**Designation: E463 – 21**

# Standard Test Method for Determination of Silica in Fluorspar by Silico-Molybdate Visible Spectrophotometry[1]

This standard is issued under the fixed designation E463; 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 covers the determination of silica in fluorspar from 0.5 % to 10 % by mass.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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

E276 Test Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials

E882 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 E135.

## 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 spectrophotometrically after extraction of the silico-molybdate complex with butyl alcohol. Spectrophotometric 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 E882.

## 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 specifications are available.[3] Other grades may be used,

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[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.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, www.usp.org.

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

7.3 *Ammonium Molybdate Solution* (100 g/L)—Dissolve 100 g of ammonium-heptamolybdate $[(NH_4)_6Mo_7O_{24}{\cdot}4H_2O]$ in 500 mL of water, dilute to 1 L, and mix.

7.4 *Silica* ($SiO_2$)—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* ($Na_2B_4O_7$)—Anhydrous powder, low-silica content.

7.5.1 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. Cool and break into pieces or grind for use.

7.6 Butyl Alcohol.

7.7 Ethyl Alcohol.

## 8. Hazards

8.1 For precautions to be observed in this method, refer to Practices E50.

## 9. Sample Preparation

9.1 The analytical sample shall be pulverized, if necessary, to pass a 150-µm sieve (see Test Method E276). Dry at 105 °C to 110 °C for a minimum of 1 h.

## 10. Procedure

10.1 Transfer 7 g of $Na_2B_4O_7$ 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 masses to provide from 5 mg to 10 mg of $SiO_2$.

10.3 Into Crucibles 3 and 4, weigh 10.0 mg of $SiO_2$ 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 1—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. Hold the crucible while cooling to avoid contamination problems. This prevents damage to the beaker. Transfer the cooled crucible to the beaker. Cover the beaker with a polyethylene sheet and secure it to the beaker with a rubber band.

10.8 Place the beaker on a steam bath and swirl occasionally until the melt is completely dissolved (Note 2). 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; if any insoluble material is noted, a new sample must be prepared. Transfer the solution to a dry polyethylene bottle.

NOTE 2—Complete dissolution of the melt requires about 2 h. If dissolution is incomplete, a new sample must be prepared.

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.

10.10.1 Since commercially available $Na_2B_4O_7$ 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 performed 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).

10.11.1 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 $H_2SO_4$ (1 + 1).

10.13 Add 75 mL of butyl alcohol and shake vigorously for 1 min. Allow the phases to separate and discard the acid (lower) layer. Add 20 mL of $H_2SO_4$ (1 + 99) to the separatory funnel, shake for 30 s, allow the phases to separate, and discard the acid layer. Repeat the washing twice more.

10.14 Transfer the butyl alcohol phase to a dry 100-mL volumetric flask. Wash the separatory funnel twice with 1-mL or 2-mL portions of butyl alcohol, and add the washings to the volumetric flask. Add 1 mL of ethyl alcohol, dilute to volume with butyl alcohol, and mix.

## 11. Spectrophotometry

11.1 *Concentration Range:*

11.1.1 The recommended concentration range is from 0.2 mg to 2.0 mg of silica per 100 mL of solution, using a 1-cm cell.