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Replacing GB/T 5195.1-2006

Fluorspar - Determination of calcium fluoride content - EDTA titration method and distillation-potentiometric titration method

萤石 氟化钙含量的测定

EDTA 滴定法和蒸馏-电位滴定法

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Table of Contents

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Fluorspar - Determination of calcium fluoride content - EDTA titration method and distillation-potentiometric titration method

Warning - The personnel using this part shall have practical experience in formal laboratory work. This part does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated by relevant national laws and regulations.

1 Scope

This part of GB/T 5195 specifies the methods for determining the content of calcium fluoride by EDTA titration and distillation-potentiometric titration.

This part applies to the determination of calcium fluoride content in fluorite. EDTA titration method has a measuring range (mass fraction): ≥ 60%; distillation-potentiometric titration method has a measuring range (mass fraction): ≥ 90%.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this standard.

GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions

GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results - Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

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it slightly; transfer it into a reagent bottle pre-filled with 250 mL hydrochloric acid (ρ = 1.19 g/mL) and 600 mL water; cool to room temperature; use water to dilute it to 1 L.

3.2.5 Hydrochloric acid, 1 + 1.

3.2.6 Calcium-containing acetic acid solution.

Weigh 2.00 g of calcium carbonate in a 500 mL beaker; add 50 mL of acetic acid (1 + 9, prepared with ρ = 1.05 g/mL glacial acetic acid); heat and boil to drive off carbon dioxide; cool to room temperature; transfer to a 1000 mL volumetric flask; use acetic acid (1 + 9, prepared with ρ = 1.05 g/mL glacial acetic acid) to dilute it to the mark; mix it uniformly.

3.2.7 Saturated boric acid solution.

Weigh 6 g of boric acid in a 250 mL beaker; add water to 100 mL; heat to dissolve it; remove it; cool. The supernatant is a saturated boric acid solution.

3.2.8 Potassium hydroxide solution, 200 g/L.

3.2.9 Magnesium sulfate solution, 5 g/L.

3.2.10 Triethanolamine, 1 + 2.

3.2.11 Calcium fluoride standard solution, 0.0015601 g/mL.

Weigh 1.0008 g of calcium carbonate (> 99.99%) that has been pre-dried at 105 °C ~ 110 °C for 2 h and placed in a desiccator to cool to room temperature in a 250 mL beaker; cover a watch glass; slowly add 25 mL of hydrochloric acid (3.2.5). After the calcium carbonate is dissolved, add 100 mL of water; heat to boil it; drive off the carbon dioxide; cool to room temperature. Transfer the solution into a 500 mL volumetric flask; use water to dilute to the mark; mix it uniformly. 1.00 mL of this calcium standard solution is equivalent to 0.0015601 g calcium fluoride.

3.2.12 EDTA standard titration solution, c(EDTA) = 0.015 mol/L.

a) Preparation:

Weigh 5.8 g of disodium ethylenediaminetetraacetate ($C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$, EDTA for short) into a 400 mL beaker; add 200 mL of water; use potassium hydroxide solution (3.2.8) to adjust the pH of the solution to 5 ~ 5.5; heat to dissolve the reagents completely; cool to room temperature, transfer it into a 1000 mL volumetric flask; use water to dilute it to the mark; mix it uniformly; place it for 3 days before calibration.

b) Calibration:

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Unless otherwise specified, the volumetric flask used shall meet the requirements of GB/T 12806; the graduated pipette bottle and one-mark pipette shall meet the requirements of GB/T 12807 and GB/T 12808, respectively.

3.4 Sample preparation

3.4.1 Prepare specimen according to GB/T 22564.

3.4.2 The specimen shall be processed to a particle size less than 0.063 mm, dried at a temperature of 105 °C ± 5 °C for 2 h, placed in a desiccator to cool to room temperature.

3.5 Analytical procedures

3.5.1 Number of determinations

For the same specimen (3.4.2), make at least 2 independent determinations.

3.5.2 Sample amount

Weigh 0.50 g of specimen, accurate to 0.0001 g.

3.5.3 Blank test

Do a blank test with the sample.

3.5.4 Sample decomposition

3.5.4.1 Alkali fusion

3.5.4.1.1 Place the sample (3.5.2) in a 100 mL beaker; add 10 mL of calcium-containing acetic acid solution (3.2.6); cover a watch glass; heat at about 90 °C for 3 min at low temperature; remove it; keep it warm for 2 min.

Note: It may also place the sample (3.5.2) in a 250 mL beaker; add 10 mL of calcium-containing acetic acid solution (3.2.6); cover a watch glass; place it at room temperature for 30 min; shake or stir it once every 5 min.

3.5.4.1.2 Use slow speed filter paper to filter it. Transfer all the insoluble matter to the filter paper; use water to wash the beaker 3 ~ 5 times; use water to wash the residue 7 ~ 8 times.

3.5.4.1.3 Place the filter paper together with the residue in a platinum crucible; put it into the high-temperature furnace below 300 °C; slowly heat up to 850 °C to burn carbon; take out the platinum crucible; cool to room temperature. Add 4 g of mixed flux (3.2.1); place it in a high-temperature furnace; slowly increase the temperature to 950 °C and melt for about 20 minutes, until it is completely clear.

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T_1 - Titer of EDTA standard titration solution to calcium fluoride, in grams per milliliter (g/mL);

V_0 - The volume of the sample solution, in milliliter (mL);

V_2 - The volume of the sample solution taken, in milliliter (mL);

V_3 - The volume of the EDTA standard titration solution consumed by the sample solution in the titration, in milliliters (mL);

V_{02} - The volume of the EDTA standard titration solution consumed by titrating the blank test solution taken, in milliliter (mL);

m - The mass of the sample, in grams (g).

3.6.2 General processing of results

3.6.2.1 Precision

The precision test in this part is determined by 8 laboratories in 2015 by conducting joint tests on 5 fluorite samples with different calcium fluoride content levels; each laboratory measures the calcium fluoride content at each level 3 times under the repeated conditions specified in GB/T 6379.1.

See Appendix A for the original data (measurement results) reported by each laboratory.

According to GB/T 6379.2, the measurement results obtained are statistically analyzed; the precision is as shown in Table 1.

Table 1 -- Precision

3.6.2.2 Determination of analysis results

According to the procedures in Appendix B, the independent repeated measurement results are calculated according to formula (2) and compared with the repeatability limit r, to determine the analysis results. Round off the final result to two decimal places according to the provisions of GB/T 8170.

3.6.2.3 Inter-laboratory precision

Inter-laboratory precision is often used to evaluate the consistency between the final results reported by two laboratories. After the two laboratories report the results according to the procedure in 3.6.2.2, calculate the average of the final results according to formula (3):

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n - The number of repeated determinations of the standard sample;

u_{CRM} - Uncertainty of CRM/RM sample standard value.

4 Distillation-potentiometric titration method

4.1 Principle

In the presence of perchloric acid, the fluorine in the sample is separated by distillation with water vapor at 135 °C ± 2 °C through a temperature control device; the distillate is absorbed by sodium hydroxide solution; the fluoride ion selective electrode is used as the indicator electrode; the lanthanum nitrate standard titration solution is used to titrate the amount of fluorine in the distillate, to calculates the mass fraction of calcium fluoride.

4.2 Reagents

Unless otherwise specified in the analysis, only use approved analytical reagents and distilled water of grade 3 or higher that meets the requirements of GB/T 6682 or water of equivalent purity.

4.2.1 Potassium permanganate, crystalline.

4.2.2 Sodium fluoride, recrystallize according to the following method.

Dissolve 5 g of sodium fluoride in 125 mL water; use a Buchner funnel to filter under reduced pressure. The filtrate is evaporated to about 60 mL in a platinum dish; cooled to about 50 °C; then the sodium fluoride crystals are separated by centrifugation. Use a small amount of cold water to wash the crystals three times by centrifugation method. Move the crystals into a platinum dish; dry them in an electric oven at 105 °C ± 2 °C; cool it in a desiccator; grind the crystals in an agate mortar; use a sieve with a 0.355 mm hole to screen it. Place the sieved material in a platinum dish; heat it in a high temperature furnace of about 600 °C for 2 h; cool it in a desiccator.

4.2.3 Ethanol or isopropanol.

4.2.4 Perchloric acid, ρ = 1.67 g/mL.

4.2.5 Perchloric acid, 1 + 6.

4.2.6 Sodium hydroxide solution, 40 g/L, stored in a plastic bottle.

4.2.7 Standard titration solution of lanthanum nitrate, about 0.01 mol/L.

a) Preparation:

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4.4 Sample preparation

4.4.1 Prepare specimens according to GB/T 22564.

4.4.2 The specimen shall be processed to a particle size of less than 0.063 mm; dried at a temperature of 105 °C ± 5 °C for 2 hours; placed in a desiccator to cool to room temperature.

4.5 Analytical procedures

4.5.1 Number of determinations

For the same specimen (4.4.2), make at least 2 independent determinations.

4.5.2 Sample quantity

Weigh 0.20 g of specimen in a distillation flask, accurate to 0.0002 g.

Note: The weighed sample can also be placed in a flat-bottomed straight borosilicate glass with an inner diameter of about 10 mm, a height of 10 mm ~ 12 mm, a wall thickness of 1 mm; then the small cup and the sample can be placed in the distillation flask.

4.5.3 Blank test

Do a blank test with the sample.

4.5.4 Distillation

Connect the distillation unit (4.3.2). Remove the contact thermometer and distillation head from the distillation device; add a small amount of potassium permanganate (4.2.1) to the distillation flask; add 15 mL of water, 35 mL of perchloric acid (4.2.4); immediately use the distillation head and contact thermometer to seal the distillation device.

Take a 500 mL volumetric flask containing 25 mL of sodium hydroxide solution (4.2.6) and 40 mL of water under the water pipe; insert the water pipe into the solution.

Turn the three-way switch between the steam generator and the distillation device to position 1 (see Figure 1); set the contact thermometer to 135 °C; turn on the power of the distillation flask and the steam generator. Heat the solution in the distillation flask to 135 °C (about 15 min); turn the three-way switch to position 2 (see Figure 1); let the steam enter the distillation flask; control the amount of steam to flow out 10 mL distillate per minute. Collect about 400 mL distillate and stop the distillation.

Use water to rinse the inner and outer walls of the connecting pipe; collect the

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