



# Standard Specification for Electrolytic Copper Cathode<sup>1</sup>

This standard is issued under the fixed designation B115; 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.

## 1. Scope\*

1.1 This specification establishes the requirements for electrolytic copper cathode; electrorefined and electrowon.

1.2 *Units*—The 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.

NOTE 1—Cathode produced to this specification corresponds to the designation “Cath” as defined in Classification B224 and may be used to produce all other coppers listed in Classification B224 that are normally produced from “Cath” copper.

NOTE 2—Grade 1 cathode conforms to the chemical compositional requirements of Copper UNS No. C11040, except for oxygen, and is suitable for the manufacture of wire rod as designated in Specification B49.

1.3 The following hazard caveat applies to the test methods described in Annex A2 of this specification: *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 ASTM Standards:<sup>2</sup>

- B49 Specification for Copper Rod for Electrical Purposes
- B193 Test Method for Resistivity of Electrical Conductor Materials
- B224 Classification of Coppers
- B846 Terminology for Copper and Copper Alloys
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

<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 B05.07 on Refined Copper.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

## E53 Test Method for Determination of Copper in Unalloyed Copper by Gravimetry

## 3. Terminology

3.1 For definition of general terms related to copper and copper alloys, refer to the current editions of Classification B224 and Terminology B846.

## 4. Ordering Information

4.1 Orders for product shall include the following information, as applicable:

- 4.1.1 ASTM designation and year of issue (for example, B115 – XX),
- 4.1.2 Cathode grade (Table 1),
- 4.1.3 Size; full cathode or cut,
- 4.1.4 Total weight of each size.

## 5. Chemical Composition

5.1 The cathode furnished shall conform to the requirements of Table 1 for the grade specified in the contract or purchase order.

5.2 These composition limits do not preclude the presence of other elements. Limits may be established and analysis required for unnamed elements by agreement between the supplier and the purchaser and such agreement shall be part of the contract or purchase order.

## 6. Physical Property Requirements

### 6.1 Electrical Resistivity:

6.1.1 The maximum electrical resistivity for product produced from Grade 2 cathode shall be  $0.153\,28\,\Omega\cdot\text{g}/\text{m}^2$  (conductivity 100.0 % minimum IACS) at 20°C (68°F) annealed<sup>3</sup> when tested in accordance with Test Method B193. Measurement error is not included in the maximum/minimum limit.

## 7. Dimensions, Mass, and Permissible Variations

7.1 Full-size cathodes or cathodes cut to size may be supplied as agreed upon between supplier and purchaser.

<sup>3</sup> NBS Notebook 100 available from National Technical Information Service (NTIS), 5301 Shawnee Rd, Alexandria, VA 22312, <http://www.ntis.gov>.

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

**TABLE 1 Chemical Composition**

| Element                 | Grade 1 <sup>A</sup>    | Grade 2 <sup>A</sup> |
|-------------------------|-------------------------|----------------------|
|                         | Percent, %              |                      |
|                         | 99.95, min <sup>B</sup> |                      |
|                         | ppm <sup>C</sup>        |                      |
| Copper                  |                         |                      |
| Selenium, max           | 2                       | 10                   |
| Tellurium, max          | 2                       | 5                    |
| Bismuth, max            | 1.0                     | 3                    |
| Group total, max        | 3                       | ...                  |
| Antimony, max           | 4                       | 15                   |
| Lead, max               | 5                       | 40                   |
| Arsenic, max            | 5                       | 15                   |
| Iron, max               | 10                      | 25                   |
| Nickel, max             | 10                      | 20                   |
| Tin, max                | 5                       | 10                   |
| Sulfur, max             | 15                      | 25                   |
| Silver, max             | 25                      | 70                   |
| Maximum allowable total | 65                      | ...                  |

<sup>A</sup> Measurement error is not incorporated in the maximum limits, refer to 10.1.1.

<sup>B</sup> Including silver.

<sup>C</sup> Determined from a melted sample.

## 8. Workmanship, Finish, and Appearance

8.1 Cathodes shall withstand ordinary handling without breakage or excessive separation of nodules. They shall be substantially free of all foreign material; for example, copper sulfate, dirt, grease, and oil.

## 9. Sampling

9.1 For routine sampling of cathodes for analysis, the method of sampling shall be at the discretion of the sampler.

9.2 In case of dispute concerning sampling for chemical composition, or electrical resistivity, or both, the method of sampling shall be in accordance with Annex A1.

9.3 In case of special requirements specified in the purchase order or contract, the method of sampling shall be as agreed between the supplier and the purchaser.

## 10. Number of Tests and Retests

### 10.1 Tests:

10.1.1 Chemical composition shall be determined as the per element mean of at least two replicate analyses of each sample.

10.1.2 Electrical resistivity shall be determined as the mean of results from four specimens.

### 10.2 Retests:

10.2.1 In the case of compositional or resistivity dispute, retests may be made under the conditions of 9.2.

### 10.3 Umpire Test:

10.3.1 In the case in which retest does not settle the dispute, further retest may be made by a qualified third-party laboratory agreeable to both parties. This provision does not preclude other contractual agreements.

## 11. Specimen Preparation

11.1 For routine testing, specimen preparation shall be at the discretion of the preparer.

11.2 In the case of special requirements specified in the purchaser order or contract, specimen preparation shall be as agreed between the supplier and the purchaser.

11.3 In the case of dispute concerning specimen preparation for chemical composition specified in Table 1 or electrical resistivity, specimen preparation shall be in accordance with Annex A1.

## 12. Test Methods

### 12.1 Chemical Composition:

12.1.1 For routine analysis of Grade 1 and Grade 2 cathode, the methods of analysis used shall be at the discretion of the analyst.

12.1.2 In the case of dispute concerning the chemical composition, the methods of analysis shall be in accordance with Annex A2, except for copper in Grade 2 cathode.

12.1.3 In the case of dispute concerning copper content of Grade 2 cathode, the method of analysis shall be in accordance with Test Methods E53.

12.1.4 In the case of dispute concerning special requirements stated in the purchase order or contract, the methods of analysis used shall be as agreed between the supplier and the purchaser.

### 12.2 Electrical Resistivity:

12.2.1 In the case of dispute concerning electrical resistivity, the method of testing shall be in accordance with Test Method B193.

## 13. Significance of Numerical Limits

13.1 Calculated values shall be rounded to the desired number of places as directed in Practice E29.

## 14. Inspection

14.1 The producer shall inspect the product and conduct such tests as are necessary to verify that the requirements of this specification are met.

## 15. Rejection and Rehearing

### 15.1 Rejection:

15.1.1 Product that fails to conform to the requirements of this specification may be rejected.

15.1.2 Rejection shall be reported to the producer or supplier promptly and in writing.

15.1.3 In the case of disagreement or dissatisfaction with the results of the test upon which rejection was based, the producer or supplier may make claim for a rehearing.

### 15.2 Rehearing:

15.2.1 As a result of product rejection, the supplier may make claim for retest to be conducted by the producer or supplier and the purchaser. Samples of the rejected product shall be taken in accordance with this specification and tested by both parties as directed in this specification, or, alternatively, upon agreement between both parties, an independent laboratory may be selected for the tests using the test methods prescribed in this specification.

## 16. Packaging and Package Marking

### 16.1 Packaging:

16.1.1 Cathodes, whether full size or cut, shall be assembled in bundles or containers of suitable weight for handling and

shall be prepared for shipment in such a manner as to ensure acceptance by common carrier for transportation and to afford protection from normal hazards of transportation.

#### 16.2 *Package Marking:*

16.2.1 Each cathode bundle or container shall be marked to identify source and grade.

16.2.2 When used, metallic identifying markers shall be firmly attached only to the strapping or shipping container.

### 17. Keywords

17.1 cathode; copper; electrolytic copper; electrorefined copper; electrowon copper

## ANNEXES

### (Mandatory Information)

#### A1. SAMPLING AND SPECIMEN PREPARATION OF ELECTROLYTIC CATHODE COPPER FOR DETERMINATION OF COMPLIANCE WITH SPECIFICATION REQUIREMENTS

##### A1.1 Scope

A1.1.1 This practice establishes a procedure for the sampling and specimen preparation of electrolytic copper cathodes, Grades 1 and 2, for the determination of conformance with specification requirements.

A1.1.2 *Units*—The values stated in inch-pound units are the standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

A1.1.3 *This standard does not purport to address 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.*

##### A1.2 Terminology

###### A1.2.1 *Definitions of Terms Specific to This Standard:*

A1.2.1.1 *lot*—One shipment, or part of one shipment, produced by one refiner. For use other than continuous cast rod production, shipments greater than 200 tons short shall be subdivided into lots not exceeding 200 tons each for sampling purposes.

A1.2.1.2 *gross sample*—The total number of test pieces selected from a lot and considered representative of the lot.

A1.2.1.3 *test piece*—An individual cathode, or cathode part, randomly selected from the lot.

A1.2.1.4 *sample*—A portion prepared from the gross sample and considered representative of the gross sample.

A1.2.1.5 *specimen*—Representative fraction taken from the sample for test.

##### A1.3 Selection of Cathode

A1.3.1 Nodules shall not be considered a sample representative of the lot.

###### A1.3.2 *Cathodes for Continuous Rod Casting:*

A1.3.2.1 The cathodes shall be available in the original packing for examination.

A1.3.2.2 The quantity of cathodes required shall be that necessary to flush the system plus 1 hour of melting furnace operation.

A1.3.2.3 All cathode bundles shall be numbered and a random number generator shall be used to determine which bundles shall be selected for the gross sample.

A1.3.2.4 Should there be an insufficient quantity of cathodes to comply with **A1.3.2.2**, then the procedure described in **A1.3.3** shall apply.

###### A1.3.3 *Cathodes for Other Uses:*

A1.3.3.1 Not less than 25 % of the original lot weight or 25 tons, whichever is the larger, shall be available in the original packing for examination.

A1.3.3.2 A gross sample of 24 cathodes, or the equivalent in sheared cathode pieces, shall be selected from a lot. To guarantee random selection, all cathodes, or sheared cathode pieces, of the lot shall be individually numbered, and a random number generator shall be used to select the individual test pieces.

A1.3.3.3 In the case of sheared cathodes, 24 full cathodes; 48 half-plate cathodes, 24 each of tops and bottoms; 96 quarter-plate cathodes, and 24 each of the four quarters, shall be selected.

A1.3.3.4 The selection of test pieces of cathode sheared smaller than quarter plate shall be by agreement between the producer, or the supplier, and the purchaser.

A1.3.3.5 Alternatively, to avoid individual numbering of cathodes, or sheared cathode pieces, in the case of large lots, provided both parties agree, individual bundles, or containers, may be selected on a random basis, and then individual cathodes, or sheared cathode pieces, within each bundle, or container, shall be numbered and test pieces selected, using a random number generator as just described.

##### A1.4 Sample Preparation

###### A1.4.1 *Cathode for Continuous Rod Casting:*

A1.4.1.1 The portion used for flushing the system shall not be used for sampling.

A1.4.1.2 The remaining gross sample, minimum of one hour's cast, shall be charged to the melting furnace. The rod coils produced from the caster shall be sequentially numbered, excluding any coils with obvious defects normally attributed to the rod casting process.

A1.4.1.3 *Chemical Composition*—Each party shall select 2 coils from which a segment of rod approximately 16 in. (406

mm) in length shall be cut at the trailing ends of the coils. Each rod segment shall be cut into 4 portions of approximate equal lengths. The 16 portions shall be divided into 4 groups; each group shall contain one portion from each of the 4 original rod segments. The 4 groups of rod portions shall be placed in separate noncontaminating containers, then sealed and identified for the supplier, the purchaser, contingency, and umpire if necessary.

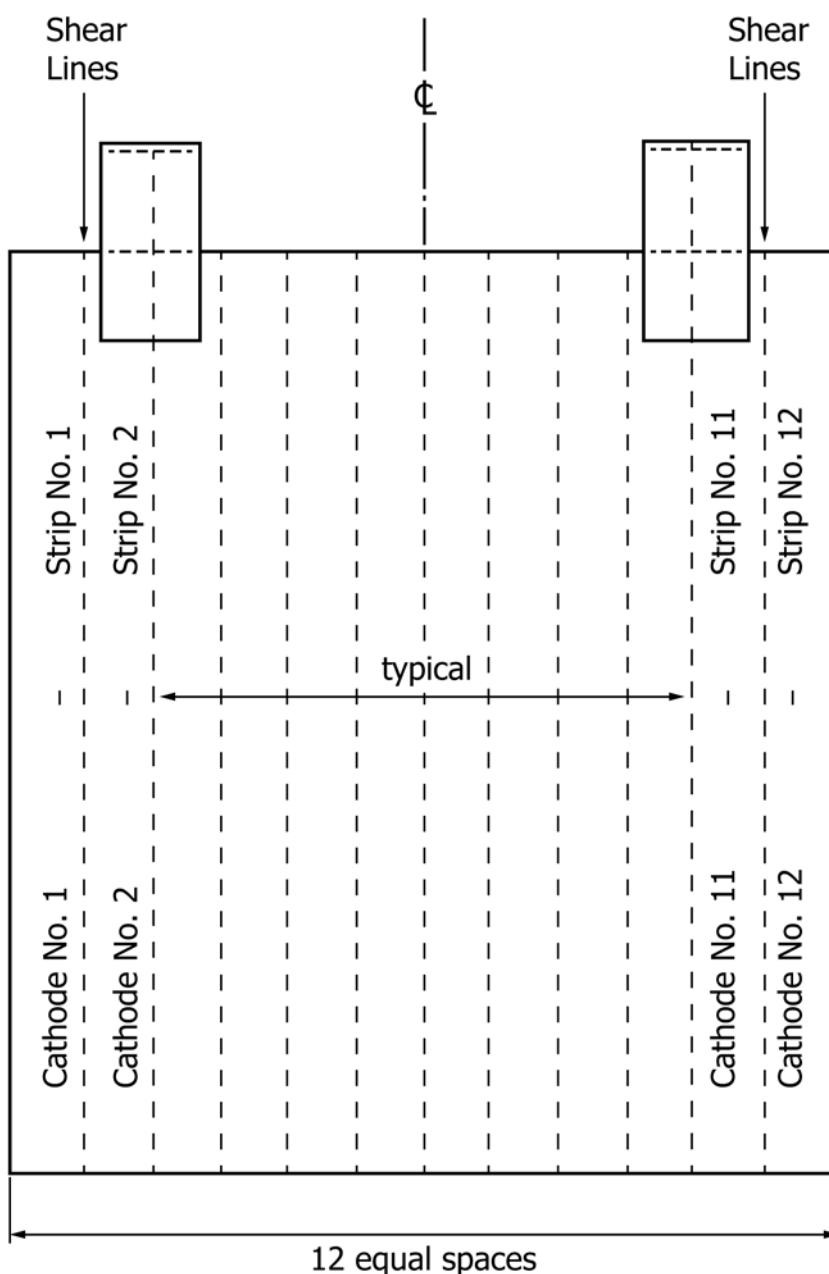
A1.4.1.4 *Electrical Resistivity*—Each party shall select 2 coils from which a rod segment of sufficient length for test shall be taken from the trailing ends of the coils. Each rod segment shall be cold drawn into a wire about 0.080 in.

diameter (2.0 mm) and at least 160 in. in length (4 m). Each wire coil shall be cut into 4 portions of approximately equal length, and the 16 portions shall be individually identified. The 16 wires shall be divided into 4 groups of 4 wires each, one from each of the 4 original rod segments; one group each for the producer, the purchaser, and the umpire, if necessary.

#### A1.4.2 *Cathodes for Other Uses:*

##### A1.4.2.1 *Chemical Composition:*

(a) From each cathode, or sheared cathode piece, of the gross sample a vertical strip shall be cut in such a position (see Fig. A1.1) that the collection of the strips so cut represents all



NOTE 1—Repeat for second set of twelve cathodes.

FIG. A1.1 Vertical Strip Sampling Pattern (Refer to A1.4.2.1(a) of text)

points of the cathode, or sheared cathode piece, including the loops (hangers) for full cathode. All vertical sections shall be approximately the same width and cut sequentially from left to right in the same order as that in which the cathodes were selected.

(b) The strips selected shall be immersed in 10 % volume/volume (v/v) hydrochloric acid at approximately 20°C for 15 min and then removed and washed in distilled or deionized water until all visible extraneous contamination has been removed.

(c) Where excessive copper sulfate surface contamination is evident, the parties shall confer to determine the extent of washing.

(d) An electric induction or resistance furnace equipped with a graphite, or other noncontaminating crucible and a close-fitting lid of the same material with provision for an inert atmosphere within the crucible shall be used for melting the selected strips.

(e) The crucible shall first be cleaned by melting in it a quantity of copper from the lot in question. The melt shall be discarded.

(f) The prepared cathode strips shall be melted in the cleaned crucible under an inert atmosphere. The molten metal shall be thoroughly stirred with a clean graphite or other noncontaminating rod.

(g) Where the available crucible is not large enough to melt the composite sample, the 24 strips shall be grouped into 2 or more batches of approximately equal weight for melting. In such cases, the metal from each melt shall be separately sampled.

(h) The metal shall be sampled by one of the following methods:

(1) *Ingots*: Equal portions of the molten metal shall be cast into graphite ingot moulds at the beginning, middle, and end of the casting operation. The moulds shall provide ingots that are at least  $\frac{3}{4}$  by  $\frac{3}{4}$  in. (20 by 20 mm) in cross section and 4 to 8 in. (100 to 200 mm) in length. A sufficient number of ingots shall be cast to provide in excess of 28 oz (800 g) of small chips when drilled, milled, or sawn, using carbide-tipped tools. The surplus metal not cast into ingots may be discharged by any convenient means.

(2) *Shot*: Remove a portion of the molten metal using a ladle coated with a noncontaminating mould wash. The molten metal shall be poured into a container of deionized or distilled water until shot in excess of 28 oz (800 g) has been produced. The depth of the water shall be such that the metal will not adhere to the container. Before sampling, the ladle shall be brought to the molten metal temperature, and the pouring rate shall be such that no metal will solidify in the ladle. The surplus metal may be discharged by any convenient means.

(3) *Pin Samples*—Take in excess of 28 oz (800 g) from the molten metal by using either commercially available evacuated glass tubes of several millimetres in diameter and 100 to 120 mm in length.

NOTE A1.1—If the vacuum pump method is elected, it is recommended that the user ensure the cleanliness of the copper tube, and the level of the impurities, if any, in the tube metal be determined to avoid potential specimen contamination.

(4) Divide the sample taken into 4 portions of approximately 7 oz (200 g) each and sealed in a noncontaminating package and individually identified; one each for the producer, the purchaser, contingencies, and the umpire, if necessary.

#### A1.4.2.2 *Electrical Resistivity*:

(a) A minimum of 4 castings shall be made by pouring the molten metal from (f) in A1.4.2.1 into a chill cast mould of sufficient size to produce a wire approximately 0.080 in. in diameter (2.0 mm) and at least 160 in. (approximately 4 m) in length.

(b) Alternatively, the disputing parties may agree to obtain a sample by drilling selected cathodes along the diagonals to obtain a total of about 140-oz (4000-g) drillings. The drillings shall be melted as described in (d) through (f) of A1.4.2.1 and chill cast as described in the preceding paragraph.

(c) The cast form may be hot worked initially, provided care is taken to avoid contamination, or excessive oxidation, or both. The external oxide shall be removed and the sample cold drawn. Each wire coil shall be cut into 4 portions of approximately equal lengths, the 16 portions thus obtained shall be divided into 4 groups of 4 wires each, one from each of the 4 original castings; one group each for the producer, the purchaser, contingencies, and the umpire if necessary.

### A1.5 Specimen Preparation

A1.5.1 *Continuous Cast Rod: Chemical Composition*—Chips, clippings, or drillings shall be taken from the rod sample using a noncontaminating tool.

A1.5.2 *Continuous Cast Rod: Electrical Resistivity*—The wire specimens shall be annealed in an inert atmosphere at approximately 500°C (932°F) for 30 min and cooled to ambient temperature under inert atmosphere. When cool, the wires are removed and cut to test length.

#### A1.5.3 *Cathodes for Others Uses: Chemical Composition*:

A1.5.3.1 Drillings from A1.4.2.1 (see *Ingots*: (1)) shall be etched in 50 % (v/v) nitric acid until the reaction is clearly visible, then washed four times with distilled or deionized water, once with alcohol or acetone, and air dried.

A1.5.3.2 Clean the shot (see *Shot*: (2)), as described in A1.5.3.1.

A1.5.3.3 Extreme care must be exercised in the removal of all glass from samples taken with evacuated glass tubes to avoid contamination from the glass.

A1.5.4 *Cathodes for Other Uses: Electrical Resistivity*—Prepare as described in A1.5.2.

## A2. TEST METHODS FOR DETERMINATION OF COMPLIANCE WITH CHEMICAL COMPOSITION REQUIREMENTS FOR ELECTROLYTIC COPPER CATHODE

### A2.1 Scope

A2.1.1 These test methods establish the procedures for the chemical analysis of electrolytic copper cathode for the elements with the specified limiting value stated in **Table 1** of this specification.

A2.1.2 *These test methods do not purport to address all of the safety concerns, if any, associated with their use. It is the responsibility of the user of these test methods to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Special hazard statements are given in **A2.10** and **A2.22**.

A2.1.3 The test methods are arranged in the following order:

|                                                                                                                                                   | Sections             |
|---------------------------------------------------------------------------------------------------------------------------------------------------|----------------------|
| Antimony, arsenic, bismuth, iron, lead, nickel, selenium, silver, tellurium, and tin by electrothermal atomization atomic absorption spectrometry | <b>A2.7 – A2.17</b>  |
| Sulfur by combustion and infrared detector                                                                                                        | <b>A2.18 – A2.29</b> |

### A2.2 Significance and Use

A2.2.1 These test methods are intended to test electrolytic copper cathode for compliance with chemical composition requirements of Specification B115.

### A2.3 Apparatus

A2.3.1 Apparatus required for each determination is listed in separate sections preceding the procedure.

### A2.4 Reagents and Material

A2.4.1 Reagents and materials required for each test method are listed in a separate section in the test method.

### A2.5 Sampling

A2.5.1 Sampling shall be in accordance with specification requirements.

### A2.6 Rounding Calculated Values

A2.6.1 Calculated values shall be rounded to the desired number of places as directed in Practice **E29**.

## TEST METHOD A—ANTIMONY, ARSENIC, BISMUTH, IRON, LEAD, NICKEL, SELENIUM, SILVER, TELLURIUM, AND TIN BY ELECTROTHERMAL ATOMIZATION ATOMIC ABSORPTION SPECTROSCOPY

### A2.7 Scope

A2.7.1 This test method covers the determination of antimony, arsenic, bismuth, iron, lead, nickel, selenium, silver, tellurium, and tin in electrolytic cathode copper.

### A2.8 Summary of Test Method A

A2.8.1 The test sample is dissolved in nitric acid and the solution diluted to a known volume. An aliquot is introduced

into an electrothermal atomic absorption spectrometer with background correction capability. The absorption of the resonance line energy from the spectrum of the element is measured and compared with that of calibration solutions of the same element in a matched matrix.

### A2.9 Significance and Use

A2.9.1 This test method is intended to test electrolytic cathode copper for compliance with antimony, arsenic, bismuth, iron, lead, nickel, selenium, silver, tellurium, and tin requirements of the specification.

### A2.10 Interferences

A2.10.1 Elements normally present in electrolytic cathode copper do not interfere.

### A2.11 Hazards

A2.11.1 **Warning**—The ultraviolet radiation must be shielded at all times to prevent eye damage.

A2.11.2 Arsenic trioxide ( $\text{As}_2\text{O}_3$ ) is a hazardous reagent and may be fatal if swallowed. Inhalation and prolonged or repeated skin contact are to be avoided.

A2.11.3 Tellurium and tellurium compounds are hazardous reagents and may be fatal if ingested. Avoid inhalation and prolonged or repeated skin contact.

A2.11.4 Selenium and selenium compounds are potentially hazardous reagents. Avoid ingestion, inhalation, or prolonged and repeated skin contact.

A2.11.5 For other specific precautions, refer to Practice **E50**.

#### A2.11.6 Technical Hazards—Warnings

A2.11.6.1 It is essential that acids and water be carefully checked for purity to avoid contamination from this source.

A2.11.6.2 Laboratory glassware should be thoroughly cleaned, soaked in 9 % by volume  $\text{HNO}_3$  for several hours, and rinsed before use. Previously etched glassware should be avoided.

A2.11.6.3 Effects of nonspecific absorption and light scattering must be compensated by matrix matching of calibration solutions and background correction.

A2.11.6.4 *Matrix Modifiers*—The copper matrix reduces loss for most elements during the char step. Modifiers such as magnesium nitrate may be found useful to stabilize further elements like nickel and tin.

A2.11.6.5 Should lack of homogeneity be suspect in the test material, a 10-g sample, weighed to the nearest 1 mg should be taken and diluted to 1 L with the appropriate amount of acid.

A2.11.6.6 The lower limit of elemental determination is affected by the residual level of the element in the copper.

A2.11.6.7 Optimum settings for operating parameters vary instrument to instrument and must be experimentally established for a particular instrument.

## A2.12 Apparatus

**A2.12.1 Atomic Absorption Spectrometer and Electrothermal Atomizer**—The instrument shall be equipped with a background corrector and high speed readout electronics or a high-speed recorder, or both. The instrument should be capable of using single-element hollow cathode lamps or electrodeless discharge lamps. Follow the manufacturer's manual for installation and system operation.

**A2.12.2 Graphite Tubes**—Pyrolytically coated graphite tubes and L'vov platforms for use in the electrothermal atomizer.

**A2.12.3 Micropipets**—5 to 250  $\mu\text{L}$ .

**A2.12.4 Operating Parameters**—Determine the sample size and optimum electrothermal atomizer parameters for the type of atomizer used as recommended by the instrument manufacturer. The analytical lines are as follows:

| Element   | Wavelength, nm |
|-----------|----------------|
| Antimony  | 217.6          |
| Arsenic   | 193.9          |
| Bismuth   | 223.0          |
| Iron      | 248.3          |
| Lead      | 283.3          |
| Nickel    | 232.0          |
| Selenium  | 196.0          |
| Silver    | 328.1          |
| Tellurium | 214.3          |
| Tin       | 224.6          |

## A2.13 Reagents and Materials

**A2.13.1 Acids**—Acids, hydrochloric and nitric, should be carefully checked for purity to ensure they do not contaminate the analysis.

**A2.13.2 Water**—The quality of the water should be carefully checked for purity to ensure it does not contaminate the analysis.

**A2.13.3 Argon**—Purity: 99.98 %, min.

**A2.13.4 Copper Solution (1 mL = 50-mg Cu)**—Transfer 10 g of certified high purity copper (NBS SRM 393 or equivalent) into a 250-mL beaker. Add 25-mL water and 25-mL  $\text{HNO}_3$  in 5-mL increments. After the last increment addition, heat gently to dissolve the copper and expel the brown fumes. Cool, transfer to a 200-mL volumetric flask, dilute to volume with 50 % by volume  $\text{HNO}_3$  and mix.

**A2.13.4.1 Known impurities in the copper metal** must be considered when determining specific element ppm concentration in [Table A2.1](#) and [Table A2.2](#).

**A2.13.5 Antimony Standard Solution (1 mL = 0.10-mg Sb)**—Dissolve 0.2740 g of potassium antimony tartrate

**TABLE A2.1 Calibration Solution**

| Flask No. | $\mu\text{L}$ | ppm: As, Sb, Bi, Fe, Pb, Ni, Ag, Se, and Te |
|-----------|---------------|---------------------------------------------|
| 1         | 5             | 0.5                                         |
| 2         | 10            | 1.0                                         |
| 3         | 25            | 2.5                                         |
| 4         | 50            | 5.0                                         |
| 5         | 100           | 10.0                                        |
| 6         | 250           | 25.0                                        |

**TABLE A2.2 Calibration Solution**

| Flask No. | $\mu\text{L}$ | ppm: As, Sb, Bi, Fe, Pb, Ni, Se, Sn, and Te |
|-----------|---------------|---------------------------------------------|
| 7         | 5             | 0.5                                         |
| 8         | 10            | 1.0                                         |
| 9         | 25            | 2.5                                         |
| 10        | 50            | 5.0                                         |
| 11        | 100           | 10.0                                        |
| 12        | 250           | 25.0                                        |

( $\text{KSbC}_4\text{H}_4\text{O}_7 \cdot 1/2\text{H}_2\text{O}$ ; purity: 99.9 %, min) with water in a 250-mL beaker. Transfer to a 500-mL volumetric flask, dilute to volume and mix.

**A2.13.6 Arsenic Standard Solution (1 mL = 0.10-mg As)**—Dissolve 0.1320 g of arsenic trioxide ( $\text{As}_2\text{O}_3$ ; purity: 99.9 %, min) in a 100-mL beaker with one or two pellets of potassium hydroxide (KOH) in 50 mL of water. Heat gently to dissolve the salt. Transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.7 Bismuth Standard Solution (1 mL = 0.10- mg Bi)**—Dissolve 50 mg of bismuth (Bi; purity: 99.90 %, min) in 10 mL of 25 % by volume  $\text{HNO}_3$ . Heat gently to dissolve the metal and expel the brown fumes. Cool, transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.8 Iron Standard Solution (1 mL = 0.10-mg Fe)**—Dissolve 50 mg of iron (Fe; purity: 99.9 %, min) in 10-mL  $\text{HNO}_3$ . Heat gently to dissolve the iron and expel the brown fumes. Cool, transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.9 Lead Standard Solution (1 mL = 0.10-mg Pb)**—Dissolve 50-mg lead (Pb; purity: 99.9 %, min) in 10 mL of 25 % by volume  $\text{HNO}_3$ . Heat gently to dissolve the lead and expel the brown fumes. Cool, transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.10 Nickel Standard Solution (1 mL = 0.10-mg Ni)**—Dissolve 50-mg nickel (Ni; purity: 99.9 %, min) in 20 mL of 50 % by volume  $\text{HNO}_3$ . Heat gently to dissolve the nickel and expel the brown fumes. Cool, transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.11 Selenium Standard Solution (1 mL = 0.10-mg Se)**—Dissolve 70.3-mg selenium dioxide ( $\text{SeO}_2$ ; purity: 99.0 %, min) in 50-mL water. Transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.12 Silver Standard Solution (1 mL = 0.10-mg Ag)**—Dissolve 50-mg silver (Ag; purity: 99.9 %, min) in 20 mL of 50 % by volume  $\text{HNO}_3$ . Heat gently to dissolve the silver and expel the brown fumes. Cool, transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.13 Tellurium Standard Solution (1 mL = 0.10-mg Te)**—Dissolve 50-mg tellurium (Te; purity: 99.9 %, min) in 10-mL  $\text{HNO}_3$ . Heat gently to dissolve the tellurium and expel the brown fumes. Cool, transfer to a 500-mL volumetric flask. Add 50-mL  $\text{HNO}_3$ , dilute to volume and mix.

**A2.13.14 Tin Standard Solution (1 mL = 0.10-mg Sn)**—Dissolve 50-mg tin (Sn; purity: 99.9 %, min) in 75 mL of 33 %

by volume HCl. Heat gently to dissolve the tin. Cool, transfer to a 500-mL volumetric flask, dilute to volume and mix.

## **A2.14 Calibration**

A2.14.1 *Calibration Solutions*—Using micropipets, transfer to individual 100-mL volumetric flasks the volume of each standard solution as indicated in [Table A2.1](#) and [Table A2.2](#):

A2.14.1.1 Add 20 mL of the copper standard solution to each flask in both tables, dilute to volume and mix. Known impurities in the copper standard solution must be considered when determining final specific element ppm concentration in both tables.

### *A2.14.2 Instrument Parameters:*

A2.14.2.1 Set the required instrument parameters and align the electrothermal atomizer according to the manufacturer's recommendation.

A2.14.2.2 Determine the optimum electrothermal atomizer parameters for the particular type atomizer and sample size as recommended by the instrument manufacturer.

### *A2.14.3 Spectrometry:*

A2.14.3.1 Zero the instrument or set the base line on the recorder, or both.

A2.14.3.2 Check the zero stability and lack of spectral interference within the atomization system by running the preset heating program for blank firing of the electrothermal atomizer. Repeat to ensure baseline stability.

A2.14.3.3 Inject and atomize the calibration solutions in the order of increasing concentrations. Inject each solution three times and record the readings. Should good replication not be achieved, repeat the process.

A2.14.3.4 Check for memory effects by running the blank firing program and reset the zero, or baseline, if necessary.

A2.14.3.5 Plot the average reading from each calibration versus concentration of the analyte in the calibration solution.

A2.14.3.6 For systems with direct instrument calibration, ensure that a sufficient number of each calibration solutions is injected and atomized to determine that proper calibration is achieved.

## **A2.15 Procedure**

A2.15.1 Dissolve a 1-g sample, weighed to the nearest 1 mg, in a 100-mL beaker with 20 mL of 50 % by volume HNO<sub>3</sub>. Heat gently to dissolve the copper and expel the brown fumes. Transfer to a 100-mL volumetric flask. Cool, dilute to volume, and mix.

A2.15.2 Ensure that the test solution is within 1°C of the calibration solutions. Inject and atomize the test solution for three readings and record the observations.

## **A2.16 Calculations**

A2.16.1 Calculate the concentration of each element to be determined using the analytical curves prepared in [A2.14.3.5](#).

A2.16.2 Systems with direct reading capability will provide results in the calibration concentration units.

## **A2.17 Precision and Bias**

A2.17.1 *Precision*—It is not possible to specify the precision of this test method but it is dependent upon the care given to sample preparation of the calibration solutions as well as the purity of the reagents.

A2.17.2 *Bias*—No information can be presented on the bias of this test method but it is dependent on the care given to the preparation of the calibration solutions as well as the purity of the reagents.

## **TEST METHOD B—SULFUR BY COMBUSTION AND INFRARED DETECTOR**

## **A2.18 Scope**

A2.18.1 This test method covers the determination of sulfur in electrolytic cathode copper.

## **A2.19 Summary of Test Method B**

A2.19.1 The sulfur is converted to sulfur dioxide (SO<sub>2</sub>) by combustion in a stream of oxygen and the SO<sub>2</sub> is measured by infrared absorption.

A2.19.2 This test method is written for use with commercial analyzers equipped to carry out the operations automatically.

## **A2.20 Interferences**

A2.20.1 The elements ordinarily present do not interfere.

## **A2.21 Apparatus**

A2.21.1 *Combustion and Analyzing Instrumentation*, capable of making the required measurements.

## **A2.22 Reagents and Material**

### *A2.22.1 Reagents:*

A2.22.1.1 *Accelerator*—Use the accelerator recommended by the instrument manufacturer which, for copper, should be sulfur and tin free.

A2.22.1.2 *Oxygen*, ultra high purity (purity: 99.95 % min): Other grades of oxygen may be used if sulfur free, or the oxygen may be purified as described in Practice [E50](#).

### *A2.22.2 Materials:*

A2.22.2.1 *Crucibles*—Use crucibles recommended by the manufacturer, or equivalent.

A2.22.2.2 *Crucible Tongs*, capable of handling recommended crucibles.

## **A2.23 Hazards**

A2.23.1 For precautions to be observed in the use of certain reagents in this test method refer to Practice [E50](#).

A2.23.2 Use care when handling hot crucibles and operating the furnace to avoid burns and electrical shock.

## **A2.24 Preparation of Apparatus**

A2.24.1 Assemble the apparatus and test the apparatus as recommended by the manufacturer.

## **A2.25 Sample Preparation**

A2.25.1 The sample should be uniform in size but not finer than 40 mesh.

**A2.26 Calibration**

A2.26.1 *Calibration Reference Materials*—Select a minimum of two reference materials with sulfur content near the mid-point and high limit.

A2.26.2 *Instrument Calibration*—Calibrate according to the manufacturer's instructions.

**A2.27 Procedure**

A2.27.1 Stabilize the furnace and analyzer according to the manufacturer's instruction.

A2.27.2 Transfer the weight of sample recommended by the manufacturer into a crucible and the add same amount of accelerator used in the calibration. Proceed as directed by the manufacturer's instructions.

**A2.28 Calculations**

A2.28.1 Since most commercially available instruments calculate percent concentrations directly, including corrections for blank and sample weight, calculations by the analyst are not required.

**A2.29 Precision and Bias**

A2.29.1 *Precision*—It is not possible to specify the precision of this test method but it is dependent upon the care given to sample preparation and the preciseness of calibration.

A2.29.2 *Bias*—No information can be presented on the bias of this test method but it is dependent on the care given to the preparation and analysis of the sulfur standards as well as their homogeneity.

**SUMMARY OF CHANGES**

Committee B05 has identified the location of selected changes to this standard since the last issue (B115 – 00 (2004)) that may impact the use of this standard. (Approved April 1, 2010.)

- (1) Annex A1.3.2.2 changed from “1 h” to “1 hour”.
- (2) The pin sampling method in Annex A1 using copper tubing and the suction apparatus shown in Figure A1.2 deleted.

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