



Designation: E2824 – 23

Standard Test Method for Determination of Beryllium in Copper-Beryllium Alloys by Phosphate Gravimetry¹

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 (ϵ) 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 phosphate 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:*²
- D1193 Specification for Reagent Water
 - 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Methods for Chemical Analysis of Metals (Withdrawn 1997)³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 EDTA 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

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, Precious Metals, 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.

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

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 cm² 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 cm². 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 cm² 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 (NH₄H₂PO₄) in water and dilute to 1 L.

8.4 (Ethylenedinitrilo) tetraacetic acid disodium salt (EDTA) (28 g/L)—To 2.5 g of EDTA 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.

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

8.7 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on

Analytical Reagents of the American Chemical Society where such specifications are available.⁴ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.8 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type I or Type II of Specification **D1193**. Type III or Type IV may be used if they effect no measurable change in the blank or sample.

9. Hazards

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

9.2 Processing beryllium and beryllium-containing materials poses a health risk if safe-handling practices are not followed. Inhalation of airborne beryllium may cause a serious lung disorder in some individuals. Occupational safety and health regulatory agencies have set mandatory limits on occupational respiratory exposures. Read and follow the guidance in the SDS before working with these materials.

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 as directed in Practice **E29**, Rounding Method, unless an alternative rounding method is specified by the customer or applicable material specification.

12. Preparation of Apparatus

12.1 Cathode—Clean the cathode in hot HNO₃, (1 + 1), rinse with distilled water, rinse in two separate baths of ethanol or acetone. Dry at a low temperature, (110 °C for 3 min 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 H₂SO₄-HNO₃ 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

⁴ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.