



Standard Test Methods for Chemical Analysis of Copper-Beryllium Alloys¹

This standard is issued under the fixed designation E 106; 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.

1. Scope

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

Element	Concentration Range, %
Copper	97 to 98
Beryllium	0.4 to 2.05
Nickel	0.0 to 0.30
Cobalt	0.0 to 0.3
Iron	0.0 to 0.30

1.2 The analytical procedures appear in the following order:

	Sections
Copper by the Electrolytic Method	8-12
Beryllium:	
Phosphate Gravimetric Method	13-19
Aluminon (Photometric) Method	20-27
Nickel by the Dimethylglyoxime (Photometric) Method	28-36
Cobalt by the Nitroso-R-Salt (Photometric) Method	37-44
Iron by the Thiocyanate (Photometric) Method	45-52

2. Referenced Documents

2.1 ASTM Standards:²

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specification
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals
- 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 76 Test Methods for Chemical Analysis of Nickel-Copper Alloys

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.

Current edition approved June 1, 2004. Published July 2004. Originally approved in 1954. Last previous edition approved in 1996 as E 106 – 83 (1996).

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.

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

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, 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 methods shall conform to Practice E 60.

5. Safety Precautions

5.1 For precautions to be observed in these methods, reference shall be made to Practices E 50. Both beryllium metal and its compounds may be toxic. Care should be exercised to prevent contact of beryllium-containing materials with the skin. The inhalation of any beryllium-containing substance, either as a volatile compound or as finely divided powder, should be especially avoided. Beryllium-containing residues (especially ignited oxide) should be carefully disposed of.

6. Sampling

6.1 Sampling shall conform to Practice E 55.

7. Rounding Off Calculated Values

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

³ Withdrawn.



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, 300 mL of H₂SO₄ to 750 mL of water. Cool and add 210 mL of HNO₃.

10. Procedure

10.1 Transfer 5.00 g of sample to a 300-mL electrolysis beaker. Add 42 mL of the H₂SO₄-HNO₃ 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 175 mL (enough to submerge the cathode when it is inserted).

10.2 Insert the electrodes, cover the solution with a pair of split watch glasses, and electrolyze at a current density of about 0.6 A/dm² for about 16 h. Wash down the cover glasses, sides of beaker, and 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.

10.3 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. Reserve the spent electrolyte.

where:

A = grams of copper, and

B = grams of sample used.

10.4 *Reserved Electrolyte*—Evaporate the spent electrolyte to dense white fumes and fume for about 5 min to dehydrate silicic acid. Cool, add about 50 mL of water, and heat until all salts are in solution. Filter through a small, medium-texture paper, catching the filtrate in a 250-mL volumetric flask. Wash the beaker and paper thoroughly with hot H₂SO₄ (1 + 99), combining the washings with the filtrate. Cool the solution in the volumetric flask, dilute to the mark, and mix. Reserve for the determinations of beryllium, nickel, cobalt, and iron as described in Sections 17, 34, 43, and 51 respectively. If the filtrate is not to be used for the gravimetric determination of beryllium, the removal of silica is not necessary and the electrolyte may be diluted to volume directly.

11. Calculation

11.1 Calculate the percentage of copper as follows:

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

12. Precision and Bias

12.1 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.

BERYLLIUM BY THE PHOSPHATE GRAVIMETRIC TEST METHOD

13. Scope

13.1 This test method covers the determination of beryllium in concentrations from 0.1 to 3.0 %.

14. Summary of Test Method

14.1 Interfering elements are complexed with (ethylenedinitrilo) tetraacetate solution. Beryllium is precipitated as the phosphate, which is filtered, ignited, and weighed as beryllium pyrophosphate.

15. Interferences

15.1 The elements ordinarily present in beryllium-copper alloys do not interfere if their concentrations are under the maximum limits shown in 1.1.

16. Reagents

16.1 *Ammonium Acetate Solution* (500 g/L)—Dissolve 500 g of ammonium acetate in water, and dilute to 1 L.

16.2 *Ammonium Acetate Wash Solution*—Dilute 5 mL of the ammonium acetate solution to 1 L, and adjust the pH to 5.2 ± 0.05 with acetic acid.

NOTE 1—Use a pH meter for all pH adjustments.

16.3 *Ammonium Dihydrogen Phosphate* (100 g/L)—Dissolve 100 g of ammonium dihydrogen phosphate (NH₄H₂PO₄) in water and dilute to 1 L.

16.4 *Ammonium (Ethylenedinitrilo) Tetraacetate Solution* (28 g/L)—To 2.5 g of (ethylenedinitrilo) tetraacetic acid add 30 mL of water and a drop of methyl red solution. Neutralize with NH₄OH (1 + 1), and warm gently to dissolve the last traces of solid. Cool and dilute to 100 mL.

16.5 *Methyl Red Indicator Solution* (0.5 g/L ethanol)—Dissolve 0.05 g of methyl red in 100 mL of ethanol.

17. Procedure

17.1 Using a pipet, transfer 50 mL of the electrolyte reserved in 10.4 to a 400-mL beaker. Add 3 drops of HF and 10 mL of H₂SO₄(1 + 2), and evaporate to fumes. Cool to room temperature and add 100 mL of water. Heat to dissolve soluble salts and again cool to room temperature.

17.2 Add 10 mL of ammonium (ethylenedinitrilo) tetraacetate solution, and adjust the pH to 2.0 ± 0.05 (see Note 1) with NH₄OH (1 + 1). Boil 1 min and cool to room temperature. Add 10 mL of ammonium dihydrogen phosphate solution and adjust the pH to 5.2 ± 0.05 with ammonium acetate solution.

17.3 Heat to boiling cautiously to prevent bumping, and then maintain just below the boiling point until the precipitate becomes granular. Remove from the source of heat and allow to stand at least 12 h.

17.4 Filter using an 11-cm fine paper and wash six times with ammonium acetate wash solution. Discard the filtrate.

Dissolve the precipitate with 100 mL of hot HCl (1 + 4), collecting the solution in the original beaker.

17.5 Add 2 mL of ammonium (ethylenedinitrilo) tetraacetate solution, and adjust the pH to 2.0 ± 0.05 with NH_4OH (1 + 1). Cool, add 2 mL of ammonium dihydrogen phosphate solution, and adjust the pH to 5.2 ± 0.05 with ammonium acetate solution. Proceed as directed in 17.3.

17.6 Filter using an 11-cm fine paper and wash six times with ammonium acetate wash solution. Transfer the paper to a weighed platinum crucible. Place the crucible in a muffle furnace, and dry and char the paper by gradually increasing the temperature to 500°C . When all the carbon has been removed, raise the temperature to 1000°C and maintain at this temperature for 4 h. Cool in a desiccator and weigh.

18. Calculation

18.1 Calculate the percentage of beryllium as follows:

$$\text{Beryllium, \%} = (A \times 0.0939/B) \times 100 \quad (2)$$

where:

A = grams of beryllium pyrophosphate, and
B = grams of sample used

19. Precision and Bias

19.1 *Precision*—Eight laboratories cooperated in testing this method and obtained the data summarized in Table 1.

19.2 *Bias*—No certified reference materials suitable for testing this test method were available when the interlaboratory testing program was conducted. The user of this standard is encouraged to employ accepted reference materials, if available, to determine the accuracy of this test method as applied in a specific laboratory.

TABLE 1 Statistical Information

Test Specimen	Beryllium Found, %	Repeatability (R ₁ , E 173)	Reproducibility (R ₂ , E 173)
(1) Beryllium copper, B-7	1.744	0.026	0.042
(2) Beryllium copper, C-7	0.460	0.020	0.046

BERYLLIUM BY THE ALUMINON (PHOTOMETRIC) TEST METHOD

20. Principle of Test Method

20.1 In a properly buffered solution, ammonium aurin tricarboxylate (aluminon) forms a red lake with beryllium. The addition of ethylenediamine tetraacetic acid (complexone) prevents the interference of aluminum, iron, copper, and similar elements. Photometric measurement is made at approximately 515 nm.

21. Concentration Range

21.1 The recommended concentration range is from 0.004 to 0.09 mg of beryllium in 100 mL of solution, using a cell depth⁴ of 2 cm.

22. Stability of Color

22.1 The intensity of the color of the beryllium lake increases slowly on standing. Therefore, a uniform standing time must be adhered to.

23. Interfering Elements

23.1 Provision is made in the procedure for preventing, or compensating for, interference from metals present in amounts not exceeding the maximum limits given in 1.1

24. Reagents

24.1 *Aluminon-Buffer Composite Solution*—Add 500 g of ammonium acetate to 1 L of water in a 2-L beaker. Add 80 mL of glacial acetic acid and stir until dissolution is complete. Filter if necessary. Dissolve 1.000 g of a suitable grade of aluminon⁵ (aurin tricarboxylic acid-ammonium salt) in 50 mL of water and add to the buffer solution. Dissolve 3 g of benzoic acid in 20 mL of methanol and add to the buffer solution while stirring. Dilute the mixture to 2 L. Add 10 g of gelatin⁶ to 250 mL of water in a 400-mL beaker. Place the beaker in a boiling water bath and allow to remain, with frequent stirring, until the gelatin has dissolved completely. Pour the warm gelatin solution into 500 mL of distilled water, while stirring. Cool to room temperature, dilute to 1 L, and mix. Transfer the aluminon and gelatin solutions to a 4-L chemically resistant glass-stoppered bottle, mix well, and store in a cool, dark place.

24.2 *Complexone Solution*—See 16.4.

24.3 *Copper Chloride Solution* (1 mL = 2 mg Cu)—Dissolve 0.54 g of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to 100 mL in a volumetric flask.

24.4 *Standard Beryllium Solution* (1 mL = 1.0 mg Be)—Dissolve 9.82 g of $\text{BeSO}_4 \cdot 4\text{H}_2\text{O}$ in 100 mL of HCl (1 + 3). Filter, if necessary, and dilute to 500 mL. Standardize as follows: Transfer 25 mL of the solution to a 250-mL beaker and proceed in accordance with Section 17.2-17.6 and 18.1.

24.5 *Standard Beryllium Solution* (1 mL = 0.01 mg Be)—Transfer 10 mL of the above solution to a 1-L volumetric flask, add 10 mL of HCl, dilute to the mark, and mix.

⁴ This procedure has been written for a cell having a 2-cm light path. Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagents used.

⁵ Certain commercially available grades of aluminon have been found to be unsatisfactory for this purpose. It may be necessary to prepare a small portion of the composite reagent before use. The currently available (1954) product from Eastman Kodak appears to be satisfactory.

⁶ Knox gelatin has been found satisfactory for this purpose.