuming perchloric acid. Hexavalent chromium forms a soluble red-violet complex with diphenyl-carbazide. Photometric measurement is made at approximately 540 nm.~~

~~41. Concentration Range~~

~~41.1 The recommended concentration range is from 0.004 to 0.08 mg of chromium per 100 mL of solution, using a cell depth of 2 cm (see Note 6).~~

~~42. Stability of Color~~

~~42.1 The color of the chromium diphenylcarbazide complex develops almost immediately but starts to fade within a short period of time. Photometric measurement must be made within 5 min after adding the diphenylcarbazide.~~

~~43. Interferences~~

~~43.1 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. Oxidizing agents cause rapid fading of the chromium diphenylcarbazide complex.~~

~~44. Reagents~~

~~44.1 *Aluminum or Aluminum-Base Alloy, Low-Chromium*— Aluminum or aluminum-base alloy containing less than 0.005% chromium.~~

~~44.2 *Chromium, Standard Solution* (1 mL=0.08 mg Cr)—*Prepare as directed for Reagent No. 10, using 0.2263 g instead of the specified weight.*~~

~~44.3 *Diphenylcarbazide Solution* (10 g/L)—*Dissolve 0.20 g of diphenylcarbazide in 20 mL of methanol. This solution decomposes within a few hours and must be prepared fresh as needed.*~~

~~44.4 *Sodium Chloride Solution* (2 g/L)—*Dissolve 2 g of sodium chloride (NaCl) in water and dilute to 1 L.*~~

~~45. Preparation of Calibration Curve~~

~~45.1 *Calibrations Solutions:*~~

~~45.1.1 Transfer 1.0, 2.0, 5.0, 10.0, 15.0, and 20.0 mL of chromium solution (1 mL=0.08 mg Cr) to six 300-mL Erlenmeyer flasks and dilute to 20 mL with water. Add 0.500 g of low-chromium aluminum, 7 mL of $HClO_4$, and 10 mL of H_2SO_4.~~

~~45.1.2 Allow to react without applying heat. When the reaction is complete, wash down the sides of the flask with a minimum of water, and add 5 mL of HNO_3 and 2 mL of HF. Heat to complete the dissolution of any metal sponge and silicon, and continue heating to copious white fumes (Note 8). Cool somewhat, add 30 mL of water, and heat to dissolve the soluble salts. Add 5 mL of NaCl solution (Note 9) and boil for 5 min to remove any chlorine formed by the presence of oxidized manganese. Cool, transfer to a 100-mL volumetric flask, dilute to volume, and mix. Proceed as directed in 45.3.~~

~~Note 8—In order to oxidize the chromium completely to chromate, it is necessary to heat the solution through the stage where copious white fumes of $HClO_4$ are evolving and refluxing. This operation may be carried out over a free flame; avoid heating to dryness.~~

~~Note 9—In some cases, manganese is precipitated as manganese dioxide (MnO_2) after fuming. The NaCl solution is added to dissolve any MnO_2 that might be present.~~

~~45.2 *Reference Solution*—Transfer 0.500 g of low-chromium aluminum to a 300-mL Erlenmeyer flask. Add 20 mL of water, 7 mL of $HClO_4$, and 10 mL of H_2SO_4. Proceed as directed in 45.1.2 and 45.3.~~

~~45.3 *Color Development*—Transfer a 5-mL aliquot to a 100-mL volumetric flask. Dilute to about 90 mL, add 1 mL of diphenylcarbazide solution, dilute to volume, and mix.~~

~~45.4 *Photometry*—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 540 nm. While maintaining this adjustment, take the photometric readings of the calibration solutions.~~

~~45.5 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of chromium per 100 mL of solution.~~

~~46. Procedure~~

~~46.1 *Test Solution*—Transfer 0.500 g of the sample, weighed to the nearest 1 mg, to a 300-mL Erlenmeyer flask. Add in the following order (**Precaution:** see Note 10), 20 mL of water, 7 mL of $HClO_4$, and 10 mL of H_2SO_4. Proceed as directed in 45.1.2.~~

~~NOTE 10—**Precaution:** The order of adding the reagents used in decomposing the sample must be strictly adhered to. Under no circumstances should $HClO_4$ be added directly to the metallic aluminum. See the Safety Precautions Section of Practices E50 for a discussion of the hazards involved in the use of $HClO_4$.~~

~~46.2 *Reference Solution*—Carry a reagent blank through the entire procedure, using the same amount of all reagents, for use as a reference solution.~~

~~46.3 *Color Development*—Proceed as directed in 45.3~~

~~46.4 *Photometry*—Take the photometric reading of the test solution as directed in 45.4.~~

~~47. Calculation~~

~~47.1 Convert the photometric reading of the test solution to milligrams of chromium by means of the calibration curve. Calculate the percentage of chromium as follows:~~

~~(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~~

~~A~~

~~where:~~

~~A = iron found in 100 mL of the final solution, mg, and~~
~~B = sample represented in 100 mL of the final solution, g.~~

(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 ~~88. Scope~~

~~88.1 This test method covers the determination of magnesium in concentrations from 0.1 to 5.0%. The range may be extended by suitably varying the sample weight.~~

~~89. Summary of Test Method~~

~~89.1 Magnesium is separated from the bulk of the alloy constituents by treatment of the sample with sodium hydroxide in the presence of hydrogen peroxide and potassium cyanide. The magnesium hydroxide is dissolved in hydrochloric acid and any remaining aluminum is separated by precipitation with ammonium hydroxide. Manganese is oxidized with bromine and separated as manganese dioxide. Magnesium is then determined by titration with disodium ethylenediamine tetraacetate.~~

~~90. Interferences~~

~~90.1 The elements ordinarily present in aluminum and aluminum-base alloys, except calcium, do not interfere if their concentrations are under the maximum limits shown in 1.1. Calcium is rarely present in significantly high concentrations.~~

~~91. Reagents~~

~~91.1 *Bromine Water (Saturated).*~~

~~91.2 *Disodium Ethylenediamine Tetraacetate (EDTA), Standard Solution* (0.05 M)—*Dissolve 18.61 g of disodium ethylenediamine tetraacetate dihydrate (EDTA) in water, transfer to a 1-L volumetric flask, dilute to volume, and mix. Standardize as follows: transfer a 20-mL aliquot of magnesium solution (1 mL=1.00 mg Mg) to a 400-mL beaker. Add 40 mL of HCl (1+1) and neutralize with NH_4OH. Dilute to 250 mL with water, add 70 mL of NH_4OH, and proceed as directed in 92.1.3 and 92.2. Calculate the magnesium equivalent of the disodium ethylenediamine tetraacetate solution as follows:*~~

~~where:~~

~~A = EDTA solution required for titration of the test solution, mL,~~

~~B = EDTA solution required for titration of the blank, mL,~~

~~C = magnesium equivalent of the EDTA solution, g/mL, and~~

~~D = sample used, g.~~

(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 ~~118. Scope~~

~~118.1 This test method covers the determination of silicon in concentrations from 0.05 to 1.0%.~~

~~119. Summary of Test Method~~

~~119.1 Silicic acid in a true (not colloidal) solution reacts with molybdate to form a soluble yellow-colored molybdisilicic acid. Maximum color development occurs over a pH range from 1.1 to 1.3. Photometric measurement is made at approximately 400 nm.~~

~~120. Concentration Range~~

~~120.1 The recommended concentration range is from 0.05 to 1.0 mg of silicon per 100 mL of solution, using a cell depth of 2 cm (see Note 6).~~

~~121. Stability of Color~~

~~121.1 The color develops in less than 5 min and does not fade appreciably for at least 20 min.~~

~~122. Interferences~~

~~122.1 The elements ordinarily present in aluminum and aluminum-base alloys do not interfere.~~

~~NOTE12—If detergents are used for cleaning, all traces of the detergents must be removed by boiling with HCl followed by thorough rinsing. Many detergents contain phosphorus compounds, which also form a yellow color with ammonium molybdate, and would cause interference if not completely removed.~~

~~123. Apparatus~~

~~123.1 *Plastic Ware* (polyethylene, polypropylene, or tetrafluoroethylene):~~

~~123.1.1 *Beakers and Covers.*~~

~~123.1.2 *Bottles,* 1-L capacity.~~

~~123.1.3 *Graduated Cylinders,* 10-mL and 1-L capacity.~~

~~123.2 *Nickel Crucibles and Covers,* 50-mL capacity.~~

~~123.3 *Nickel or Stainless Steel Beakers and Covers,* 250-mL and 1-L capacity.~~

~~123.4 *pH Meter*—Any commercial instrument capable of determining pH in the range from 0 to 7 ± 0.1 is suitable.~~

~~124. Reagents~~

~~124.1 *Aluminum Nitrate Solution* ($Al(NO_3)_3$)—*Transfer 1.0 g of aluminum, low-silicon, to a 250-mL nickel beaker. Add 100 mL of NaOH solution and cover. Allow to react without applying heat until the reaction subsides, and then warm gently to complete the dissolution. Cool, and transfer to a 400-mL beaker containing 125 mL of* HNO_3 *(1+1). Cover and warm gently until the salts dissolve and the solution becomes clear. Cool, transfer to a 250-mL volumetric flask, dilute to volume, and mix.*~~

~~124.2 *Ammonium Molybdate Solution* (100 g/L)—*Dissolve 100 g of ammonium molybdate tetrahydrate* $((NH_4)_6Mo_7O_{24}\cdot 4H_2O)$ *in water and dilute to 1 L. Store in a plastic bottle. Filter before using. If a precipitate forms on standing, the solution should be discarded.*~~

~~124.3 *Silicon, Standard Solution* (1 mL=0.05 mg Si)—*Fuse 0.1070 g of anhydrous silicon dioxide* (SiO_2) *with 1.0 g of sodium carbonate* (Na_2CO_3) *in a covered platinum crucible. Cool, dissolve the melt completely in water in a plastic beaker, cover, and heat on a steam bath for ½ to 1 h. Cool, transfer to a 1-L volumetric flask, dilute to volume, and mix. Store the solution in a plastic bottle. If anhydrous* SiO_2 *is not available, the weight should be adjusted according to the actual silicon content of the* SiO_2 *used, as determined by gravimetric analysis.*~~

~~124.4 *Sodium Hydroxide Solution* (300 g/L)—Dissolve 300 g of sodium hydroxide (NaOH) in about 600 mL of water, using a nickel or stainless steel beaker. Cool and dilute to 1 L. Store in a plastic bottle.~~

~~125. Preparation of Calibration Curve~~

~~125.1 *Calibration Solutions*—Transfer 12.5 mL of $(NO_3)_3$ solution and 1.0 mL of HNO_3 (1+1) to seven 100-mL beakers. Transfer 1.0, 2.0, 4.0, 8.0, 12.0, 16.0, and 20.0 mL of silicon solution (1 mL=0.05 mg Si) to each of the 100-mL beakers. Dilute to about 80 mL and mix. Proceed as directed in 125.3.~~

~~125.2 *Reference Solution*—Transfer 12.5 mL of $Al(NO_3)_3$ solution and 1 mL of HNO_3 (1+1) to a 100-mL beaker, dilute to about 80 mL, and mix. Proceed as directed in 125.3.~~

~~125.3 *Color Development*—Using a pH meter, check the pH of the solution. If the pH is not within 1.1 to 1.3, adjust it to this range using HNO_3 (1+1) or NaOH solution as required. Add 10 mL of ammonium molybdate solution, transfer the solution to a 100-mL volumetric flask, dilute to volume, and mix. Let stand at least 5 min but no longer than 20 min before taking the photometric reading.~~

~~125.4 *Photometry*—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 400+. While maintaining this adjustment, take the photometric readings of the calibration solutions.~~

~~125.5 *Calibration Curve*—Plot the photometric readings of the calibration solutions against milligrams of silicon per 100 mL of solution.~~

~~126. Procedure~~

~~126.1 *Test Solution:*~~

~~126.1.1 Transfer 0.10 g of the sample, weighed to the nearest 0.1 mg, to a 50-mL nickel crucible, add 10.00 mL of NaOH solution, and cover. Allow to react without applying heat until the reaction subsides, wash down the cover and sides of the crucible with a minimum amount of water, and boil gently until the reaction is complete and the silicon completely oxidized (Note 13). Cool, dilute to approximately 30 mL with water, and transfer quantitatively to a 150-mL beaker containing 12.5 mL of HNO_3 (1+1). Cover and warm gently until the solution becomes clear (Notes 14 and 15). Cool, transfer to a 100-mL volumetric flask, dilute to volume, and mix.~~

~~NOTE13—With certain alloys it may be necessary to evaporate the caustic solution to pastiness in order to completely dissolve the silicon. Care should be taken to avoid mechanical loss by spattering.~~