



Designation: **E2824 – 18** ~~E2824 – 18a~~

## Standard Test Method for Determination of Beryllium in Copper-Beryllium Alloys by Phosphate Gravimetric Method ~~Gravimetry~~<sup>1</sup>

This standard is issued under the fixed designation E2824; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 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 ~~gravimetry~~.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 9.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

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E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals (Withdrawn 1998)<sup>3</sup>

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.

<sup>1</sup> 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, Precious Metals, their Alloys, and Related Metals.

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<sup>2</sup> 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.

<sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.

## 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, 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 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<sup>2</sup> 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~~ 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<sup>2</sup>. 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<sup>2</sup> 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. Use a pH meter for all pH adjustments.

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<sub>4</sub>H<sub>2</sub>PO<sub>4</sub>) 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<sub>4</sub>OH (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<sub>2</sub>SO<sub>4</sub> to 750 mL of water. Cool and add 210 mL of HNO<sub>3</sub>.

## 9. Hazards

9.1 For precautions to be observed in this ~~test method, refer method, reference shall be made to Practices E50~~. Both beryllium metal and its compounds may be toxic. ~~Exercise care~~ 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 ~~Rounding of test results obtained using this test method shall be performed in accordance with Practice~~ Calculated values shall be rounded to the desired number E29, Rounding Method, unless an alternative of places as directed in Practice E29 rounding method is specified by the customer or applicable material specification.

## 12. Preparation of Apparatus

12.1 *Cathode*—Clean the cathode in hot HNO<sub>3</sub>, (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.