



Standard Test Method for Silica in Fluorspar by the Silico-Molybdate Visible Spectrometry¹

This standard is issued under the fixed designation E 463; 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 in concentrations from 0.5 to 10 %.

1.2 *This standard does not purport to address all of the safety problems, 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:

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals²

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 Materials²

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory³

3. Summary of Test Method

3.1 The sample is fused with anhydrous sodium borate and the melt is dissolved in dilute hydrochloric acid. 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.

4. Significance and Use

4.1 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.

¹ This test method is under the jurisdiction of ASTM Committee E-1 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.

Current edition approved Jan. 15, 1993. Published March 1993. Originally published as E463 – 72. Last previous edition E463 – 72 (1998).

² *Annual Book of ASTM Standards*, Vol 03.05.

³ *Annual Book of ASTM Standards*, Vol 03.06.

5. Concentration Range

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

NOTE 1—Cells having other dimensions may be used, provided suitable adjustments can be made in the amounts of sample and reagent used.

6. Stability of Color

6.1 After the addition of the ammonium molybdate, color is fully developed within 10 min and is stable after extraction with butyl alcohol.

7. Interferences

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

8. Reagents

8.1 *Purity and Concentration of Reagents*—The purity and concentration of the common chemical reagents used shall conform to Practices E 50. Special apparatus and reagents are located in separate sections preceding the procedure.

8.2 *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.

8.3 *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.

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

NOTE 2—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.

9. Sample Preparation

9.1 The analytical sample shall be pulverized, if necessary, to pass a No. 100 (150- μ m) sieve (see Test Method E 276). Dry at 105 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.