Designation: E 815 – 99

Standard Test Method for Determination of Calcium Fluoride in Fluorspar by Complexometric Titration[1]

This standard is issued under the fixed designation E 815; 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 calcium fluoride in acid-grade fluorspar and other types of fluorspar that can be rendered soluble by the procedure described in the test method.

1.2 *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:*

D 1193 Specification for Reagent Water[2]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[3]

E 276 Test Methods for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Finer for Metal-Bearing Ores and Related Materials[3]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[4]

3. Summary of Test Method

3.1 The sample is decomposed by digesting with nitric and perchloric acids and the fluorine is expelled by fuming. The residue is dissolved in dilute hydrochloric acid, the solution made alkaline, and the calcium titrated with standard EDTA solution. Calcium present as carbonate is determined in a separate sample with EDTA solution, after extracting the former with dilute acetic acid. A correction for calcium fluoride, solubilized by dilute acetic acid digestion, is applied, by determining the fluoride in the acetic acid extract by fluoride ion-selective electrode. The CaF_2 content is then calculated.

4. Significance and Use

4.1 Fluorspar is used as a flux in steelmaking, glass industry, and manufacture of hydrofluoric acid.

4.2 This test method is intended to be used for compliance with compositional specifications for calcium fluoride content. It is assumed that all who use these procedures 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 and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed such as those described in Guide E 882.

5. Interferences

5.1 None of the elements normally found in fluorspar interfere with this test method.

6. Apparatus

6.1 *Fluoride Ion-Selective Electrode.* [5]

6.2 *Magnetic Stirrer and TFE-Fluorocarbon-Coated Spin Bar*.

6.3 *pH Meter with High Impedance*—Suitable for ion-selective electrode.

6.4 *Polyethylene Beakers*, 100-mL.

6.5 *Single Junction Ag/AgCl Reference Electrode.* [6]

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.[7] Other grades may be used,

[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.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] Orion model 94-91 has been found suitable for this purpose.

[6] Orion model 90-01-00 has been found suitable for this purpose.

[7] *Reagent Chemicals, American Chemical Society Specifications*, 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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.