Designation: E1506 – 08

Standard Test Methods for Analysis of Acid-Grade Calcium Fluoride (Fluorspar)[1]

This standard is issued under the fixed designation E1506; 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 These test methods cover the chemical analyses of acid-grade calcium fluoride (fluorspar). These test methods appear in the following sections:

	Sections
Volatiles as Moisture	6 – 13
Silica	14 – 22
Assay as Calcium Fluoride (CaF_2)	23 – 32
Soluble Chloride as NaC1	33 – 50
Calcium Carbonate	51 – 59
Phosphorus	60 – 69
Arsenic	70 – 78
Mixed Oxides (R_2O_3)	79 – 87
Sulfide Sulfur	88 – 96

1.2 The values stated in SI units are to be regarded as the 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.*

1.4 Review the current Material Safety Data Sheet (MSDS) for each chemical used in this standard for detailed information concerning toxicity, first-aid procedures, handling, and safety precautions.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

E300 Practice for Sampling Industrial Chemicals

3. Significance and Use

3.1 Calcium fluoride is available in nature in various forms and purities. A major use for it is in the manufacture of hydrofluoric acid. The test methods listed in 1.1 provide procedures for analyzing calcium fluoride to determine whether it is suitable for this use.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that 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.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II or Type III reagent water conforming to Specification D1193.

5. Sampling

5.1 Sampling of calcium fluoride is not within the scope of these test methods. See the appropriate sections of Practice E300 for sampling procedures.

VOLATILES AS MOISTURE

6. Scope

6.1 This test method covers the determination of volatiles as percent moisture.

7. Summary of Test Method

7.1 The sample is dried in an air oven at 105 to 110°C, and the mass loss is calculated as percent moisture.

8. Apparatus

8.1 *Top-Loading Balance,* capable of weighing 1000 g to the nearest 0.01 g.

[1] These test methods are under the jurisdiction of ASTM Committee E15 on Industrial and Specialty Chemicalsand are the direct responsibility of Subcommittee E15.02 on Product Standards.

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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] The last approved version of this historical standard is referenced on www.astm.org.

[4] *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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

***A Summary of Changes section appears at the end of this standard**

8.2 *Sample Pan,* stainless steel or borosilicate glass, 152 by 152 by 51 mm deep.

8.3 *Cooling Rack,* wood or metal, able to allow circulation of air around the entire sample pan (for example, a "baker's rack").

8.4 *Forced Air Oven,* capable of maintaining temperatures of 105 to 110°C.

9. Hazards

9.1 See 1.3 and 1.4.

10. Procedure

10.1 Tare a clean, dry sample pan to the nearest 0.01 g.

10.2 Add approximately 1000 g of representative sample to the pan and spread evenly. Wipe all external surfaces of the pan free of sample. Weigh again to the nearest 0.01 g.

10.3 Place the pan containing the sample in an air oven at 105 to 110°C for a minimum of 12 h.

10.4 Remove the pan from the oven and place on a cooling rack for 1 h.

10.5 Weigh the cooled pan to the nearest 0.01 g.

10.6 Return the pan to the cooling rack and cool for an additional 30 min. Then reweigh the pan to the nearest 0.01 g.

10.7 Repeat 11.6 until consecutive weights agree within 0.05 g.

10.8 Once a consistent weight has been obtained, dump the sample on a flat, dry surface and spread it with a spatula. If the fluorspar is dry, it will appear dusty, powdery, and flour-like in consistency. If the fluorspar does not appear as such, repeat the analysis using fresh sample.

11. Calculation

11.1 Calculate percent volatiles as moisture as follows:

$$\text{volatiles as moisture, \% mass (m/m)} = \frac{(B - C) \times 100}{(B - A)} \quad (1)$$

where:

A = mass of empty pan, g (10.1),
B = mass of pan plus sample before drying, g (10.2), and
C = mass of pan plus sample after drying to consistent mass, g (10.7).

12. Report

12.1 Report the percent volatiles as moisture to the nearest 0.01 %.

13. Precision and Bias

13.1 *Precision*—The following criteria should be used for judging the acceptability of results (see Note 1):

13.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be the value shown in Table 1 at the indicated degrees of freedom. The 95 % limit for the difference between two such runs is the value shown in Table 1.

TABLE 1 Volatiles as Moisture Checking Limits for Duplicates

Volatiles Level, %	Standard Deviation	Degrees of Freedom	95 % Limit, % Absolute
6	0.0257	18	0.072
9	0.0822	18	0.230

13.1.2 *Laboratory Precision (Within-Laboratory, Between-Days)*—The standard deviation of results (each the average of duplicates) obtained by the same analyst on different days has been estimated to be the value shown in Table 2 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the value shown in Table 2.

13.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be the value shown in Table 2 at the indicated degrees of freedom. The 95 % limit for the difference between two such averages is the value shown in Table 2.

NOTE 1—These precision estimates are based on an interlaboratory study performed in 1992 in which samples of fluorspar from two lots, one containing about 6 % volatiles as moisture and the other about 9 % volatiles as moisture, were each analyzed in duplicate by one analyst on each of two days in each of ten laboratories for a total of 120 determinations.[5] Practice E180 was used in developing these precision estimates.

13.2 *Bias*—The bias of this test method has not been determined due to the unavailability of suitable reference materials.

SILICA

14. Scope

14.1 This test method covers the determination of percent silica.

15. Summary of Test Method

15.1 The sample is treated with 10 % acetic acid to remove carbonates and soluble salts, the residue is ignited in a 650°C muffle furnace, treated with 48 % hydrofluoric acid (HF), and then heated again at 650°C. The mass loss after the HF treatment is calculated as percent silica.

16. Apparatus

16.1 *Analytical Balance,* capable of weighing to the nearest 0.1 mg.

16.2 *Beaker,* 150-mL glass, unscratched, and watchglass cover.

16.3 *Graduated Cylinder,* 25-mL glass.

16.4 *Graduated Cylinder,* 10-mL polypropylene.

16.5 *Platinum Crucible,* 30-mL capacity with lid.

16.6 *Platinum Wire,* 4 cm by 2 mm.

16.7 *Stirring Rod,* borosilicate glass, unscratched.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:E15-1027.

TABLE 2 Volatiles as Moisture

Volatiles Level,%	Repeatability			Reproducibility		
	Standard Deviation	Degrees of Freedom	95 % Limit, % Absolute	Standard Deviation	Degrees of Freedom	95 % Limit,% Absolute
6	0.0238	9	0.067	0.0807	8	0.226
9	0.0666	9	0.186	0.0865	8	0.242

16.8 *Muffle Furnace,* capable of maintaining a temperature of 650 ± 10°C or higher.

16.9 *Desiccator,* with desiccant.

16.10 *Steam Bath.*

16.11 *Glass Filter Funnel.*

16.12 *Bunsen Burner,* ringstand, ring, and heating mesh.

16.13 *Disposable Pipets.*

16.14 *Mortar and Pestle,* 102-mm diameter, agate.

16.15 *Tongs,* platinum-tipped.

17. Reagents

17.1 *Acetic Acid Solution* (100 mL/L)—Dilute 10 mL of glacial acetic acid to 100 mL with water; mix well.

17.2 *Hydrofluoric Acid* (HF), 48 %.

17.3 *Ashless Cellulose Filter Aid,* Whatman accelerator powder,[6] or equivalent.

17.4 *Filter Paper,* 9-cm diameter, low-ash, acid-washed, medium-porosity, able to retain 8-µm particles.

17.5 *Filter Paper,* 9-cm diameter, low-ash, acid-washed, fine-porosity, able to retain 2.5-µm particles.

17.6 *Ethanol,* pure or denatured.

17.7 *Filter Pulp Slurry* (40 g/L)—Slurry 10 g of cellulose filter aid with 250 mL of water.

18. Hazards

18.1 See 1.3 and 1.4.

19. Procedure

19.1 Transfer 8 to 10 g of sample (previously dried to constant weight at 105 to 110°C) into a mortar. Grind with a pestle until the particle size is 100 to 500 mesh.

19.2 Weigh 1.0 g of the ground sample to the nearest 0.0001 g, and transfer it to a 150-mL beaker.

19.3 Wet the sample with 1 mL of ethanol, then add 15 mL of 10 % acetic acid to the beaker.

19.4 Add a glass stirring rod to the beaker, cover with a watchglass, and place on a steam bath.

19.5 Heat for 30 ± 1 min, stirring every 5 min.

19.6 Remove from the steam bath, add 5 mL of filter pulp slurry to the beaker, cover, and allow to sit for approximately 12 h.

19.7 Gravity filter the solution through medium-porosity filter paper.

19.8 Rinse the beaker several times with minimal portions of hot water (total wash water approximately 35 mL), filtering each wash through the same filter paper. Save the filtrate for the determination of Mixed Oxides (Section 79).

19.9 Wipe the beaker clean with one fourth of a fine-porosity filter paper, and transfer the wipe paper and the filter paper with the residue into a 30-mL platinum crucible.

19.10 Place a platinum wire across the top of the platinum crucible. Rest the crucible lid on the wire and place the crucible into a cool muffle furnace.

19.11 Heat the furnace slowly (1-h cycle) to 650 ± 10°C. Once the temperature has reached 650°C, check the crucible every 10 min until the paper is entirely burned off.

19.12 Cool the crucible to room temperature in a desiccator, then weigh the crucible, cover, and residue to 0.0001 g.

19.13 Using a 10-mL polypropylene graduate cylinder, carefully pour 3 mL of 48 % HF into the crucible.

19.14 Gently heat the crucible over a Bunsen burner in a hood until dry (see Note 2).

NOTE 2—The solution must be heated below boiling. Excess heat will cause erratic results. If unable to control heating using a bunsen burner, heat the solution on a hot plate at 60°C or below. Evaporation of the 6 mL of HF used in this procedure should take approximately 2 h.

19.15 Cool the crucible, then repeat 19.13 and 19.14.

19.16 Cover the crucible with a platinum lid; then carefully place it into a muffle furnace maintained at 650 ± 10°C.

19.17 Heat the crucible for 5 min; then place it into a desiccator to cool.

19.18 Weigh the crucible, cover, and residue to 0.0001 g.

20. Calculation

20.1 Calculate percent silica as follows:

$$\text{silica, \% mass (m/m)} = \frac{(B - C) \times 100}{A} \quad (2)$$

where:

A = mass of sample, g (19.2),
B = mass of crucible, cover, and residue before HF treatment, g (19.12), and
C = mass of crucible, cover, and residue after HF treatment, g (19.18).

21. Report

21.1 Report the percent silica to the nearest 0.01 %.

22. Precision and Bias

22.1 *Precision*—The following criteria should be used for judging the acceptability of results (see Note 3):

22.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.0319 % absolute at 50 df. The 95 % limit for the difference between two such runs is 0.09 % absolute.

[6] Available from Whatman LabSales, P.O. Box 1359, Hillsboro, OR, 97123-9981.