



Designation: E53-02 Designation: E 53 - 07

Standard Test Method for Determination of Copper in Unalloyed Copper by Gravimetry¹

This standard is issued under the fixed designation E 53; 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 Section 9 and~~ Specific precautionary statements are given in 8.4 and Section 9.*

2. Referenced Documents

2.1 *ASTM Standards:*²

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E 121 Test Methods for Chemical Analysis of Copper-Tellurium Alloys

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³

E 255 Practice for Sampling Copper and Copper Alloys for the Determination of Chemical Composition

E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry

E 1601 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 E 135.⁴

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.

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, 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*, Vol 14.02, volume information, refer to the standard's Document Summary page on the ASTM website.

³ Annual Book of ASTM Standards, Vol 03.05.

⁴ Withdrawn.

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 E 121.

7. Apparatus

7.1 Electrodes for Electroanalysis:

7.1.1 *Electrodes*—~~Platinum electrodes of the stationary type are recommended as described in~~—Recommended stationary type platinum electrodes are described in 7.1.2 and 7.1.3, but strict adherence to the exact size and shape of the electrodes is not mandatory. When agitation of the electrolyte is permissible in order to decrease the time of deposition, one of the types of rotating forms of electrodes, generally available, may be employed. The surface of the platinum electrodes should be smooth, clean, and bright to promote uniform deposition and good adherence. Sandblasting is not recommended.

7.1.2. 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 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 formed either from plain open or perforated sheets or closed cylinders formed from wire gauze, and may be either open sheets that are plain or perforated, or closed cylinders from gauze. Gauze cathodes are recommended, and shall be made recommended; preferably from 50-mesh gauze woven from wire approximately 0.21 mm (0.0085 in.) in diameter. The cathode diameter wire. The top and bottom of gauze cathodes should be stiffened reinforced by doubling the gauze for about 3 mm at the top and the bottom of the cylinder onto itself, or by reinforcing the gauze at the top and bottom with a 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 1.301.3 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 cm² exclusive of the stem.

~~7.1.3 Anodes~~—Platinum anodes may be of the 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 the amounts not over concentrations below 0.2 %). When used in analyses where both cathodic and anodic plates are to be determined, the anodes should be of wire gauze. Spiral anodes should be made from 1.00-mm 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, the mm with an overall height being of approximately 130 mm. A spiral anode of this description these dimensions will have a surface area of 9 cm². Platinum gauze. When both cathode and anode plates are to be determined, the anodes should be made of the same material and of the same general design as platinum gauze cathodes; 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 cm². Both areas are exclusive of the stem. exclusive of the stem.

7.2 Atomic Absorption Spectrophotometer—Atomic Absorption Spectrometer:

7.2.1 Determine that the atomic absorption spectrophotometerspectrometer is suitable for use as described in Guide E 1024. The variability for the highest calibration solution should not exceed 1 %.

7.2.2 Operating Parameters:

Wavelength	3275 Å
Wavelength	327.5 nm
Bandpass	About 2 Å
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 ~~useable~~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 HNO₃ (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 H₂SO₄ to 750 mL of H₂O. Cool to ambient temperature, and while stirring, add 210 mL of HNO₃.

8.4 *Potassium Cyanide Solution (100 g/L)*—Dissolve 100 g of KCN in water and dilute to 1 L. (**Warning**—The preparation,