



Designation: E439 – 23

Standard Test Methods for Chemical Analysis of Beryllium¹

This standard is issued under the fixed designation E439; 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 reappraisal. A superscript epsilon (ε) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 These test methods cover the chemical analysis of beryllium having chemical compositions within the following limits:

Element	Range, %
Aluminum	0.05 to 0.30
Beryllium	97.5 to 100
Beryllium Oxide	0.3 to 3
Carbon	0.05 to 0.30
Copper	0.005 to 0.10
Chromium	0.005 to 0.10
Iron	0.05 to 0.30
Magnesium	0.02 to 0.15
Nickel	0.005 to 0.10
Silicon	0.02 to 0.15

1.2 The test methods in this standard are contained in the sections as follows.

	Sections
Chromium by the Diphenylcarbazide Spectrophotometric Test Method	
[0.004 % to 0.04 %]	10 – 19
Iron by the 1,10-Phenanthroline Spectrophotometric Test Method	
[0.05 % to 0.25 %]	20 – 29
Manganese by the Periodate Spectrophotometric Test Method	
[0.008 % to 0.04 %]	30 – 39
Nickel by the Dimethylglyoxime Spectrophotometric Test Method	
[0.001 % to 0.04 %]	40 – 49

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *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.*

1.5 *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.*

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, Precious Metals, their Alloys, and Related Metals.

Current edition approved July 1, 2023. Published July 2023. Originally published in 1971. Last previous edition approved in 2017 as E439 – 17. DOI: 10.1520/E0439-23.

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
- E55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition
- E60 Practice for Analysis of Metals, Ores, and Related Materials by Spectrophotometry
- E88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition
- 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
- 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 test method, refer to Terminology E135.

4. Significance and Use

4.1 These test methods for the chemical analysis of beryllium metal are primarily intended as referee methods 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.

5. Apparatus, Reagents, and Spectrophotometric Practice

5.1 Apparatus and reagents required for each determination are listed in separate sections preceding the procedure unless

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.

otherwise specified. The apparatus and standard solutions shall conform to the requirements prescribed in Practices E50. Spectrophotometers shall conform to the requirements prescribed in Practice E60.

5.2 Spectrophotometric practice prescribed in these test methods shall conform to Practice E60.

5.3 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all reagents shall 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.

6. Hazards

6.1 For precautions to be observed in these test methods, refer to Practices E50.

6.2 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.

7. Sampling

7.1 Wrought products shall be sampled in accordance with Practice E55. Cast products shall be sampled in accordance with Practice E88. However, these practices do not supersede any sampling requirements specified in a specific ASTM material specification.

8. Rounding Calculated Values

8.1 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.

9. Interlaboratory Studies

9.1 These test methods have been evaluated in accordance with Practice E173, unless otherwise noted in the precision section.

CHROMIUM BY THE DIPHENYLCARBAZIDE SPECTROPHOTOMETRIC TEST METHOD

10. Scope

10.1 This test method covers the determination of chromium from 0.004 % to 0.04 %.

11. Summary of Test Method

11.1 Chromium is oxidized by peroxydisulfate in the presence of silver nitrate, and the chromium diphenylcarbazide complex is then developed. Spectrophotometric measurement is made at 540 nm.

12. Chromium Concentration Range

12.1 The recommended concentration range is from 0.02 mg to 0.10 mg of chromium per 250 mL of solution, using a 2-cm cell.

NOTE 1—This test method has been written for cells 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.

13. Stability of Color

13.1 The color of the chromium complex develops almost immediately but starts to fade after about 10 min. Spectrophotometric measurements should be made within 5 min after developing the color.

14. Interferences

14.1 The elements ordinarily present do not interfere if their mass fractions are under the maximum limits shown in 1.1.

15. Reagents

15.1 *Acetone* (CH_3COCH_3).

15.2 *Ammonium Peroxydisulfate Solution* (100 g/L)—Dissolve 10 g of ammonium peroxydisulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$) in water and dilute to 100 mL. Do not use a solution that has stood more than 12 h.

15.3 *Chromium, Standard Solution* (1 mL = 0.005 mg Cr)—Dissolve 0.2830 g of potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in water in a 1-L volumetric flask, dilute to volume, and mix. Using a pipet, transfer 5 mL to a 100-mL volumetric flask, dilute to volume, and mix.

15.4 *Diphenylcarbazide Solution* (5 g/L)—Dissolve 0.50 g of diphenylcarbazide (1,5-diphenylcarbohydrazide) in 100 mL of acetone. Do not use a solution that has stood for more than 1 h.

15.5 *Potassium Pyrosulfate* ($\text{K}_2\text{S}_2\text{O}_7$).

15.6 *Silver Nitrate Solution* (2.5 g/L)—Dissolve 0.25 g of silver nitrate (AgNO_3) in water and dilute to 100 mL.

15.7 *Sodium Hydroxide Solution* (500 g/L)—Dissolve 50 g of NaOH in water and dilute to 100 mL.

15.8 *Purity of Water*—Unless otherwise indicated, references to water shall 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.

16. Preparation of Calibration Curve

16.1 *Calibration Solutions*:

16.1.1 Using a pipet, transfer (5, 10, 15, and 20) mL of chromium standard solution (1 mL = 0.005 mg Cr) to 400-mL beakers. Add 1 mL of H_3PO_4 (1 + 1) and dilute to 250 mL with water.

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.