



Designation: E1506 – 08

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

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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 NaCl	33 – 50
Calcium Carbonate	51 – 59
Phosphorus	60 – 69
Arsenic	70 – 78
Mixed Oxides (R ₂ O 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Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals \(Withdrawn 2009\)](#)³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](#)

¹ These test methods are under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and are the direct responsibility of Subcommittee D16.16 on Industrial and Specialty 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.

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.

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 $650 \pm 10^\circ\text{C}$ 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 $650 \pm 10^\circ\text{C}$. 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 $650 \pm 10^\circ\text{C}$.

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.

22.1.2 *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 0.0362 % absolute at 25 df. The 95 % limit for the difference between two such averages is 0.10 % absolute.

22.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.0529 % absolute at 11 df. The 95 % limit for the difference between two such averages is 0.15 % absolute.

NOTE 3—These precision estimates are based on an interlaboratory study performed in 1992 in which samples of fluor spar from two lots, one containing about 0.5 % silica and the other about 1 % silica, were each analyzed in duplicate on each of two days by one analyst in each of 14 laboratories for a total of 112 determinations.⁵ Practice E180 was used in developing these precision estimates.

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

ASSAY AS CALCIUM FLUORIDE (CaF₂)

23. Scope

23.1 This test method covers the determination of assay as percent calcium fluoride (CaF₂).

24. Summary of Test Method

24.1 The residue remaining after the determination of silica (see 19.18) is treated with H₂SO₄, dried, then dissolved in HCl. Ammonium oxalate is added to the HCl solution to precipitate calcium oxalate, then the precipitate is dried and weighed. Percent CaF₂ is calculated from the mass of the calcium oxalate collected.

25. Interferences

25.1 Iron causes a positive interference. If iron is suspected to be present, its effect can be minimized by adding 1 mL of concentrated HNO₃ to the solution described in 29.8 before boiling.

25.2 Strontium precipitates, as the oxalate, along with calcium oxalate to produce erroneously high results.

25.3 A small amount of CaF₂ is lost in the acetic acid treatment used in 19.3, resulting in an erroneously low result. To correct for this loss, the term 0.15 is included in the calculation in 30.1.

26. Apparatus

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

26.2 *Beakers*, borosilicate glass, 800-mL, 400-mL, and watchglass covers.

26.3 *Graduated Cylinders*, borosilicate glass, 10-mL, 25-mL.

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

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

26.6 *Stirring Rod*, borosilicate glass.

26.7 *Muffle Furnace*, capable of maintaining a temperature of 1200 ± 10°C or higher.

26.8 *Desiccator*, with desiccant.

26.9 *Funnel*, borosilicate glass.

26.10 *Bunsen Burner*.

26.11 *Ringstand*, equipped with ring and heating gauze.

26.12 *Tongs*, platinum- or nickel-tipped.

27. Reagents

27.1 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H₂SO₄).

27.2 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

27.3 *Hydrochloric Acid Solution* (1 + 1)—Wearing goggles, carefully add 250 mL of concentrated HCl (sp gr 1.19) to 250 mL of water. Mix well.

27.4 *Ammonium Chloride* (NH₄Cl).

27.5 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH₄OH).

27.6 *Ammonium Oxalate Solution (Saturated)*—Add 30 g of ammonium oxalate to a 1-L polyethylene bottle. Add 1000 mL of hot water to the bottle and mix well. Allow the solution to cool. Add additional ammonium oxalate if necessary to keep crystals present at the bottom of the bottle at all times.

27.7 *Ammonium Oxalate Solution* (1 g/L)—Add 0.1 g of ammonium oxalate to 100 mL of water and mix well.

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

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

27.10 *pH Paper*—Litmus.

28. Hazards

28.1 See 1.3 and 1.4.

29. Procedure

29.1 Add 5 mL of concentrated sulfuric acid to the residue remaining in the crucible from 19.18.

29.2 Partially cover the crucible and gently heat over a bunsen burner in a hood until all H₂SO₄ is driven off (see Note 4).

NOTE 4—Do not heat directly with the flame.

29.3 Repeat 29.1 and 29.2 using 3 mL of concentrated sulfuric acid.

29.4 Cool the crucible and transfer the crucible, cover, and residue into a 400-mL beaker.

29.5 Add 10 mL of concentrated hydrochloric acid, 5 g of ammonium chloride, and 200 mL of hot water to the beaker; mix well.

29.6 Warm the solution to between 70 and 80°C on a hot plate in a hood; keep at this temperature for 2 h.

29.7 Remove the crucible and lid from the solution using platinum or nickel-tipped tongs. Rinse each with warm water, collecting the washings in the beaker. Scrape any remaining residue from the crucible into the solution with a rubber policeman.

29.8 Cover with a watchglass, then boil the solution for 10 min to dissolve any solid matter (see **Note 5** and **25.1**).

NOTE 5—If insolubles are still present after the 10-min boil, filter the solution through medium-porosity filter paper, then return the residue and paper to the crucible. Place platinum wire across the top of the crucible, rest the lid on the wire, and place the crucible into a cool muffle furnace. Heat the furnace slowly to 650°C ($\pm 10^\circ\text{C}$, 1-h cycle). At 650°C, check the crucible every 10 min until the paper burns off. Repeat **29.1** to **29.8** using 1 mL of concentrated sulfuric acid. Combine the filtrates in one beaker, then continue with **29.9**.

29.9 Allow the solution to cool, then add ammonium hydroxide dropwise while mixing, until the solution tests basic (blue) to Litmus paper.

29.10 Mix well, cover, and then boil for 1 min.

29.11 Allow the solution to cool slightly. If necessary, add ammonium hydroxide dropwise while mixing until the solution tests basic (blue) to Litmus paper.

29.12 Gravity filter this solution through medium-porosity filter paper, into an 800-mL beaker.

29.13 Wash the filter paper and residue several times with hot water, collecting the filtrates in the 800-mL beaker (see **29.12**).

29.14 Wash the filter paper and residue with 20 mL of hot 1 + 1 HCl solution, then four 20-mL portions of hot distilled water, collecting the filtrates in the 400-mL beaker.

29.15 Adjust the pH of the solution in the 400-mL beaker with ammonium hydroxide until it tests basic (blue) to Litmus paper.

29.16 Boil this solution for 1 min, then allow to cool slightly just below boiling.

29.17 Filter through the original filter paper. Wash with hot water and collect the filtrate in the 800-mL beaker. Save the filter cake for the determination of Mixed Oxides (Section **79**). Bring the filtrate to a boil. Add 100 mL of saturated ammonium oxalate solution, then add 5 mL of filter pulp slurry and stir to mix.

29.18 Boil the solution for 30 s, then allow it to cool until precipitate settles. The sample can sit overnight before filtering, if necessary or convenient.

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

29.20 Rinse the beaker with 10 to 15 mL of cold 0.1 % ammonium oxalate solution. Transfer the washings into the filter, using them to wash the precipitate.

29.21 Repeat **29.20** two more times.

29.22 Wash the residue with three 10 to 15-mL portions of 0.1 % ammonium oxalate solution.

29.23 Wash the residue with three 10 to 15-mL portions of cold water.

29.24 Weigh a 30-mL platinum crucible and cover to 0.0001 g (mass *B*).

29.25 Transfer the filter paper and residue into the platinum crucible.

29.26 Place a platinum wire across the top of the platinum crucible, rest the lid on the wire, and place the crucible into a cool muffle furnace.

29.27 Heat the furnace slowly to 1200°C. Check to see if all paper is burned off.

29.28 Keep crucible at 1200°C for 20 min.

29.29 Remove crucible and place in a desiccator containing fresh desiccant; allow to cool to room temperature.

29.30 Immediately weigh the crucible, cover, and residue to 0.0001 g (mass *C*).

30. Calculation

30.1 Calculate assay as percent CaF₂ as follows:

$$\text{assay as CaF}_2, \% \text{ mass (m/m)} = \frac{(C - B) \times 1.3923 \times 100}{A} + 0.15 \quad (3)$$

where:

- A* = mass of fluor spar sample, g, (see **19.2**),
- B* = mass of crucible and cover, g, (see **29.24**),
- C* = mass of crucible, cover, and residue, g (see **29.30**),
- 1.3923 = conversion factor for CaO (molecular weight (MW) = 56.08) to CaF₂ (MW = 78.08), and
- 0.15 = correction for amount of calcium fluoride lost in the acetic acid treatment, considered to be 0.0015 g CaF₂/g sample.

31. Report

31.1 Report the percent calcium fluoride to the nearest 0.01 %.

32. Precision and Bias

32.1 *Precision*—The following criteria should be used for judging the acceptability of results (see **Note 6**):

32.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 0.1778 % absolute at 30 df. The 95 % limit for the difference between two such runs is 0.50 % absolute.

32.1.2 *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 0.1169 % absolute at 15 df. The 95 % limit for the difference between two such averages is 0.33 % absolute.

32.1.3 *Reproducibility (Multilaboratory)*—The standard deviation of results (each the average of duplicates) obtained by analysts in different laboratories has been estimated to be 0.3559 % absolute at 6 df. The 95 % limit for the difference between two such averages is 1.00 % absolute.

NOTE 6—These precision estimates are based on an interlaboratory study performed in 1992 on two samples (one a commercial sample, the

other a reference material) each containing approximately 98 % calcium fluoride. One analyst in each of eight laboratories performed duplicate determination of a sample of NIST Standard Reference Material 79a on each of two separate days for a total of 32 determinations. The same protocol was used on a sample of commercial material except that seven laboratories participated for a total of 28 determinations.⁵ Practice E180 was used in developing precision estimates.

32.2 *Bias*—An average of 97.71 % calcium fluoride was obtained on NIST Standard Reference Material 79a which has a certified value of 97.39 %. This certified value, with a standard deviation of 0.06 % absolute for a single determination, was obtained using the U.S. Customs Laboratory Method (volumetric permanganate) as given in the certificate.

SOLUBLE CHLORIDE AS NaCl

33. Volumetric Procedure, Scope

33.1 This test method covers the volumetric determination of trace quantities of soluble chloride as percent NaCl.

34. Summary of Test Method

34.1 Soluble chloride is extracted from fluor spar with hot water; the extract is filtered, then titrated to a colorimetric end point with standardized silver nitrate solution.

35. Apparatus

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

35.2 *Pipets*, 1-mL, 10-mL glass.

35.3 *Graduated Cylinder*, 100-mL glass.

35.4 *Beakers*, 150-mL, 250-mL.

35.5 *Burets*, 10-mL, 25-mL glass.

35.6 *Volumetric Flask*, 1-L glass.

36. Reagents

36.1 *Potassium Chromate Indicator Solution* (50 g/L)—Dissolve 50 g K_2CrO_4 in 500 mL of water. Add silver nitrate solution (see 36.3) until a definite red precipitate is formed. Allow to stand 12 h, filter through fine-porosity filter paper and dilute the filtrate to 1 L with water.

36.2 *Sodium Chloride Standard Solution* (0.0141 meq/mL)—Dissolve 0.8241 g of NaCl (previously dried to constant weight at 105 to 110°C) in water and dilute to 1 L; mix well.

36.3 *Silver Nitrate Standard Titrant* (0.0141 meq/mL)—Dissolve 2.395 g of $AgNO_3$ (previously dried to constant weight at 105 to 110°C) in water and dilute to 1 L; mix well.

36.3.1 Pipet 10.0 mL of 0.0141 meq/mL NaCl (see 36.2) into a 150-mL beaker, add 40 mL of water and 1 mL of K_2CrO_4 indicator solution. Using a 25-mL buret, titrate this solution with the 0.0141 meq/mL $AgNO_3$ to a faint brown end point (see Note 7). Similarly determine a blank using all of the above reagents, but no NaCl. The titer of the $AgNO_3$ in μg Cl/mL, A , is as follows:

$$A = \frac{(500 \times 10)}{B} \quad (4)$$

where:

500 = Cl in 0.0141 meq/mL NaCl, $\mu g/mL$, and

B = $AgNO_3$ required for titration of the solution, net mL.

Store standardized solution in a brown glass bottle.

36.4 *Filter Paper*, 12.5-cm diameter, fine-porosity.

36.5 *Denatured Alcohol*.

37. Hazards

37.1 See 1.3 and 1.4.

38. Procedure

38.1 Weigh 25 g of sample (previously dried to constant weight at 105 to 110°C) to the nearest 1 g into a 150-mL beaker; wet the sample with 10 mL of denatured alcohol.

38.2 Add 100 mL of hot distilled water to the beaker. Using a magnetic stirrer, stir the mixture for 1 h; allow the mixture to cool and the fluor spar to settle.

38.3 After a minimum of 2-h settling time, gravity-filter the solution through a 12.5-cm diameter fine-porosity filter paper, collecting the filtrate in a 250-mL beaker.

38.4 Pipet 1 mL of potassium chromate indicator solution into the beaker.

38.5 Using a 10-mL buret, titrate with 0.0141 meq/mL $AgNO_3$ dropwise to a faint brown end point; mL = A (see Note 7).

NOTE 7—To aid in the determination of the end point, place a 250-mL beaker with the same volume of water and indicator next to the sample, as a comparator. The first brownish color that appears in the sample is the end point.

38.6 Similarly, determine a blank using all of the above reagents, but no sample; mL = B .

39. Calculation

39.1 Calculate percent soluble chloride as NaCl as follows:

$$\text{Soluble chloride as NaCl, \% mass (m/m)} = \frac{(A - B) \times C \times 1.6485}{D \times 10^4} \quad (5)$$

where:

A = 0.0141 meq/mL $AgNO_3$ used for sample, mL,

B = 0.0141 meq/mL $AgNO_3$ used for blank, mL,

C = Cl/mL of titrant, μg ,

D = mass of sample, g (see 38.1), and

1.6485 = conversion Cl (MW = 35.45) to NaCl (MW = 58.45); and $58.56/35.45 = 1.6485$.

40. Report

40.1 Report the soluble chloride as NaCl to the nearest 0.01 %.

41. Precision and Bias

41.1 *Precision*—The following criteria should be used for judging the acceptability of results (see Note 8):

41.1.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be