Designation: E2824 – 11

Standard Test Method for Determination of Beryllium in Copper-Beryllium Alloys by Phosphate Gravimetric Method[1]

1. Scope

1.1 This test method describes the determination of beryllium in copper-beryllium alloys in percentages from 0.1 % to 3.0 % by the phosphate gravimetric method.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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 hazard statements are given in Section 9.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials

E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials

E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)[3]

E255 Practice for Sampling Copper and Copper Alloys for the Determination of Chemical Composition

E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method

3. Terminology

3.1 For definitions of terms used in this method, refer to Terminology E135.

4. Summary of Test Method

4.1 Beryllium is precipitated as the phosphate, which is filtered, ignited, and weighed as beryllium pyrophosphate. Interfering elements, if present, may be complexed with (ethylenedinitrilo) tetraacetate solution.

5. Significance and Use

5.1 This test method for the chemical analysis of metals and alloys is 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.

6. Interferences

6.1 The elements ordinarily present in beryllium-copper alloys do not interfere.

7. Apparatus

7.1 *Electrodes for Electroanalysis*—Recommended stationary type platinum electrodes are described in 7.1.1 and 7.1.2. The surface of the platinum electrode 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.

7.1.1 *Cathodes*—Platinum cathodes may be either open or closed cylinders formed from sheets that are plain or perforated, or from gauze. Gauze cathodes are recommended; preferably from 50-mesh gauze woven from approximately 0.21 mm diameter wire. The top and bottom of gauze cathodes should be reinforced by doubling the gauze about 3 mm onto itself, or by the 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

---

[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.

Current edition approved May 1, 2011. Published July 2011. DOI: 10.1520/E2824-11.

[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.

[3] The last approved version of this historical standard is referenced on www.astm.org.

such as platinum-iridium, platinum-rhodium, or platinum-ruthenium, having a diameter of approximately 1.3 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 cm^2 exclusive of the stem.

7.1.2 *Anodes*—Platinum anodes may be 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 concentrations below 0.2 %). Spiral anodes should be made from 1.0 mm or larger platinum wire formed into a spiral of seven turns having a height of approximately 130 mm. A spiral anode of these dimensions will have a surface area of 9 cm^2. When both cathode and anode plates are to be determined, the anode should be made of the same material and design as the electrode described in 7.1.1. 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 cm^2 exclusive of the stem.

7.1.3 Gauze cathodes are recommended where rapid electrolysis is used.

8. Reagents

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

8.2 *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.

8.3 *Ammonium Dihydrogen Phosphate* (100 g/L)—Dissolve 100 g of ammonium dihydrogen phosphate ($NH_4H_2PO_4$) in water and dilute to 1 L.

8.4 *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 NH_4OH (1 + 1), and warm gently to dissolve the last traces of solid. Cool and dilute to 100 mL.

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

8.6 *Sulfuric-Nitric Acid Mixture*—Add slowly, while stirring in a cold water bath, 300 mL of H_2SO_4 to 750 mL of water. Cool and add 210 mL of HNO_3.

9. Hazards

9.1 For precautions to be observed in this method, reference shall be made to Practices E50. 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.

10. Sampling

10.1 Sampling shall conform to Practice E255. However, this method does not supersede any sampling requirements specified in a specific ASTM material specification.

11. Rounding Calculated Values

11.1 Calculated values shall be rounded to the desired number of places as directed in Practice E29.

12. Preparation of Apparatus

12.1 *Cathode*—Clean the cathode in hot HNO_3, (1 + 1), rinse with distilled water, rinse in two separate baths of ethanol or acetone. Dry at a low temperature, (110 °C for 3 to 5 min), and cool to room temperature in a desiccator.

12.2 *Anode*—Clean in HCl, (1 + 1), rinse with distilled water.

12.3 Weigh the cathode to the nearest 0.1 mg and record the weight. The anode does not have to be weighed.

13. Procedure

13.1 Transfer 5.00 g of sample to a 300-mL electrolysis beaker. Add 42 mL of the H_2SO_4-HNO_3 mixture, cover, and allow to stand a few minutes until the reaction has nearly ceased. Heat at 80 °C 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).

13.2 Insert the electrodes, cover the solution with a pair of split watch glasses, and electrolyze at a current density of 0.6 A/dm^2 for about 16 h. Wash down the cover glasses, sides of the 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.

13.3 Quickly withdraw the cathode from the electrolyte while directing a gentle stream of water from a wash bottle over its surface.

13.4 Evaporate the spent electrolyte to dense white fumes and fume for about 5 min to dehydrate the 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 H_2SO_4 (1 + 99), combining the washings with the filtrate. Cool the solution in the volumetric flask, dilute to the mark, and mix.

13.5 Using a pipet, transfer 50 mL of the solution in 13.4 to a 400-mL beaker. Add 3 drops of HF and 10 mL of H_2SO_4 (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.

13.6 Add 10 mL of ammonium (ethylenedinitrilo) tetraacetate solution, and adjust the pH to 2.0 ± 0.05 (see Note 1 for all pH adjustments) with NH_4OH (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.

13.7 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.

13.8 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.

13.9 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 13.7.

13.10 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.

14. Calculation

14.1 Calculate the percentage of beryllium as follows:

$$\text{Beryllium, } \% = (A \times 0.0939/B) \times 100 \quad (1)$$

where:

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

15. Precision and Bias

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

15.2 *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.

15.3 This test method was evaluated in accordance with Practice E173 (discontinued 1997). Practice E173 has been replaced by Practice E1601. The Reproducibility R_2 of Practice E173 corresponds to the Reproducibility Index R of Practice E1601. The Repeatability R_1 of Practice E173 corresponds to the Repeatability Index r of Practice E1601.

16. Keywords

16.1 beryllium; copper-beryllium alloys; gravimetric

TABLE 1 Statistical Information

Test Specimen	Beryllium Found, %	Repeatability (R_1, E173)	Reproducibility (R_2, E173)
(1) Beryllium copper, B-7	1.744	0.026	0.042
(2) Beryllium copper, C-7	0.460	0.020	0.046