



## Standard Specification for Copper-Beryllium Alloy Plate, Sheet, Strip, and Rolled Bar<sup>1</sup>

This standard is issued under the fixed designation B 194; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

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

<sup>€1</sup> NOTE—Referenced Documents were editorially corrected in November 2003.

### 1. Scope\*

1.1 This specification establishes the requirements for copper-beryllium alloy plate, sheet, strip, and rolled bar. The following alloys are specified:<sup>2</sup>

| Copper Alloy<br>UNS No. <sup>2</sup> | Previously Used Commercial<br>Designations | Nominal Beryllium<br>Content, % |
|--------------------------------------|--------------------------------------------|---------------------------------|
| C17000                               | Alloy 165                                  | 1.7                             |
| C17200                               | Alloy 25                                   | 1.9                             |

1.2 Unless otherwise required, Copper Alloy UNS No. C17200 shall be the alloy furnished whenever Specification B 194 is specified without any alloy designation.

1.3 *Units*—With the exception of Grain Size values, the values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units, which are provided for information only and not considered standard.

1.4 The following hazard statement pertains only to the test method portions in the annex of this specification:

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 The following documents of the issue in effect on date of material purchase form a part of this specification to the extent referenced herein:

#### 2.2 ASTM Standards:

B 248 Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar<sup>3</sup>

B 601 Classification for Temper Designations for Copper and Copper Alloys—Wrought and Cast<sup>3</sup>

B 846 Terminology for Copper and Copper Alloys<sup>3</sup>

E 8 Test Methods for Tension Testing of Metallic Materials<sup>4</sup>

E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials<sup>4</sup>

E 112 Test Methods for Determining Average Grain Size<sup>4</sup>

E 527 Practice for Numbering Metals and Alloys (UNS)<sup>5</sup>

### 3. General Requirements

3.1 The following sections of Specification B 248 constitute a part of this specification:

3.1.1 Terminology

3.1.2 Materials and Manufacture

3.1.3 Dimensions, Weights, and Permissible Variations

3.1.4 Workmanship, Finish, and Appearance

3.1.5 Sampling

3.1.6 Number of Tests and Retests

3.1.7 Specimen Preparation

3.1.8 Test Methods

3.1.9 Significance of Numerical Limits

3.1.10 Inspection

3.1.11 Rejection and Rehearing

3.1.12 Certification

3.1.13 Mill Test Report

3.1.14 Packaging and Package Marking

3.2 In addition, when a section with a title identical to that referenced in 3.1 above appears in this specification, it contains additional requirements that supplement those appearing in Specification B 248.

### 4. Terminology

4.1 For terms relating to copper and copper alloys, refer to Terminology B 846.

### 5. Ordering Information

5.1 Include the following information in orders for products:

<sup>1</sup> This specification is under the jurisdiction of ASTM Committee B05 on Copper and Copper Alloys and is the direct responsibility of Subcommittee B05.01 on Plate, Sheet, and Strip.

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<sup>2</sup> The UNS system for copper and copper alloys (see Practice E 527) is a simple expansion of the former standard designation system accomplished by the addition of a prefix “C” and a suffix “00.” The suffix can be used to accommodate composition variations of the base alloy.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 02.01.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 03.01.

<sup>5</sup> *Annual Book of ASTM Standards*, Vol 01.01.

**\*A Summary of Changes section appears at the end of this standard.**

- 5.1.1 Quantity,
- 5.1.2 Copper Alloy UNS number (1.1),
- 5.1.3 Form of material: plate, sheet, strip, or rolled bar,
- 5.1.4 Temper (7.1),
- 5.1.5 Dimensions: thickness and width, and length if applicable.
- 5.1.6 How furnished: rolls, stock lengths with or without ends, specific lengths with or without ends,
- 5.1.7 Type of edge, if required: slit, sheared, sawed, square corners, rounded corners, rounded edges, or full-rounded edges (Specification B 248, Section 5.6),
- 5.1.8 Type of width and straightness tolerances, if required: slit-metal tolerances, square-sheared-metal tolerances, sawed-metal tolerances, straightened or edge-rolled-metal tolerances (Specification B 248, Section 5.3),
- 5.1.9 Special thickness tolerances, if required (Specification B 248, Table 3),
- 5.1.10 Tension test or hardness as applicable (Section 8),
- 5.1.11 Bend test, if required (Section 11),
- 5.1.12 Grain size or grain count if required (Section 9 or 10),
- 5.1.13 Certification if required (see Specification B 248, Section 14),
- 5.1.14 Mill Test Report, if required (see Specification B 248, Section 15),
- 5.1.15 Specification number and year of issue, and
- 5.1.16 Special tests or exceptions, if any.
- 5.2 When material is purchased for agencies of the U.S. government, this shall be specified in the contract or purchase order, and the material shall conform to the Supplementary requirements as defined in the current issue of Specification B 248.

## 6. Chemical Composition

- 6.1 The material shall conform to the chemical requirements specified in Table 1.
- 6.2 These specification limits do not preclude the presence of other elements. Limits for unnamed elements may be established, by agreement between manufacturer or supplier and purchaser. Copper may be given as remainder, and may be taken as the difference between the sum of all elements analyzed and 100 %. When all elements in Table 1 are analyzed, their sum shall be 99.5 % minimum.

## 7. Temper

- 7.1 Tempers available under this specification are defined in Practice B 601. The standard tempers of product are as

designated in Table 2, Table 3, and Table 4. Plate is generally available in the TB00 (A), TD04 (H), TF00 (AT), and TH04 (HT) tempers.

## 8. Mechanical Property Requirements

- 8.1 For product less than 0.050 in. (0.127 mm) in thickness:
  - 8.1.1 Tensile test results shall be the product acceptance criteria, when tested in accordance with Test Method E 8.
  - 8.1.2 The tensile strength requirements are given in Table 2, Table 3, and Table 4.
- 8.2 For product 0.050 in. (1.270 mm) and greater in thickness:
  - 8.2.1 Rockwell hardness is the product acceptance criteria, when tested in accordance with Test Method E 18.
  - 8.2.2 The referee product rejection criteria shall be tensile test results, when tested in accordance with Test Method E 8.
  - 8.2.3 Rockwell hardness and tensile strength requirements are given in Table 2, Table 3, and Table 4.
- 8.3 Product, as specified in 7.1, shall conform to the requirements specified in Table 2, in the solution heat-treated, or solution heat-treated and cold-worked conditions, and in Table 3, after precipitation heat-treatment or Table 4 in the mill-hardened condition. Precipitation heat-treatment parameters for Table 2 and Table 3 are shown in Section 12.

## 9. Grain Size

- 9.1 Material over 0.010 in. (0.254 mm) in thickness shall have an average grain size in accordance with Test Method E 112, not exceeding the limits specified in Table 5. The determinations are made on the separate samples and in a plane perpendicular to the surface and perpendicular to the direction of rolling.

## 10. Grain Count

- 10.1 The grain count of a sample of material, in any temper, over 0.004 to 0.010 in. (0.102 to 0.254 mm), inclusive, in thickness shall not be less than the limits specified in Table 6.
- 10.2 Grain count is the number of grains per stock thickness, averaged for five locations one stock thickness apart. Grain count shall be determined in a plane perpendicular to the surface and perpendicular to the direction of rolling.

## 11. Bend-Test Requirements

- 11.1 The optional bend test is a method for evaluating the ductility of precipitation heat-treated copper-beryllium strip in thin gages.
- 11.2 When specified in the order (see 5.1.6), material in any temper 0.004 to 0.020 in. (0.102 to 0.508 mm), inclusive, in thickness shall conform to the requirements specified in Table 7, when tested in accordance with 14.2.
- 11.3 Five specimens,  $\frac{3}{8} \pm \frac{1}{16}$  in. (9.52  $\pm$  1.59 mm) in width, of any convenient length, with the rolling direction parallel to the  $\frac{3}{8}$ -in. dimension, shall be precipitation heat-treated in accordance with 12.2. To pass the bend test, at least four specimens out of five, and at least 80 % of the total specimens tested from a lot shall withstand the 90° bend without visible crack or fracture, when tested in accordance with 15.3.

**TABLE 1 Chemical Requirements**

| Element                     | Composition, %              |                             |
|-----------------------------|-----------------------------|-----------------------------|
|                             | Copper Alloy UNS No. C17000 | Copper Alloy UNS No. C17200 |
| Beryllium                   | 1.60–1.79                   | 1.80–2.00                   |
| Additive elements:          |                             |                             |
| Nickel + cobalt, min        | 0.20                        | 0.20                        |
| Nickel + cobalt + iron, max | 0.6                         | 0.6                         |
| Aluminum, max               | 0.20                        | 0.20                        |
| Silicon, max                | 0.20                        | 0.20                        |
| Copper                      | remainder                   | remainder                   |

**TABLE 2 Mechanical Property Requirements for Material in the Solution-Heat-Treated or Solution-Heat-Treated and Cold-Worked Condition**

| Temper Designation <sup>A</sup> |        | Material Thickness, in. |       | Tensile Strength,<br>ksi <sup>B</sup> (MPa) <sup>C</sup> | Elongation <sup>D</sup> in 2 in. or<br>50 mm, min, % | Rockwell Hardness <sup>E</sup> |           |           |
|---------------------------------|--------|-------------------------|-------|----------------------------------------------------------|------------------------------------------------------|--------------------------------|-----------|-----------|
| Standard                        | Former | Over                    | Incl  |                                                          |                                                      | B Scale                        | 30T Scale | 15T Scale |
| TB00                            | A      | ...                     | ...   | 60–78<br>(410–540)                                       | 35                                                   | 45–78                          | 46–67     | 75–85     |
| TD01                            | ¼ H    | ...                     | 0.188 | 75–88<br>(520–610)                                       | 15                                                   | 68–90                          | 62–75     | 83–89     |
| TD02                            | ½ H    | ...                     | 0.188 | 85–100<br>(590–690)                                      | 9                                                    | 88–96                          | 74–79     | 88–91     |
| TD04                            | H      | ...                     | 0.188 | 100–130<br>(690–900)                                     | 2                                                    | 96–104                         | 79–83     | 91–94     |
| TD04                            | H      | 0.188                   | 0.375 | 90–130<br>(660–900)                                      | ...                                                  | 91–103                         | 77        | 90        |
| TD04                            | H      | 0.375                   | 1.000 | 90–120<br>(620–830)                                      | ...                                                  | 90–102                         | ...       | ...       |
| TD04                            | H      | over 1.000              |       | 85–115<br>(590–800)                                      | 8                                                    | 88–102                         | ...       | ...       |

<sup>A</sup> Standard designations defined in Practice B 601.

<sup>B</sup> ksi = 1000 psi.

<sup>C</sup> See Appendix X1.

<sup>D</sup> Elongation requirement applies to material 0.004 in. (0.102 mm) and thicker.

<sup>E</sup> The thickness of material that may be tested by use of the Rockwell hardness scales is as follows:

B Scale.....0.040 in. (1.016 mm) and over

30T Scale.....0.020 to 0.040 in. (0.508 to 1.016 mm), excl.

15T Scale.....0.015 to 0.020 in. (0.381 to 0.508 mm), excl.

Hardness values shown apply only to direct determinations, not converted values.

## 12. Precipitation Heat-Treatment

12.1 Solution-heat-treated or solution-heat-treated and cold-worked material is normally precipitation hardened by the purchaser after forming or machining. For the purpose of determining conformance to specified mechanical properties of Table 3, a sample of the as-supplied material shall be heat treated as shown in Table 8. Other heat treating temperatures and times may be preferred for end products of this material.

12.2 The solution-heat-treated and cold-worked test specimens shall be heat treated at a uniform temperature of 600 to 675°F (316 to 357°C) for the time shown in Table 8.

12.3 Special combinations of properties such as increased ductility, electrical conductivity, dimensional accuracy, endurance life, and resistance to elastic drift and hysteresis in springs may be obtained by special precipitation-hardening heat treatments. The mechanical requirements of Table 3 do not apply to such special heat treatments.

12.4 Mill-hardened products have been precipitation heat-treated by the manufacturer. Further thermal treatment is not normally required.

## 13. Sampling

13.1 Sampling shall be in accordance with Specification B 248, Section 7, except that the heat size is defined as 12 000 lbs (5455 kg) or fraction thereof.

## 14. Specimen Preparation

14.1 The tension specimen direction shall have the longitudinal test-axis parallel to the rolling direction, unless mutually agreed upon between the supplier and purchaser at the time the order is placed.

14.2 When required, five bend-test specimens per test set shall be cut  $\frac{3}{8} \pm \frac{1}{16}$  in. (9.52  $\pm$  1.59 mm) in width and any convenient length. Specimens shall be precipitation heat-treated after cutting and prior to testing. Precipitation heat-treatment parameters for these bend tests shall be in accordance with 12.2.

## 15. Test Methods

15.1 The method for determining chemical analysis for compliance and preparation of certifications and test reports shall be at the discretion of the reporting laboratory.

15.2 In case of dispute, the test methods found in Annex A1 shall be used for determining chemical requirements for the elements and ranges shown in Table 1.

15.2.1 When analysis for unnamed or residual elements is required in the purchase order, the method of analysis shall be mutually agreed upon between manufacturer or supplier and purchaser.

15.3 Bend-test specimens, shall be tested by clamping them firmly between a flat jaw and the test radius, as shown in Fig. 1. The test specimen shall be bent approximately 90° around the test radius, using a tangential wiping motion with adequate radial pressure to ensure continuous contact between the specimen and the test radius. Test specimens shall be bent to the full 90° bend position. The test radius shall be within  $\pm$  6 % of the nominal radius up to 0.010 in. (0.254 mm), exclusive, and within  $\pm$  4 % for radii 0.010 in. and over.

## 16. Keywords

16.1 C17000; C17200; copper-beryllium; flat products; copper plate; copper rolled bar; copper strip

TABLE 3 Mechanical Property Requirements After Precipitation Heat-Treatment<sup>A</sup>

| Temper Designation          |        | Material Thickness, in. |       | Tensile Strength,<br>ksi <sup>B</sup> (MPa) <sup>C</sup> | Yield Strength, ksi<br>(MPa),<br>min, 0.2 % Offset | Elongation in<br>2 in.<br>(50 mm), min,<br>% <sup>D</sup> | Rockwell Hardness <sup>E</sup> , min |           |           |
|-----------------------------|--------|-------------------------|-------|----------------------------------------------------------|----------------------------------------------------|-----------------------------------------------------------|--------------------------------------|-----------|-----------|
| Standard                    | Former | Over                    | Incl  |                                                          |                                                    |                                                           | C Scale                              | 30N Scale | 15N Scale |
| Copper Alloy UNS No. C17000 |        |                         |       |                                                          |                                                    |                                                           |                                      |           |           |
| TF00                        | AT     | ...                     | 0.188 | 150–180 <sup>F</sup><br>(1030–1240)                      | 130<br>(890)                                       | 3                                                         | 33                                   | 53        | 76.5      |
| TF00                        | AT     | 0.188                   | ...   | 165–195 <sup>F</sup> (1140–1340)                         | 130                                                | 3                                                         | 36                                   | 56        | 78        |
| TH01                        | ¼ HT   | ...                     | ...   | 160–190 <sup>F</sup><br>(1100–1310)                      | 135<br>(930)                                       | 2.5                                                       | 35                                   | 55        | 77        |
| TH02                        | ½ HT   | ...                     | ...   | 170–200 <sup>F</sup><br>(1170–1380)                      | 145<br>(1000)                                      | 1                                                         | 37                                   | 57        | 78.5      |
| TH04                        | HT     | ...                     | ...   | 180–210 <sup>F</sup><br>(1240–1450)                      | 155<br>(1070)                                      | 1                                                         | 38                                   | 58        | 79.5      |
| Copper Alloy UNS No. C17200 |        |                         |       |                                                          |                                                    |                                                           |                                      |           |           |
| TF00                        | AT     | ...                     | ...   | 165–195 <sup>F</sup><br>(1140–1340)                      | 140<br>(960)                                       | 3                                                         | 36                                   | 56        | 78        |
| TH01                        | ¼ HT   | ...                     | 0.188 | 175–205 <sup>F</sup><br>(1210–1410)                      | 150<br>(1030)                                      | 2.5                                                       | 36                                   | 56        | 79        |
| TH02                        | ½ HT   | ...                     | 0.188 | 185–215 <sup>F</sup><br>(1280–1480)                      | 160<br>(1100)                                      | 1                                                         | 38                                   | 58        | 79.5      |
| TH04                        | HT     | ...                     | 0.188 | 190–220 <sup>F</sup><br>(1310–1520)                      | 165<br>(1140)                                      | 1                                                         | 38                                   | 58        | 80        |
| TH04                        | HT     | 0.188                   | 0.375 | 180–215 <sup>F</sup><br>(1240–1480)                      | 160<br>(1100)                                      | 1                                                         | 38                                   | 58        | 80        |
| TH04                        | HT     | 0.375                   | 1.000 | 180–210 <sup>F</sup><br>(1240–1450)                      | 155<br>(1070)                                      | 1                                                         | 38                                   | ...       | ...       |
| TH04                        | HT     | 1.000                   | 2.000 | 175–205 <sup>F</sup><br>(1210–1410)                      | 150<br>(1030)                                      | 2                                                         | 37                                   | ...       | ...       |
| TH04                        | HT     | over 2.000              |       | 165–200 <sup>F</sup><br>(1140–1380)                      | 130<br>(890)                                       | 2                                                         | 36                                   | ...       | ...       |

<sup>A</sup> These values apply to mill products (Section 14). See 12.3 for exceptions in end products.<sup>B</sup> ksi = 1000 psi.<sup>C</sup> See Appendix X1.<sup>D</sup> Elongation requirement applies to material 0.004 in. (0.102 mm) and thicker.<sup>E</sup> The thickness of material that may be tested by use of the Rockwell Hardness scales is as follows:

C Scale.....0.040 in. (1.016 mm) and over

30N Scale.....0.020 to 0.040 in. (0.508 to 1.016 mm), excl.

15N Scale.....0.015 to 0.02 in. (0.381 to 0.508 mm), excl.

Hardness values shown apply only to direct determinations, not converted values.

<sup>F</sup> The upper limits in the tensile strength column are for design guidance only.

**TABLE 4 Strip Mechanical Property Requirements—Mill-Hardened Condition<sup>A</sup>**

| Temper Designation          |                     | Tensile Strength,<br>ksi <sup>B</sup> (MPa) <sup>C</sup> | Yield Strength,<br>ksi (MPa),<br>0.2 % Offset | Elongation in 2<br>in. (50 mm),<br>min, % <sup>D</sup> | Rockwell Hardness <sup>E</sup> , min |           |           |
|-----------------------------|---------------------|----------------------------------------------------------|-----------------------------------------------|--------------------------------------------------------|--------------------------------------|-----------|-----------|
| Standard                    | Former <sup>B</sup> |                                                          |                                               |                                                        | C Scale                              | 30N Scale | 15N Scale |
| Copper Alloy UNS No. C17000 |                     |                                                          |                                               |                                                        |                                      |           |           |
| TM00                        | AM                  | 100–110 <sup>F</sup><br>(690–760)                        | 70–95<br>(480–660)                            | 18                                                     | 18                                   | 37        | 67.5      |
| TM01                        | ¼ HM                | 110–120 <sup>F</sup><br>(760–830)                        | 80–110<br>(550–760)                           | 15                                                     | 20                                   | 42        | 70        |
| TM02                        | ½ HM                | 120–135 <sup>F</sup><br>(830–930)                        | 95–125<br>(660–860)                           | 12                                                     | 24                                   | 45        | 72        |
| TM04                        | HM                  | 135–150 <sup>F</sup><br>(930–1040)                       | 110–135<br>(760–930)                          | 9                                                      | 28                                   | 48        | 75        |
| TM05                        | SHM                 | 150–160 <sup>F</sup><br>(1030–1100)                      | 125–140<br>(860–970)                          | 9                                                      | 31                                   | 52        | 75.5      |
| TM06                        | XHM                 | 155–175 <sup>F</sup><br>(1070– 1210)                     | 135–165<br>(930–1140)                         | 3                                                      | 32                                   | 52        | 76        |
| Copper Alloy UNS No. C17200 |                     |                                                          |                                               |                                                        |                                      |           |           |
| TM00                        | AM                  | 100–110 <sup>F</sup><br>(690–760)                        | 70–95<br>(480–660)                            | 16                                                     | R <sub>B</sub> 95                    | 37        | 67.5      |
| TM01                        | ¼ HM                | 110–120 <sup>F</sup><br>(760–830)                        | 80–110<br>(550–760)                           | 15                                                     | 20                                   | 42        | 70        |
| TM02                        | ½ HM                | 120–135 <sup>F</sup><br>(830–930)                        | 95–125<br>(660–860)                           | 12                                                     | 23                                   | 44        | 72        |
| TM04                        | HM                  | 135–150 <sup>F</sup><br>(930–1030)                       | 110–135<br>(760–930)                          | 9                                                      | 28                                   | 48        | 75        |
| TM05                        | SHM                 | 150–160 <sup>F</sup><br>(1030–1100)                      | 125–140<br>(860–970)                          | 9                                                      | 31                                   | 52        | 75.5      |
| TM06                        | XHM                 | 155–175 <sup>F</sup><br>(1070–1210)                      | 135–170<br>(930–1170)                         | 4                                                      | 32                                   | 52        | 76        |
| TM08                        | XHMS                | 175–190 <sup>F</sup><br>(1210–1310)                      | 150–180<br>(1030–1240)                        | 3                                                      | 33                                   | 53        | 76.5      |

<sup>A</sup> These values apply to mill products (Section 14). See 12.3 for exceptions in end products.

<sup>B</sup> ksi = 1000 psi.

<sup>C</sup> See Appendix X1.

<sup>D</sup> Elongation requirement applies to material 0.004 in. (0.102 mm) and thicker.

<sup>E</sup> The thickness of material that may be tested by use of the Rockwell Hardness scales is as follows:

C Scale.....0.040 in. (1.016 mm) and over

30N Scale.....0.020 to 0.040 in. (0.508 to 1.016 mm), excl.

15N Scale.....0.015 to 0.020 in. (0.381 to 0.508 mm), excl.

Hardness values shown apply only to direct determinations, not converted values.

<sup>F</sup> The upper limits in the tensile strength column are for design guidance only.

**TABLE 5 Grain-Size Requirements for TB00 (Solution-Heat-Treated) Material**

| Thickness, in. (mm)                        | Grain Size Specified | Maximum Average Grain Size, mm |
|--------------------------------------------|----------------------|--------------------------------|
| Over 0.010 to 0.030 (0.254 to 0.762), incl | OS035                | 0.035                          |
| Over 0.030 to 0.090 (0.762 to 2.24), incl  | OS045                | 0.045                          |
| Over 0.090 to 0.188 (2.24 to 4.78), incl   | OS060                | 0.060                          |

**TABLE 6 Grain-Count Requirements**

| Thickness, in. (mm)                        | Minimum Number of Grains |
|--------------------------------------------|--------------------------|
| Over 0.004 to 0.006 (0.102 to 0.152), incl | 6                        |
| Over 0.006 to 0.008 (0.152 to 0.203), incl | 7                        |
| Over 0.008 to 0.010 (0.203 to 0.254), incl | 8                        |

**TABLE 7 Bend-Test Requirements After Precipitation Heat Treatment**

| Temper Designation |        | Test Radius <sup>A</sup> |
|--------------------|--------|--------------------------|
| Standard           | Former |                          |
| TF00               | AT     | 5 <i>t</i>               |
| TH01               | ¼ AT   | 6 <i>t</i>               |
| TH02               | ½ HT   | 9 <i>t</i>               |
| TH04               | HT     | 15 <i>t</i>              |

<sup>A</sup> The *t* refers to the measured average stock thickness to be tested.

**TABLE 8 Precipitation-Heat-Treatment Time for Acceptance Tests**

| Temper Designation (Before Precipitation Heat Treatment) |        | Time at 600 to 675°F<br>(316 to 357°C), h |
|----------------------------------------------------------|--------|-------------------------------------------|
| Standard                                                 | Former |                                           |
| TB00                                                     | A      | 3                                         |
| TD01                                                     | ¼ H    | 2                                         |
| TD02                                                     | ½ H    | 2                                         |
| TD04                                                     | H      | 2                                         |

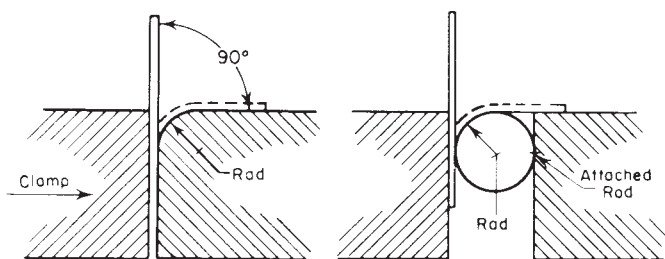


FIG. 1 Methods for Clamping Specimen to Radius for Bend Test

## ANNEX

### (Mandatory Information)

#### A1. TEST METHODS FOR DETERMINATION OF COMPLIANCE WITH COPPER-BERYLLIUM ALLOYS—CHEMICAL COMPOSITION REQUIREMENTS

##### A1.1 Scope

A1.1.1 These test methods establish the procedure(s) for the determination of chemical composition of copper-beryllium alloys.

A1.1.2 The analytical procedures appear in the following order:

| Procedure                                                                                                            | Sections         |
|----------------------------------------------------------------------------------------------------------------------|------------------|
| Test Method A—Copper by the Electrolytic Method                                                                      | A1.8 to A1.15    |
| Test Method B—Aluminum, Beryllium, Cobalt, Iron, and Nickel by the Flame Atomic Absorption Spectrophotometric Method | A1.16 to A1.24   |
| Test Method C—Silicon by the Ammonium Molybdate Spectrophotometric Method                                            | A1.28.2 to A1.35 |

##### A1.2 Referenced Documents

###### A1.2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>6</sup>
- E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials<sup>7</sup>
- E 60 Practice for Analysis of Metals, Ores, and Related Materials by Molecular Absorption Spectrometry<sup>7</sup>
- E 255 Practice for Sampling Copper and Copper Alloys for the Determination of Chemical Composition<sup>7</sup>
- E 663 Practice for Flame Atomic Absorption Analysis<sup>8</sup>
- E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry<sup>8</sup>

##### A1.3 Significance and Use

A1.3.1 These test methods are primarily intended to test for compliance with composition 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.

##### A1.4 Apparatus, Reagents, and Photometric Practice

A1.4.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure. The apparatus, standard solutions, and certain other reagents are referred to by number and shall conform to the requirements prescribed in Practice E 50.

A1.4.2 Flame atomic-absorption spectrophotometric practice prescribed in these test methods shall conform to the requirements prescribed in Practice E 663 and Guide E 1024.

A1.4.3 Spectrophotometric practice prescribed in these test methods shall conform to requirements prescribed in Practice E 60.

##### A1.5 Hazards

A1.5.1 For precautions to be observed in these test methods, refer to Practice E 50.

A1.5.2 Both beryllium metal and its compounds may be toxic. Exercise care to prevent contact of beryllium-containing solutions with the skin. Especially avoid the inhalation of any beryllium-containing substance, either as a volatile compound or as a finely divided powder. The proper precautions are to be observed in the disposition of beryllium-containing residues, especially ignited oxide.

##### A1.6 Sampling

A1.6.1 Sampling shall conform to the requirements of Practice E 255.

##### A1.7 Rounding Off Calculated Values

A1.7.1 Calculated values shall be rounded off to the proper number of places in accordance with the method given in 3.4 and 3.5 of Practice E 29.

#### TEST METHOD A—COPPER BY ELECTROLYTIC DEPOSITION AND ATOMIC-ABSORPTION SPECTROPHOTOMETRY

##### A1.8 Scope

A1.8.1 This test method establishes a procedure for the determination of copper in copper-beryllium alloys with silver reported as copper.

<sup>6</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>7</sup> Annual Book of ASTM Standards, Vol 03.05.

<sup>8</sup> Annual Book of ASTM Standards, Vol 03.06.

## A1.9 Summary of Test Methods

A1.9.1 The sample is dissolved in an acid mixture. A small amount of fluorohydric acid (HF) is added to minimize possible interferences. Copper is electrolytically deposited on a tared platinum cathode. Copper remaining in the electrolyte is determined by atomic absorption spectrophotometry.

## A1.10 Interferences

A1.10.1 Elements normally present do not interfere.

## A1.11 Apparatus

A1.11.1 *Electrodes for Electrolysis*—Apparatus No. 9, in Practice E 50.

A1.11.2 *Atomic Absorption Spectrophotometer*—Determine the instrument to be suitable for use as directed in Guide E 1024. Instrument response must permit estimation of copper concentration to within 1 mg/

A1.11.3 *Operating Parameters*—Wavelength, fuel/oxidant, and flame conditions are as follows:

| Wavelength, nm | Fuel/Oxidant  | Flame Condition |
|----------------|---------------|-----------------|
| Copper 327.5   | Acetylene/air | Oxidizing       |

## A1.12 Reagents

A1.12.1 *Sulfuric-Nitric Acid Mixture*—While stirring, slowly add 500 mL of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) to 1 L of water. Cool and transfer to a 2-L volumetric flask. Add 300 mL of nitric acid (HNO<sub>3</sub>). Cool, dilute to volume, and mix.

A1.12.2 *Copper Standard Solution* (1 mL = 1.0 mg Cu)—Transfer 1.000 g of copper metal (purity, 99.9 % min) into a 250-mL beaker. Add 20 mL of the acid mixture. Cover the beaker and allow to stand until dissolution is nearly complete. Heat at 80 to 90°C until dissolution is complete and brown fumes have been expelled. Cool, transfer into a 1-L volumetric flask, dilute to volume, and mix.

A1.12.3 *Calibration Solutions*—Pipet 5, 10, 15, 20, and 25-mL portions of the copper standard solution into individual 1-L volumetric flasks. Add 50 mL of the acid mixture to each flask, dilute to volume, and mix. These solutions are equivalent to 0.005, 0.010, 0.015, 0.020, and 0.025 g of copper respectively.

A1.12.4 *Zero-Calibration Solution*—Transfer 50 mL of the acid mixture into a 1-L volumetric flask, dilute to volume, and mix.

## A1.13 Procedure

A1.13.1 Transfer a 2.500-g portion into each of two electrolysis beakers, normally 300-mL. Add 50 mL of the mixed acid, cover the beaker, and allow to stand until the reaction subsides. Heat at 80 to 90°C until dissolution is complete and brown fumes have been expelled. Cool and wash down cover glass and inside of beaker. Add 1.0 mL of HF (1 + 9) from a plastic pipet and dilute to about half volume.

A1.13.2 Insert the electrodes and dilute to submerge the cathode. Cover the beaker with a pair of split cover glasses and electrolyze at a current density of about 0.6 A/dm<sup>2</sup> for about 16 h.

A1.13.3 Wash the cover glasses, the electrode stems, and inside the beaker with water, then continue the electrolysis for a minimum of 15 min. Should copper plate-out on the newly

exposed cathode surface, dilute a second time and continue electrolysis for an additional 15 min. Copper deposition shall be considered completed, when no copper is deposited on a newly exposed surface.

A1.13.4 Quickly withdraw the cathode from the electrolyte while maintaining current flow (should the electrolysis system permit), and direct a gentle stream of water from a wash bottle over its surface. Rinse the cathode in a water bath and then dip in two successive baths of ethanol or acetone. Dry at 110°C for 3 to 5 min, cool at balance room temperature, and weigh.

A1.13.5 Transfer the spent electrolyte into individual 1-L volumetric flask, dilute to volume, and mix.

A1.13.6 Set the atomic-absorption instrument parameters according to Practice E 663 and the manufacturer's recommendations. Ignite the burner and aspirate water until the instrument reaches thermal equilibrium.

A1.13.7 Adjust the wavelength, lamp position, fuel, oxidizer, burner, and nebulizer to obtain maximum absorbance, while aspirating the highest calibration solution.

A1.13.8 Aspirate water until a steady signal is obtained and adjust the instrument read-out system to obtain zero absorbance.

A1.13.9 Aspirate the calibration solutions in order of increasing absorbance, starting with the zero calibration solution. When a stable response is obtained, record the readings. Aspirate the test solutions and record their absorbance. Aspirate water between samples to flush the nebulizer and burner systems. Repeat all measurements a minimum of two times.

## A1.14 Calculation

A1.14.1 When necessary, convert the average readings for each solution to absorbance. Obtain the net absorbance for each calibration solution by subtracting the average absorbance for the zero-calibration solution from the average absorbance of each of the other calibration solutions.

A1.14.2 Obtain the net absorbance of the zero-calibration solution from the average absorbance of the test solution.

A1.14.3 Prepare a calibration curve by plotting net absorbance for the calibration solutions versus grams of copper.

A1.14.4 Convert the net absorbance of the test solution to grams of copper by means of the calibration curve.

A1.14.4.1 Most atomic-absorption spectrophotometers can be calibrated to yield direct concentration readings. This method may be used, provided additional calibration solutions are analyzed as samples to test for precision and linearity. Should the instrument be equipped for multi-point calibration, make sure that several additional solutions still are analyzed to ensure that error has not been introduced by the curve-fitting routine.

A1.14.5 Calculate the concentration percent copper as follows:

$$\text{Copper, \%} = (A - B + C) \times 100/D \quad (\text{A1.1})$$

where:

A = weight of cathode plus deposited copper, g,

B = weight of cathode, g,

C = weight of copper in spent electrolyte, g, and

D = sample used, g.

## A1.15 Precision and Bias

A1.15.1 *Precision*—The precision of this test method is dependent upon the care and precision exercised during instrument calibration and sample preparation, as well as, the purity of the reagents.

A1.15.2 *Bias*—The accuracy of this test method can be judged by analyzing material of known composition.

## TEST METHOD B—ALUMINUM, BERYLLIUM, COBALT, IRON, LEAD, AND NICKEL BY THE FLAME ATOMIC-ABSORPTION SPECTROPHOTOMETRIC METHOD

### A1.16 Scope

A1.16.1 This test method establishes a flame atomic-absorption spectrophotometric procedure for the determination of aluminum, beryllium, cobalt, iron, lead, and nickel in copper-beryllium alloys.

### A1.17 Summary of Test Methods

A1.17.1 The sample is dissolved in dilute nitric acid and aspirated into the flame of an atomic absorption spectrophotometer. The absorption of the resonance line energy specific to each element is measured and compared with the absorption measured for calibration solutions prepared in the same matrix.

### A1.18 Interferences

A1.18.1 Elements normally present in copper-beryllium alloys do not interfere.

### A1.19 Apparatus

A1.19.1 *Atomic-Absorption Spectrophotometer*—Determine the instrument to be suitable for use as directed in Guide E 1024. Instrument response for each analyte element must be adequate to permit an estimation of analyte concentration to within 0.01 % for aluminum, iron, and lead and 0.02 % for beryllium, cobalt, and nickel on a sample basis.

A1.19.2 *Operating Parameters*—The flame conditions and wavelengths for the analyte elements are as follows:

| Element   | Wavelength, nm | Fuel/Oxidant and Flame Condition     |
|-----------|----------------|--------------------------------------|
| Aluminum  | 309.3          | Acetylene/nitrous oxide and reducing |
| Beryllium | 234.9          | Acetylene/nitrous oxide and reducing |
| Cobalt    | 240.7          | Acetylene/air and oxidizing          |
| Iron      | 248.3          | Acetylene/air and oxidizing          |
| Lead      | 283.3          | Acetylene/air and oxidizing          |
| Nickel    | 341.5          | Acetylene/air and oxidizing          |

### A1.20 Reagents

A1.20.1 *Copper Stock Solution*—Transfer 50.0 g of copper (purity, 99.99 % min) into a 2-L beaker. Cover with 200 mL of water. Cover the beaker and cautiously add 200 mL of nitric acid (HNO<sub>3</sub>) in small increments. Allow to stand until dissolution is nearly complete. Boil to complete dissolution and expel brown fumes. Cool, transfer the solution into a 1-L volumetric flask, dilute to volume, and mix.

A1.20.2 *Aluminum Standard Solution* (1 mL = 0.15 mg Al)—Weigh 0.1500 g of aluminum wire (purity, 99.9 % min) into a 400-mL beaker. Add 20 mL of water and cover with a watch glass. Cautiously add 40 mL of HNO<sub>3</sub> (1 + 1) in small increments. Add a small crystal of mercurous nitrate (HgNO<sub>3</sub>)

and two drops of hydrochloric acid (HCl) after the first increment to catalyze the reaction. Boil to expel the brown fumes. Rinse the watch glass and inside of the beaker with water. Transfer the solution into a 1-L volumetric flask.

A1.20.3 *Beryllium Standard Solution* (1 mL = 1.25 mg Be):

A1.20.3.1 Transfer 1.250-g equivalent of beryllium,<sup>9</sup> containing less than 1000 ppm each of cobalt, iron, lead, and nickel, into a 600-mL beaker, add 20 mL of water and cover with a watch glass. Cautiously add 35 mL of HNO<sub>3</sub> in small increments. Add two drops of HCl after the first increment to catalyze the reaction. After the reaction subsides, rinse the watch glass and inside of the beaker with water and dilute to approximately 200 mL. Boil to expel the brown fumes. Filter hot water through a fine porosity ashless paper into a 1-L plastic volumetric flask. Rinse the beaker several times with water and filter, collecting the rinse solutions into the volumetric flask. Rinse the filter paper ten times with small portions of hot water, collecting the rinse solutions in the volumetric flask.

A1.20.3.2 Transfer the filter paper into a platinum crucible and reduce to a white ash over a Meker type burner, heating gently initially to avoid losses. Allow the crucible to cool and add 5 drops of HF and 10 drops of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>). Place the crucible on a hot plate and slowly evaporate just to dryness. *Do not bake.* Allow the crucible to cool. Add 5 mL of HNO<sub>3</sub>, 1 drop of fluorohydric (HF), and heat to boiling. Allow the crucible to cool, add 10 mL of water, and filter the solution through a medium porosity filter paper collecting the solution into the original 1-L volumetric flask. Rinse the filter paper a minimum of four times, collecting the rinse solutions into the same 1-L volumetric flask.

A1.20.3.3 Dilute the combined solutions to volume and mix.

A1.20.4 *Cobalt Standard Solution* (1 mL = 1.5 mg Co)—Dissolve 1.500 g of cobalt (purity, 99.9 % min) in 80 mL of HNO<sub>3</sub> (1 + 1). Boil to expel the brown fumes. Cool, transfer into a 1-L volumetric flask, dilute to volume, and mix.

A1.20.5 *Iron Standard Solution* (1 mL = 0.3 mg Fe)—Dissolve 0.3000 g of iron (purity, 99.9 % min) in 80 mL of HNO<sub>3</sub> (1 + 1). Boil to expel the brown fumes. Cool, transfer into a 1-L volumetric flask, dilute to volume, and mix.

A1.20.6 *Lead Standard Solution* (1 mL = 0.3 mg Pb)—Dissolve 0.3000 g of lead (purity, 99.9 % min) in 80 mL of HNO<sub>3</sub> (1 + 1). Boil to expel the brown fumes, cool, dilute to volume, and mix.

A1.20.7 *Nickel Standard Solution* (1 mL = 1.25 mg Ni)—Dissolve 1.250 g of nickel (purity, 99.9 % min) in 80 mL of HNO<sub>3</sub> (1 + 1). Boil to expel the brown fumes, cool, dilute to volume, and mix.

### A1.21 Calibration

A1.21.1 *Calibration Solutions*—Label eight plastic 500-mL volumetric flasks A, B, C, D, E, F, G, and H respectively. Transfer by pipet 50 mL of the copper stock solution into each

<sup>9</sup> Beryllium reference material NBL-85 (99.0 Be), available from U.S. Department of Energy, New Brunswick Laboratory, 9800 South Cass Avenue, Argonne, IL, has been found suitable. A1.263 g-portion of this reference material contains 1.250 g of beryllium.

flask. To the 8 volumetric flasks add the volumes of the standard solutions and HNO<sub>3</sub> (1 + 4) as in Table A1.1.

A1.21.1.1 The concentration percent of the analyte elements on a sample basis for each calibration solution are as in Table A1.2.

A1.21.2 *Zero-Calibration Solution*—Transfer by pipet 50 mL of the copper stock solution into a 500-mL volumetric flask, add 50 mL of HNO<sub>3</sub>, dilute to volume, and mix.

## A1.22 Procedure

A1.22.1 *Test Solutions*—Transfer two portions of 2500 g each into individual 400-mL beakers and cover with 50 mL of water. Cover the beaker, add 20 mL of HNO<sub>3</sub>, and allow to stand until dissolution is nearly complete. Heat at 80 to 90°C until dissolution is complete. Cool, wash down the cover glass and inside of the beaker. Transfer each of the solutions into individual 500-mL volumetric flasks, dilute to volume, and mix.

A1.22.2 *Reagent Blank*—Carry a reagent blank through the entire procedure starting with A1.22.1.

A1.22.3 *Final Dilution*—Immediately prior to analysis, transfer by pipet aliquots of the calibration solutions, test solutions, and reagent blank into respective volumetric flasks and dilute to volume as in Table A1.3.

### A1.22.4 Atomic-Absorption Measurements:

A1.22.4.1 Set the required instrument parameters according to Practice E 663 and the manufacturer's recommendations. Light the burner and aspirate water until the instrument reaches thermal equilibrium.

A1.22.4.2 Adjust the wavelength, lamp position, fuel, oxidant, burner and nebulizer to obtain maximum absorbance, while aspirating the appropriate dilution of the highest calibration solution.

A1.22.4.3 Aspirate water until a steady signal is obtained and adjust the instrument readout system to obtain zero absorbance.

A1.22.4.4 Aspirate the appropriate dilutions of the calibration solutions in order of increasing absorbance starting with the zero-calibration solution. When a stable response is obtained, record the readings. Aspirate the appropriate dilution of the reagent blank and the test solutions and record their absorbance readings. Aspirate water between samples to flush the nebulizer and burner system. Repeat all measurements at least two times.

**TABLE A1.1 Calibration Solutions**

| Flask <sup>A</sup> | Volume of Standard Solution, mL |           |        |      |      |        | HNO <sub>3</sub><br>(1 + 4) |
|--------------------|---------------------------------|-----------|--------|------|------|--------|-----------------------------|
|                    | Aluminum                        | Beryllium | Cobalt | Iron | Lead | Nickel |                             |
| A                  | 5                               | 5         | 2.5    | 2.5  | 2.5  | 2.5    | 47                          |
| B                  | 10                              | 10        | 5      | 5    | 5    | 5      | 44                          |
| C                  | 15                              | 15        | 7.5    | 7.5  | 7.5  | 7.5    | 40                          |
| D                  | 20                              | 20        | 10     | 10   | 10   | 10     | 35                          |
| E                  | 25                              | 30        | 20     | 20   | 20   | 20     | 26                          |
| F                  | 30                              | 40        | 30     | 30   | 30   | 30     | 18                          |
| G                  | 40                              | 50        | 40     | 40   | 40   | 40     | 9                           |
| H                  | 50                              | 60        | 50     | 50   | 50   | 50     | 0                           |

<sup>A</sup> Dilute each flask to volume and mix.

**TABLE A1.2 Concentration of Analyte Elements**

| Solution | Sample Basis Analyte Concentration, % |           |        |      |       |       |
|----------|---------------------------------------|-----------|--------|------|-------|-------|
|          | Aluminum                              | Beryllium | Cobalt | Iron | Lead  | Ni    |
| A        | 0.03                                  | 0.25      | 0.15   | 0.03 | 0.035 | 0.125 |
| B        | 0.06                                  | 0.50      | 0.30   | 0.06 | 0.07  | 0.25  |
| C        | 0.09                                  | 0.75      | 0.45   | 0.09 | 0.105 | 0.375 |
| D        | 0.12                                  | 1.00      | 0.60   | 0.12 | 0.14  | 0.50  |
| E        | 0.15                                  | 1.50      | 1.20   | 0.24 | 0.28  | 1.00  |
| F        | 0.18                                  | 2.00      | 1.80   | 0.36 | 0.42  | 1.50  |
| G        | 0.24                                  | 2.50      | 2.40   | 0.48 | 0.56  | 2.00  |
| H        | 0.30                                  | 3.00      | 3.00   | 0.60 | 0.70  | 2.50  |

**TABLE A1.3 Calibration Solutions at Final Dilution**

| Solutions, %             | Aliquot (mL) | Final Volume      | Applicable<br>Calibrated<br>Solution |
|--------------------------|--------------|-------------------|--------------------------------------|
| Aluminum (0.01 to 0.30)  | 50           | 50                | A—H                                  |
| Beryllium (0.02 to 1.00) | 25           | 1000              | A—D                                  |
| Beryllium (1.00 to 3.00) | 5            | 1000 <sup>A</sup> | D—H                                  |
| Cobalt (0.06 to 0.60)    | 25           | 250               | A—D                                  |
| Cobalt (0.60 to 3.00)    | 20           | 1000              | D—H                                  |
| Iron (0.01 to 0.12)      | 50           | 100               | A—D                                  |
| Iron (0.12 to 0.60)      | 50           | 500               | D—H                                  |
| Lead (0.01 to 0.14)      | 50           | 50                | A—D                                  |
| Lead (0.14 to 0.70)      | 50           | 250               | D—H                                  |
| Nickel (0.02 to 0.50)    | 25           | 100               | A—D                                  |
| Nickel (0.50 to 2.50)    | 25           | 500               | D—H                                  |

<sup>A</sup> Employ serial dilution to achieve the final dilution: Dilute 50 mL to 500 mL and then dilute 50 mL of the second dilution to 1000 mL.

## A1.23 Calculation

A1.23.1 When necessary, convert the average readings of each solution to absorbance. Obtain the net absorbance for each calibration solution by subtracting the average absorbance for the zero-calibration solution from the average absorbance of each of the calibration solutions.

A1.23.2 Obtain the net absorbance of each test solution by subtracting the average absorbance of the reagent blank from the average absorbance of the test solutions.

A1.23.3 Prepare a calibration curve by plotting net absorbance for the calibration solutions versus percent analyte element for each of the five analytes.

A1.23.4 Convert the net absorbance of the test solutions to percent analyte by means of the calibration curve.

A1.23.4.1 Most state-of-the-art atomic-absorption spectrophotometers can be calibrated to yield a direct concentration reading. This method of calibration may be used provided that additional calibration solutions are analyzed as samples to test for precision and linearity. Should the instrument be equipped for multi-point calibration, several additional calibration solutions shall be analyzed to ensure that error has not been introduced by the curve-fitting routine.

## A1.24 Precision and Bias

A1.24.1 *Precision*—The precision of this test method is dependent upon the care and precision exercised during instrument calibration and sample preparation, as well as, the purity of the reagents.

A1.24.2 *Bias*—The accuracy of this test method can be judged by analyzing material of known composition

## TEST METHOD C—SILICON BY THE ALUMINUM MOLYBDATE SPECTROPHOTOMETRIC METHOD

### A1.25 Scope

A1.25.1 This test method establishes a procedure for the spectrophotometric determination of silicon in concentrations from 0.01 to 0.30 % in copper-beryllium alloys.

### A1.26 Summary of Test Methods

A1.26.1 The sample is dissolved in a mixture of nitric and fluorohydric (HF) acids. Silicon present in the sample is converted to silicic or fluosilicic acid. An acidic solution of silicic or fluosilicic acid between pH 1.10 and 1.20, when treated with an excess of ammonium molybdate, forms yellow molybdisilicic acid in less than 10 min under the conditions described in this test method. Spectrophotometric measurement is made at 400 nm.

A1.26.1.1 With this test method, better results are normally obtained at 400 nm than at the absorption maximum at 355 nm, due to high and variable background absorption at 355 nm.

### A1.27 Color Stability

A1.27.1 Full color develops in less than 10 min and gradually fades. A uniform for color development should be established and then used for both calibration and test solutions.

### A1.28 Interferences

A1.28.1 Samples in contact with soft glass, such as spectrophotometer cells, may dissolve silica slowly from the glass giving an increased color reading even in the presence of excess boric acid ( $\text{H}_3\text{BO}_3$ ). Samples should be transferred to the spectrophotometer cell just prior to reading.

A1.28.2 Phosphorous present in the final solution in excess of 0.05 mg will interfere unless the solution is treated with citric acid to selectively destroy molybdiphosphoric acid.

### A1.29 Apparatus

A1.29.1 *Spectrophotometer*—Determine the instrument suitable for use as directed in Practice E 60. Instrument response must be adequate to permit an estimation of silicon to within 0.01 % on a sample basis.

### A1.30 Reagents

A1.30.1 *Ammonium Molybdate Solution* (95 g  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}/\text{L}$ )—Dissolve (100 g of  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ ) in water. When turbid, filter and dilute to 1 L.

A1.30.2 *Boric Acid Solution (Saturated)*—Dissolve 60 g of  $\text{H}_3\text{BO}_3$  in hot water. Cool to ambient, allowing the excess boric acid to recrystallize, and filter.

A1.30.3 *Citric Acid Solution* (50 g/L)—Dissolve 5.0 g of citric acid in water and dilute to 100 mL. This solution shall be freshly prepared as needed.

A1.30.4 *Copper (Low Silicon)*—Copper containing less than 0.10 mg silicon.

A1.30.5 *Silicon Standard Solution* (1 mL = 0.10 mg Si)—Fuse 0.2139 g of anhydrous silicon oxide ( $\text{SiO}_2$ ) with 2.0 g of anhydrous sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) in a platinum crucible.

Cool the melt, dissolve completely in water, and dilute to 1 L in a plastic volumetric flask. Store in plastic container.

A1.30.6 *Urea Solution* (100 g/L)—Dissolve 10 g urea in water and dilute to 100 mL. This solution shall be freshly prepared as needed.

### A1.31 Calibration Curve Preparation

A1.31.1 *Calibration Solutions*—Transfer 1.00-g portions of low-silicon copper into each of eight TFE-fluoropolymer 100-mL beakers. Add to each beaker 0.4 mL of HF followed by 11.0 mL  $\text{HNO}_3$  (1 + 2). Cover the beakers with TFE-fluoropolymer watch glasses and let stand for 5 min. Should dissolution not be complete, the beakers may be heated in a water bath at 60 to 65°C. Add 25 mL of the boric acid solution to each of 8 200-mL plastic volumetric flasks. Transfer each of the cool solutions from the beakers into individual volumetric flasks through plastic funnels. Dilute to approximately 100 mL and mix. To seven of the flasks add 2.0, 5.0, 10.0, 15.0, 20.0, 30.0, and 40.0-mL portions respectively. These correspond to silicon concentrations of 0.02, 0.05, 0.10, 0.15, 0.20, 0.30, and 0.40 % respectively on a sample basis. Continue as directed in A1.31.3.

A1.31.1.1 Copper salts decrease the color intensity of the molybdisilicic acid complex. Therefore, it is necessary to have the same amount of copper,  $\pm 100$  mg, present in the final dilutions of both the calibration and test solutions.

A1.31.1.2 The dissolving acid mixture is designed to convert the silicon quantitatively to fluosilicic acid. The HF is necessary to obtain dissolution of refractory silicides and also to prevent the formation of colloidal silicic acid, which does not react with ammonium molybdate.

A1.31.2 *Zero-Calibration Solution*—Treat the solution from A1.31.1 to which no silicon has been added as directed in A1.31.3.

A1.31.3 *Color Development*—Add 10 mL of the urea solution and swirl the flask vigorously. Let stand for 1 to 2 min to allow nitrogen to escape. Add 10.0 mL ammonium molybdate solution, dilute to volume and mix. Let stand for 10 min. Measure the absorbance of the solutions as directed in A1.33.1.

### A1.32 Procedure

#### A1.32.1 Test Solutions:

A1.32.1.1 Transfer three 1.000-g portions of the sample to individual 100-mL TFE fluorocarbon beakers. Add 0.4 mL of HF and 11.0 mL of  $\text{HNO}_3$  (1 + 2). Cover the beakers with TFE-fluorocarbon watch glasses and let stand 5 min. Should dissolution not be complete, the beakers may be heated in a water bath at 60 to 65°C. When dissolution is complete, add 25 mL of boric acid solution to three 200-mL plastic flasks. Transfer the solutions from the three beakers into individual plastic flasks through a plastic funnel. Dilute to approximately 100 mL and mix. Reserve one portion for measurement of the background color and treat the remaining two in accordance with A1.32.2.

A1.32.1.2 Fine particles of metal and light feathery drillings should be avoided, as they react too vigorously with the dissolving mixture. Heavy pieces of metal should also be avoided, as they dissolve too slowly.

#### A1.32.2 Color Development:

A1.32.2.1 Should less than 0.1 phosphorous be present in the final solution, develop the color as described in A1.31.3.

A1.32.2.2 Should 0.1 to 0.5 mg of phosphorous be present in the final solution, develop the color as described in A1.31.3 through the addition of ammonium molybdate solution; then dilute to about 180 mL, mix, and let stand for 10 min. Add 10.0 mL of citric acid, dilute to volume and mix.

A1.32.2.3 Without delay, take the absorbance reading as described in A1.33.1.

A1.32.3 *Background Color*—Treat the solution reserved in A1.32.1 as described in A1.32.2 omitting the addition of the ammonium molybdate solution. Measure the absorbance as described in A1.33.1.

### A1.33 Spectrophotometric Measurements

A1.33.1 Adjust the wavelength setting of the spectrophotometer to 400 nm. Transfer a portion of the zero-calibration solution to a 1-cm pathlength spectrophotometer cell and adjust the instrument readout system to obtain zero absorbance. While maintaining these adjustments, obtain the absorbance readings for the calibration solution, test solutions, and the background color solution using matched 1-cm pathlength cells.

### A1.34 Calculation

A1.34.1 Plot the absorbance readings for the calibration solutions versus percent silicon.

A1.34.2 Obtain the net absorbance of the test solutions by subtracting the absorbance of the background color solution from the absorbance of the test solutions.

A1.34.3 Convert the net absorbance of the test solutions to percent silicon by means of the calibration curve.

A1.34.3.1 Some spectrophotometers can be calibrated to yield a direct concentration reading. This method of calibration may be used provided additional calibration solutions are analyzed as samples to test for the precision and linearity. Should the instrument be equipped for multi-point calibration, several additional calibration solutions should be analyzed to ensure error has not been introduced by the curve-fitting routine.

### A1.35 Precision and Bias

A1.35.1 *Precision*—The precision of this test method is dependent upon the care and precision exercised during instrument calibration and sample preparation, as well as, the purity of the reagents.

A1.35.2 *Bias*—The accuracy of this test method can be judged by analyzing material of known composition.

A1.35.3 The precision and bias of these test methods are being determined.

## APPENDIX

### (Nonmandatory Information)

#### X1. METRIC EQUIVALENTS

X1.1 The SI unit for strength properties now shown is in accordance with the International System of Units (SI). The derived SI unit for force is the newton (N), which is defined as that force which when applied to a body having a mass of one kilogram gives it an acceleration of one metre per second squared ( $N = kg \cdot m/s^2$ ). The derived SI unit for pressure or

stress is the newton per square metre ( $N/m^2$ ), which has been named the pascal (Pa) by the General Conference on Weights and Measures. Since  $1 \text{ ksi} = 6\,894\,757 \text{ Pa}$  the metric equivalents are expressed as megapascal (MPa), which is the same as  $MN/m^2$  and  $N/mm^2$ .

## SUMMARY OF CHANGES

This section identifies principle changes to this specification that have been incorporated since the 1996 issue as follows:

(1) Document was revised to meet the requirements of *Form and Style for ASTM Standards* (Blue Book) and the Committee

B05 *Outline of Form of Specifications* (OFS).

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