



Designation: ~~E463-08~~ Designation: E 463 – 09

Standard Test Method for Analysis/Determination of Silica in Fluorspar by Silico- Molybdate Visible Spectrometry¹

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1. Scope

- 1.1 This test method covers the determination of silica in fluorspar in concentrations from 0.5 % to 10 %.
- 1.2 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.*

2. Referenced Documents

2.1 ASTM Standards:²

- D 1193 Specification for Reagent Water
- E 50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials
- E 135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials
- E 276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials
- E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory

3. Terminology

- 3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology E 135.

4. Summary of Test Method

4.1 The sample is fused with anhydrous sodium borate and the melt is dissolved in dilute HCl. Silica is determined photometrically after extraction of the silico-molybdate complex with normal butyl alcohol. Photometric measurement of the extract is made at 400 nm.

5. Significance and Use

5.1 This test method is intended as a referee method for compliance with compositional specifications for impurity content. It is assumed that all who use this procedure will be trained analysts capable of performing common laboratory practices skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Follow appropriate quality control practices such as those described in Guide E 882.

6. Interferences

- 6.1 The elements ordinarily present in commercial fluorspars do not interfere in this test method.

7. Reagents and Materials

7.1 *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

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 E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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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.

specifications are available.³ 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D 1193.

7.3 *Ammonium Molybdate Solution (100 g/L)*—Dissolve 100 g of ammonium-heptamolybdate [(NH₄)₆Mo₇O₂₄·4H₂O] in 500 mL of water, dilute to 1 L, and mix.

7.4 *Silica (SiO₂)*—Heat pure silicic acid in a platinum crucible to expel combined water by gradually increasing temperature to 1050 °C. Maintain at 1050 °C for at least 5 min. Cool to room temperature in a desiccator.

7.5 *Sodium Borate (Na₂B₄O₇)*—Anhydrous powder, low-silica content.

NOTE 1—If low silica sodium borate is not available, prepare the reagent as follows: Transfer 247 g of boric acid to a large platinum dish. Expel water by gradually increasing the temperature to about 1000 °C. When effervescence ceases, gradually introduce 106 g of sodium carbonate into the molten mass. Maintain at a temperature of about 1000 °C until a clear melt is obtained.

8. Hazards

8.1 For precautions to be observed in this method, refer to Practices E 50.

9. Sample Preparation

9.1 The analytical sample shall be pulverized, if necessary, to pass a 150-µm sieve (see Test Method E 276). Dry at 105 °C to 110 °C for a minimum of 1 h.

10. Procedure

10.1 Transfer 7 g of Na₂B₄O₇ to each of six 25-mL platinum crucibles. Form a cavity in the center of the flux.

10.2 Into Crucibles 1 and 2, weigh 0.100 g to 1 g of the dry sample. Choose sample weights to provide from 5 mg to 10 mg of SiO₂.

10.3 Into Crucibles 3 and 4, weigh 10.0 mg of SiO₂ reagent.

10.4 Crucibles 5 and 6 serve as blanks.

10.5 Mix the contents of the crucibles with a platinum or polyethylene rod. Transfer adhering particles to the crucible.

10.6 Cover the crucible and heat gently until moisture is expelled. Increase the temperature until complete fusion results.

NOTE 2—A Meker burner or a muffle furnace maintained at 1000 °C may be used for this purpose.

10.7 Transfer the platinum crucible and cover to a 400-mL polyethylene or TFE-fluorocarbon beaker containing 150 mL water and 25 mL HCl (1 + 1). Cool the crucible for about 3 s, then pour the melt dropwise into the beaker so that most of the flux settles on the crucible cover (Note 3). Transfer the cooled crucible to the beaker. Cover the beaker with a polyethylene sheet and secure it to the beaker with a rubber band.

NOTE 3—Hold the crucible while cooling to avoid contamination problems. This prevents damage to the beaker.

10.8 Place the beaker on a steam bath and swirl occasionally until the melt is completely dissolved (Note 4). Cool, remove and rinse the platinum crucible and cover, and add the washings to the beaker. Transfer the solution to a 250-mL volumetric flask. Rinse the beaker and add the rinsings to the flask. Dilute to volume, mix, and examine the solution for any insoluble material (Note 4). Transfer the solution to a dry polyethylene bottle.

NOTE 4—Complete dissolution of the melt requires about 2 h. In cases of incomplete dissolution, a new sample must be taken.

10.9 Transfer 50-mL aliquots of the blank and sample solutions to 200-mL polyethylene or TFE-fluorocarbon beakers.

10.10 Transfer (10.0, 20.0, 30.0, 40.0, and 50.0)-mL aliquots of the standard solutions to 200-mL polyethylene or TFE-fluorocarbon beakers. Dilute, if necessary, to a 50-mL volume with the remaining blank solution.

NOTE 5—Since commercially available Na₂B₄O₇ frequently contains appreciable amounts of silica, each standard and sample solution must contain the same amounts of this reagent. The dilution of the aliquots to a 50-mL volume should, therefore, be carried out using a buret.

10.11 Determine the pH of the blank, standard, and sample solutions using a pH meter. If the pH of the solutions lies between 0.5 and 0.9 and within 0.1 units of each other, proceed to 10.12. If the pH lies outside these parameters, adjust the pH with HCl (1 + 1).

NOTE 6—Accurate pH adjustments are essential for maximum color development and color stability.

10.12 Add, while stirring, 10 mL of ammonium molybdate solution. Allow 10 min for color development, then dilute to 100 mL. Transfer the solution to a 250-mL separatory funnel and add 25 mL of cool H₂SO₄ (1 + 1).

³ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, <http://www.chemistry.org>. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., <http://uk.vwr.com>, and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, <http://www.usp.org>.