



Designation: E 53 – 02

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 8.4.

2. Referenced Documents

2.1 ASTM Standards:

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications²

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 Alloys³

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials³

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 Composition³

E 1024 Guide for Chemical Analysis of Metals and Metal Bearing Ores by Flame Atomic Absorption Spectrophotometry⁴

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method⁴

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology **E 135**.

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 codeposit. 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 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.

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 *Annual Book of ASTM Standards*, Vol 14.02.

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

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

7.1.2 *Cathodes*—Platinum cathodes may be formed either from plain or perforated sheets or from wire gauze, and may be either open or closed cylinders. Gauze cathodes are recommended, and shall be made preferably from 50-mesh gauze woven from wire approximately 0.21 mm (0.0085 in.) in diameter. The cathode should be stiffened by doubling the gauze for about 3 mm at the top and the bottom of the cylinder or by reinforcing the gauze at the top and bottom with a platinum band or ring. 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 1.30 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 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 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 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 overall height being approximately 130 mm. A spiral anode of this description will have a surface area of 9 cm². Platinum gauze anodes should be made of the same material and of the same general design as platinum gauze cathodes. 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.

7.2 Atomic Absorption Spectrophotometer:

7.2.1 Determine that the atomic absorption spectrophotometer 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:

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|-------------|---------------|
| Wavelength | 3275 Å |
| Bandpass | About 2 Å |
| 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 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, 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 E 50.)

8.5 *Sulfamic Acid Solution (100 g/L)*—Dissolve 10 g of sulfamic acid (H(NH₂)SO₃) 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 E 50.

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 E 255. 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, etc) 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 HNO₃(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, etc.

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 HNO₃(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.