Designation: E53 – 07 (Reapproved 2013)

Standard Test Method for Determination of Copper in Unalloyed Copper by Gravimetry[1]

This standard is issued under the fixed designation E53; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

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

1. Scope

1.1 This test method covers the chemical analysis of copper having minimum purity of 99.75 % to 99.95 %.

1.2 This test method covers the electrolytic determination of copper in chemical, electrolytic, and fire refined copper. In this method silver is deposited with the copper, and is reported as copper.

1.3 *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.* Specific precautionary statements are given in 8.4 and Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

E121 Test Methods for Chemical Analysis of Copper-Tellurium Alloys (Withdrawn 2010)[3]

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

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E255 Practice for Sampling Copper and Copper Alloys for the Determination of Chemical Composition

E1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry (Withdrawn 2004)[3]

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

3. Terminology

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

4. Summary of Test Method

4.1 The sample is dissolved in an acid mixture and the copper is electrolytically deposited and weighed on a tared platinum cathode. Copper remaining in the electrolyte is determined by atomic absorption spectroscopy.

5. Significance and Use

5.1 This test method for the chemical analysis of copper is primarily intended to test for compliance with compositional specifications. It is assumed that all who use this method 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.

6. Interferences

6.1 Elements normally present in refined copper with a minimum purity of 99.85 % do not interfere.

6.2 Approximately one-half of any selenium or tellurium present will co-deposit. If interfering amounts are present, proceed in accordance with Test Methods E121.

7. Apparatus

7.1 *Electrodes for Electroanalysis:*

7.1.1 *Electrodes*—Recommended stationary type platinum electrodes are described in 7.1.2 and 7.1.3. The surface of the platinum electrodes should be smooth, clean, and bright to promote uniform deposition and good adherence. Deviations from the exact size and shape are allowable. In instances where it is desirable to decrease the time of deposition and agitation

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.05 on Cu, Pb, Zn, Cd, Sn, Be, Precious Metals, their Alloys, and Related Metals.

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

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

of the electrolyte is permissible, a generally available, rotating type of electrode may be employed. Cleaning of the electrode by sandblasting is not recommended.

7.1.2 *Cathodes*—Platinum cathodes may be either open or closed cylinders formed from sheets that are plain or perforated, or from gauze. Gauze cathodes are recommended; preferably from 50-mesh gauze woven from approximately 0.21 mm diameter wire. The top and bottom of gauze cathodes should be reinforced by doubling the gauze about 3 mm onto itself, or by the use of platinum bands or rings. The cylinder should be approximately 30 mm in diameter and 50 mm in height. The stem should be made from a platinum alloy wire such as platinum-iridium, platinum-rhodium, or platinum-ruthenium, having a diameter of approximately 1.3 mm. It should be flattened and welded the entire length of the gauze. The overall height of the cathode should be approximately 130 mm. A cathode of these dimensions will have a surface area of 135 cm^2 exclusive of the stem.

7.1.3 *Anodes*—Platinum anodes may be a spiral type when anodic deposits are not being determined, or if the deposits are small (as in the electrolytic determination of lead when it is present in concentrations below 0.2 %). Spiral anodes should be made from 1.0-mm or larger platinum wire formed into a spiral of seven turns having a height of approximately 50 mm and a diameter of 12 mm with an overall height of approximately 130 mm. A spiral anode of these dimensions will have a surface area of 9 cm^2. When both cathode and anode plates are to be determined, the anodes should be made of the same material and design as the electrode described in 7.1.2. The anode cylinder should be approximately 12 mm in diameter and 50 mm in height and the overall height of the anode should be approximately 130 mm. A gauze anode of these dimensions will have a surface area of 54 cm^2 exclusive of the stem.

7.2 *Atomic Absorption Spectrometer:*

7.2.1 Determine that the atomic absorption spectrometer is suitable for use as described in Guide E1024. The variability for the highest calibration solution should not exceed 1 %.

7.2.2 *Operating Parameters:*

Wavelength	327.5 nm
Bandpass	About 0.2 nm
Gas mixture	Air-acetylene
Flame type	Lean

7.2.3 *Instrument Response*—Adequate instrument response is obtained if the difference between the readings of the two highest of five equally spaced calibration solutions is sufficient to permit an estimation equivalent to one twentieth of the difference.

7.2.4 *Curve Linearity*—The upper limit of the usable portion of a calibration curve is normally set such that the difference between the readings of the two highest of five equally spaced calibration solutions is more than 0.7 times the difference between the lowest of the calibration solutions. Absorbance values are used in this calculation.

7.3 *Glassware,* shall be borosilicate glass unless otherwise stated.

8. Reagents

8.1 *Copper, Standard Solution A* (1 mL = 1.0 mg Cu)—Transfer 1.000 g of electrolytic copper (purity: 99.9 % min) to a 250-mL beaker, add 10 mL of HNO_3 (1 + 1) and cover. After dissolution, warm to dispel fumes, cool, transfer to a 1-L volumetric flask, dilute to volume, and mix.

8.2 *Copper, Standard Solution B* (1 mL = 0.20 mg Cu)—Using a pipet, transfer 20 mL of copper Solution A to a 100-mL volumetric flask, dilute to volume, and mix.

8.3 *Sulfuric-Nitric Acid Mixture*—While stirring, slowly add 300 mL of H_2SO_4 to 750 mL of H_2O. Cool to ambient temperature, and while stirring, add 210 mL of HNO_3.

8.4 *Potassium Cyanide Solution* (100 g/L)—Dissolve 100 g of KCN in water and dilute to 1 L. (**Warning**—The preparation, storage, and use of KCN require care and attention. Avoid inhalation of fumes and exposure of the skin to the chemical and its solutions. Work in a well-ventilated hood. Refer to the applicable section of Practices E50.)

8.5 *Sulfamic Acid Solution* (100 g/L)—Dissolve 10 g of sulfamic acid (HNH_2SO_3) in water and dilute to 100 mL. Prepare fresh daily.

9. Hazards

9.1 For precautions to be observed in this method, refer to Practices E50.

9.2 Cyanides must be disposed of with care, avoiding contact with acids that release hydrogen cyanide gas.

10. Sampling

10.1 For procedures in sampling refer to Practice E255. However, this practice does not supersede any sampling requirements specified in a specific ASTM material specification nor preclude a procedure agreed upon by the producer and consumer.

10.2 For all trace element determinations, care must be taken to limit sample exposure to contaminations, and to remove any contaminations that occur.

10.3 Wherever possible, non-metallic tools shall be used to obtain chips (millings, drillings, sawings, nibblings, and so forth) from the sample.

10.4 Except for the estimation of oxygen or hydrogen, or when analyzing standard reference materials that forbid cleaning, the chips shall be cleaned prior to weighing a portion for analysis. Immerse in HNO_3 (1 + 3), rinse in running water followed by distilled or deionized water and alcohol, and allow to air-dry. Exercise great care to prevent re-contamination of the specimen by metal tools, or from zinc in rubber stoppers, or chlorides from HCl vapor, and so forth.

10.5 In methods for the determination of impurities in copper, particular care must be taken to prevent specimen contamination by reagents or glassware.

10.6 The interior of glassware shall be cleaned immediately prior to use by a rinse in HNO_3 (1 + 3) followed by running water and by distilled or deionized water, all in an area free from HCl fumes.

10.7 Reagent acid should be taken from a bottle reserved for trace metal analysis. Extra-purity acids, intended for trace metal analysis are recommended but not required.

11. Rounding Calculated Values

11.1 Calculated values shall be rounded to the desired number of places, as directed in Practice E29, including as an option, the special rounding off to a nearest final number of five.

12. Interlaboratory Studies

12.1 This test method has been evaluated in accordance with Practice E173 unless otherwise noted in the precision and bias section.

13. Preparation of Electrodes

13.1 *Cathode*—Clean the cathode in hot nitric acid (HNO_3), (1 + 1), rinse with distilled water, rinse in two separate baths of ethanol or acetone. Dry at a low temperature (110 °C for 3 to 5 min), and cool to room temperature in a desiccator.

13.2 *Anode*—Clean in hydrochloric acid (HCl), (1 + 1), rinse with distilled water.

13.3 Weigh the cathodes to the nearest 0.1 mg and record the weight. The anode does not have to be weighed.

14. Procedure

14.1 Clean the metal that is to be analyzed in KCN solution. Rinse with water, then alcohol, and air-dry thoroughly at ambient temperature.

14.2 Transfer 5 g of the cleaned metal, weighed to the nearest 0.1 mg, to a 400-mL tall-form beaker. Add 45 mL of the H_2SO_4-HNO_3 mixture and immediately cover with a close fitting cover glass. Cool as required to prevent the reaction from becoming violent. When the reaction has subsided, heat moderately until dissolution is complete. Continue heating at approximately 90 °C until the brown fumes are expelled. Never boil. Cool slightly and carefully wash down the cover glass and sides of the beaker. Add 10 mL of sulfamic acid solution, stir, and dilute to approximately 175 to 200 mL.

14.3 With the electrolyzing current off, position the anode and the tared cathode, weighed to the nearest 0.1 mg, in the solution and add water so that the gauze is completely immersed. Cover the beaker with a split cover glass.

14.4 Electrolyze at a current density of about 0.6 A/dm^2 (Note 1). When the solution becomes colorless, wash down the cover glass, electrode stems, and sides of the beaker, add 10 mL of sulfamic acid, and continue the electrolysis until deposition is essentially complete, as indicated by failure to plate on a new surface of the cathode stem when the solution level is raised.

NOTE 1—When a current density of 0.6 A/dm^2 is used, the electrolysis requires about 16 h and is conveniently carried out overnight.

14.5 Wash the cathode with a stream of water as it is being removed from the spent solution. Immediately wash successively in two baths of water and two baths of ethanol or methanol. Reserve the electrolyte. Dry at 110 °C for 3 to 5 min, cool to ambient temperature, and weigh.

14.6 *Determination of the Residual Copper in the Electrolyte by Atomic Absorption Spectrometry:*

14.6.1 *Calibration:*

14.6.1.1 *Calibration Solutions*—Using pipets, transfer 5, 10, 15, 20, and 25 mL portions of copper Solution B to 250-mL volumetric flasks. Add 20 mL of H_2SO_4 (1 + 1), dilute to volume, and mix. These are equivalent to 0.001, 0.002, 0.003, 0.004, and 0.005 g of Cu/250 mL.

14.6.1.2 *Reference Solution*—Transfer 20 mL of H_2SO_4 to a 250-mL volumetric flask, dilute to volume, and mix.

14.6.2 *Analysis:*

14.6.2.1 *Test Solution*—If necessary evaporate the spent electrolyte from 14.5 to below 250 mL and cool. Transfer to a 250-mL volumetric flask, dilute to volume, and mix.

14.6.2.2 *Measurements*—Optimize the response of the instrument, take preliminary readings, and complete the analysis and determine the grams of copper in 250 mL by one of the procedures, graphical, ratio, or single point in accordance with Guide E1024.

15. Calculations

15.1 Calculate the weight of deposited copper as follows:

$$\text{Copper, g} = A - B \qquad (1)$$

where:

A = weight of cathode plus deposited copper, g, and

B = weight of cathode, g.

15.2 Calculate the percentage of copper as follows:

$$\text{Copper, \%} = \left(\left(C+D\right)/E\right) \times 100 \qquad (2)$$

where:

C = grams of deposited copper found in 15.1,

D = grams of copper in 250 mL of electrolyte found in 14.6.2.2, and

E = grams of sample used.

16. Precision and Bias[4]

16.1 *Precision*—Six laboratories cooperated in testing this method and obtained the data summarized in Table 1. The interlaboratory test was conducted in accordance with Practice E173 and calculated using Practice E1601 software.

TABLE 1 Statistical Information—Copper

Test Specimen	Copper Found, %	S min (E1601)	R (E1601)	% R (E1601)
1. Copper	99.959	0.008	0.023	0.02
2. Copper	99.723	0.009	0.051	0.05

16.2 *Bias*—No certified reference materials suitable for testing this test method were available when the interlaboratory testing program was conducted. The user of this test method is encouraged to employ accepted reference materials, if available, to validate the test method as implemented in a specific laboratory and to obtain estimates of uncertainty due to bias.

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[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E01-1089.

17. Keywords

17.1 copper; copper concentration

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