



Standard Test Methods for Chemical Analysis of Copper-Chromium Alloys¹

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1. Scope

1.1 These test methods cover procedures for the chemical analysis of chromium-copper alloys having chemical compositions within the following limits:

| | |
|-------------|-------------|
| Copper, % | 98 and over |
| Chromium, % | 0.6 to 1.2 |
| Silicon, % | 0 to 0.10 |
| Iron, % | 0 to 0.10 |

1.2 The analytical procedures appear in the following order:

| | Sections |
|--|----------|
| Copper by the Electrolytic Method | 8-11 |
| Chromium by the Dichromate (Potentiometric) Method | 12-15 |
| Silicon by the Perchloric Acid Dehydration Method | 16 |

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 Precautions for Chemical Analysis of Metals
- E 54 Test Methods for Chemical Analysis of Special Brasses and Bronzes
- E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition
- E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals
- E 62 Test Methods for Chemical Analysis of Copper and Copper Alloys (Photometric Methods)

3. Significance and Use

3.1 These test methods for the chemical analysis of metals and alloys are primarily intended to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts

¹ These test methods are under the jurisdiction of ASTM Committee E01 on Analytical Chemistry for Metals, Ores, and Related Materials and are 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* volume information, refer to the standard's Document Summary page on the ASTM website.

capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus, Reagents, and Photometric Practice

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 used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

4.2 Photometric practice prescribed in these test methods shall conform to Practice E 60.

5. Hazards

5.1 For hazards to be observed in these test methods, reference shall be made to Practices E 50.

6. Sampling

6.1 Wrought products shall be sampled in accordance with Practice E 55.

7. Rounding Calculated Values

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

COPPER BY THE ELECTROLYTIC TEST METHOD

8. Apparatus

8.1 *Electrodes for Electroanalysis*—Apparatus No. 9.

9. Reagents

9.1 *Sulfuric-Nitric Acid Mixture*—Add slowly, while stirring, 500 mL of H₂SO₄ to 1700 mL of water. Cool, and add 300 mL of HNO₃.

9.2 *Hydrogen Peroxide* (3 %).

10. Procedure

10.1 Transfer 2.0000 g of the sample to a 250-mL electrolysis beaker, cover, and dissolve in 60 mL of the H₂SO₄-HNO₃ mixture. Allow the beaker to remain on a steam bath until

solution of the sample is complete, add a few drops of HF and carefully evaporate to fumes to decompose the sample completely. Cool, dilute with water, add 3 mL of HNO₃, and warm to dissolve all soluble salts. Wash down the cover glass and sides of the beaker and dilute to about 150 mL with water.

10.2 Insert the electrodes into the solution with a pair of split watch glasses, and electrolyze overnight at a current density of 0.5 A/dm² or for 2½ h at a current density of 4 A/dm² (Note 1). The more rapid procedure requires the use of gauze cathodes. When the solution is colorless, wash down the cover glasses, electrodes, and the sides of the beaker, and continue the electrolysis until deposition of the copper is complete, as indicated by failure to plate on a new surface when the level of the solution is raised (Note 2).

NOTE 1—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.

NOTE 2—If the electrolyte is not to be used for subsequent determinations, remove a few drops of the solution, place on a porcelain spot plate, and treat with saturated H₂S solution. Continue electrolysis until no CuS precipitate is observed.

10.3 When deposition of the copper is complete, with the current still on, lower the beaker slowly, while washing the cathode with water. Remove the cathode, rinse it in water, and dip it in two successive baths of ethanol or methanol. Dry in an oven at 110°C for 3 to 5 min, cool, and weigh the deposit as metallic copper.

10.4 *Calculation*—Calculate the percentage of copper as follows:

$$\text{Copper, \%} = \frac{A}{B} \times 100 \quad (1)$$

where:

A = grams of copper, and
B = grams of sample used.

11. Precision and Bias

11.1 This test method was originally approved for publication before the inclusion of precision and bias statements within standards was mandated. The original interlaboratory test data for this test method are no longer available. The user is cautioned to verify by the use of reference materials, if available, that the precision and bias of this test method are adequate for the contemplated use.

CHROMIUM BY THE DICHROMATE (POTENTIOMETRIC) TEST METHOD

12. Apparatus

12.1 *Apparatus for Potentiometric Oxidation and Reduction Titrations*—Apparatus No. 3B.

13. Reagents

13.1 *Ammonium Persulfate Solution* (100 g/L)—Dissolve 100 g of (NH₄)₂S₂O₈ in water and dilute to 1 L. Prepare fresh before use.

13.2 *Ferrous Ammonium Sulfate Standard Solution* (0.06 N)—Dissolve 23 g of Fe(NH₄)₂(SO₄)₂·6H₂O in 800 mL of water containing 100 mL of H₂SO₄ (1 + 2), cool, and dilute to 1 L in a volumetric flask.

13.2.1 Immediately prior to use, determine the dichromate equivalent of the solution as follows: Dilute 25 mL of the ferrous ammonium sulfate solution to 150 mL in a 400-mL beaker. Titrate potentiometrically with the K₂Cr₂O₇ solution (1 mL = 0.001 g Cr) to the first large deflection of the needle. Record the millilitres of K₂Cr₂O₇ solution required for the titration.

13.3 *Potassium Dichromate Standard Solution* (1 mL = 0.001 g Cr)—Grind 3.0 g of K₂Cr₂O₇ to a fine powder and dry at 150°C to constant weight. Dissolve 2.829 g of the dried powder and 5 g of Na₂CO₃ in water and dilute to 1 L in a volumetric flask.

13.4 *Silver Nitrate Solution* (2.5 g/L)—Dissolve 2.5 g of AgNO₃ in water and dilute to 1 L.

14. Procedure

14.1 Transfer 2.00 g of the sample, in the form of wire, to a 600-mL beaker and cover. Add 15 mL of HNO₃ and 5 drops of HF and allow to stand. When dissolution is complete, wash down the cover glass and the sides of the beaker and add 20 mL of H₂SO₄. Evaporate to dense white fumes and fume gently until oxides of chromium have dissolved.

14.2 Cool, add 350 mL of water, and heat until the solution is clear. Add 15 mL of AgNO₃ solution and 40 mL of (NH₄)₂S₂O₈ solution. Add a few boiling granules, cover, heat to boiling, and boil moderately for 8 to 9 min. Add 5 mL of HCl (1 + 3), continue boiling for 5 min, and cool to 20°C.

14.3 Place the beaker containing the solution in the potentiometric titration apparatus stand, insert the electrodes, and, while stirring the solution, add 25 mL of freshly standardized ferrous ammonium sulfate solution. Backtitrate potentiometrically with K₂Cr₂O₇ solution to the first large deflection of the needle.

14.4 *Calculation*—Calculate the percentage of chromium as follows:

$$\text{Chromium, \%} = [(A - B) \times 0.001]/C \times 100 \quad (2)$$

where:

A = millilitres of K₂Cr₂O₇ solution required to titrate 25 mL of ferrous ammonium sulfate solution,
B = millilitres of K₂Cr₂O₇ solution (1 mL = 0.001 g Cr) required to titrate the excess ferrous ammonium sulfate solution, and
C = grams of sample used.

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SILICON BY THE PERCHLORIC ACID DEHYDRATION TEST METHOD

16. Procedure

16.1 Determine silicon in accordance with Sections 18 and 19 of Test Methods E 54, except for the following modifica-

tion: If the precipitate of silica obtained in accordance with 19.3 is badly discolored, return the paper and precipitate to the casserole and boil for 10 to 15 min with 20 mL of HCl. Add 5 mL of HNO₃ and 25 mL of HClO₄, and continue in accordance with 21.3 to 21.5 of Test Methods E 54.

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