



# Standard Test Methods for Chemical Analysis of Copper-Tellurium Alloys<sup>1</sup>

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## 1. Scope

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

Copper, <sup>A</sup> %	98 and over
Tellurium, %	0.04 to 0.6

<sup>A</sup>Includes silver.

1.2 The analytical procedures appear in the following order:

Copper by the Electrolytic Method	Sections 8 to 12
Tellurium by the Dichromate (Volumetric) Method	13 to 17

## 2. Referenced Documents

### 2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications<sup>2</sup>
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals<sup>3</sup>
- E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition<sup>3</sup>

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

## 4. Apparatus and Reagents

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.

## 5. Precautions

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

## 6. Sampling

6.1 Sampling shall conform to 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. Summary of Test Method

8.1 Copper is deposited electrolytically on a platinum cathode. The addition of manganese nitrate to the electrolyte prevents the deposition of tellurium.

## 9. Apparatus

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

## 10. Reagents

10.1 *Manganese Nitrate Solution* (20 g/L)—Dilute 4 mL of the commercially available  $\text{Mn}(\text{NO}_3)_2$  solution (50 %) to 100 mL with water.

10.2 *Potassium Permanganate Solution* (20 g/L)—Dissolve 2 g of  $\text{KMnO}_4$  in water and dilute to 100 mL.

10.3 *Sulfuric-Nitric Acid Mixture*—Add slowly, while stirring, 300 mL of  $\text{H}_2\text{SO}_4$  to 750 mL of water. Cool and add 210 mL of  $\text{HNO}_3$ .

## 11. Procedure

11.1 Transfer 2.00 g of the sample to a 300-mL electrolysis beaker. Add 30 mL of  $\text{H}_2\text{SO}_4$ - $\text{HNO}_3$  mixture, cover, and allow to stand a few minutes until reaction has nearly ceased. Heat at 80 to 90°C until dissolution is complete and brown fumes have been expelled. Wash down the cover glass and the sides of the beaker and dilute to about 200 mL. Add  $\text{KMnO}_4$  solution dropwise until a faint, permanent pink color is produced and then add 5 mL of  $\text{Mn}(\text{NO}_3)_2$  solution.

11.2 Insert the electrodes, cover the solution with a pair of split watch glasses, and electrolyze at a current density of about 0.6 A/dm<sup>2</sup> for about 16 h or at a current density of 4 A/dm<sup>2</sup> for

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee E-1 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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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 03.05.

about 2.5 h. Wash down the cover glasses, the sides of the beaker, and the electrode stems, and continue electrolysis for about 15 min. If no copper plates on the newly exposed cathode surface, copper deposition may be considered completed.

11.3 Quickly withdraw the cathode from the electrolyte while directing a gentle stream of water from a wash bottle over its surface. Rinse the cathode in a water bath and then dip in two successive baths of ethanol or acetone. Dry in an oven at 110°C for 3 to 5 min, cool, and weigh.

11.4 Strip the copper deposit from the cathode by immersion in HNO<sub>3</sub> (1+1) for a few minutes. When dissolution of the copper is complete, rinse the cathode in a water bath and then dip in two successive baths of ethanol or acetone. Dry in an oven at 110°C for 3 to 5 min, cool, and reweigh.

## 12. Calculation

12.1 Calculate the percentage of copper as follows:

$$\text{Copper, \%} = [(A - B)/C] \times 100 \quad (1)$$

where:

*A* = weight of copper plus cathode,

*B* = weight of cathode, and

*C* = weight of sample used.

## 13. Precision and Bias

13.1 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.

### TELLURIUM BY THE DICHROMATE (VOLUMETRIC) TEST METHOD

## 14. Summary of Test Method

14.1 In a nitrous acid-free solution, tellurous ions are gradually oxidized to the hexavalent state by an excess of standard potassium dichromate. On completion of the reaction the excess dichromate is titrated potentiometrically with standard ferrous ammonium sulfate.

## 15. Apparatus

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

## 16. Reagents

16.1 *Ferrous Ammonium Sulfate Standard Solution* (0.05 *N*)—See Reagent No. 5.

16.1.1 Immediately prior to use, determine the dichromate equivalent of the solution as follows: Pipet 15 mL of 0.05 *N* K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution into a 400-mL beaker. Add 200 mL of water and 40 mL of H<sub>2</sub>SO<sub>4</sub> (1+1). Cool to room temperature. Titrate with the ferrous ammonium sulfate solution to a potentiometric end point. Record the millilitres of ferrous ammonium sulfate solution required for the titration.

16.2 *Potassium Dichromate Standard Solution* (0.05 *N*)—See Reagent No. 10.

## 17. Procedure

17.1 Transfer 5.00 g of the sample to a 400-mL beaker. Add 40 mL of HNO<sub>3</sub> (1+1), cover, and allow to stand a few minutes until reaction has nearly ceased. Heat gradually to boiling and boil gently until brown fumes have been expelled. Cool the solution, add about 1 g of urea, dilute to about 200 mL, and stir. Add 40 mL of H<sub>2</sub>SO<sub>4</sub> (1+1), stir, and cool to room temperature in a water bath.

17.2 Pipet 15 mL of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution (0.05 *N*) into the beaker, stir well, and allow to stand for 15 min. Titrate the excess K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> with freshly standardized ferrous ammonium sulfate solution (0.05 *N*) to a potentiometric end point.

## 18. Calculation

18.1 Calculate the percentage of tellurium as follows:

$$\text{Tellurium, \%} = [(15 - (15B/A)) \times 0.00319]/C \times 100 \quad (2)$$

where:

*A* = millilitres of ferrous ammonium sulfate solution required to titrate 15 mL of the 0.05 *N* K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution,

*B* = millilitres of ferrous ammonium sulfate solution required to titrate the excess 0.05 *N* K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution, and


*C* = grams of sample used.

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