

ICS 73.080

CCS D 52



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 5195.2-2006

Fluorspar -- Determination of carbonate content

萤石 碳酸盐含量的测定

(English Translation)

Issued on: August 16, 2006

Implemented on: January 1, 2007

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of PRC**

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1 Scope

This standard specifies the principles, reagents, apparatus, specimens, analytical procedures, calculation of analytical results, permissible tolerances for the determination of carbonate content in fluorspar.

This standard comprises two methods: the EDTA titration method, with a determination range (expressed as the mass fraction of calcium carbonate) of 0.10% ~ 3.00%; the acid-base titration method, with a determination range (expressed as the mass fraction of calcium carbonate) of $w_{\text{CaCO}_3} \geq 0.040\%$.

This standard is applicable to the determination of carbonate content in fluorspar.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 601 Chemical reagent Preparations of standard volumetric solutions

3.1 Principle

The test material is leached with dilute acetic acid at room temperature and filtered. An aliquot of the filtrate is taken to determine the amount of leached calcium fluoride, using the zirconium-xyleneol orange decolorization photometric method. The remaining filtrate is treated with EDTA titration to determine the calcium content; subtract the amount of calcium fluoride extracted during the leaching process, to calculate the mass fraction of carbonates (expressed as calcium carbonate).

3.2 Reagents

3.2.1

Hydrochloric acid

1 + 1.

3.2.2

Acetic acid

1 + 9, prepared using glacial acetic acid ($\rho \sim 1.05 \text{ g/mL}$).

3.2.3 Zirconium-hydrochloric acid solution

Weigh 100 mg of zirconyl chloride ($\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$) into a beaker; add 50 mL of hydrochloric acid ($\rho \sim 1.19 \text{ g/mL}$) to dissolve it; use water to transfer the solution into a 1000 mL volumetric flask; add 450 mL of hydrochloric acid ($\rho \sim 1.19 \text{ g/mL}$);

use water to dilute it to the mark; mix well.

3.2.4 Triethanolamine: 1 + 1.

3.2.5 Potassium hydroxide solution: 200 g/L.

3.2.6 Magnesium sulfate solution: 5 g/L.

3.2.7 Xylenol orange solution: 2 g/L.

3.2.8 Calcium indicator: Finely grind and thoroughly mix 0.5 g of calcium indicator

with 50 g of dried potassium chloride; store in a ground-glass stoppered bottle for future use.

3.2.9 Fluoride standard solutions

3.2.9.1

Fluoride stock solution

1.00 mg/mL.

Weigh 2.2100 g of sodium fluoride ($> 99.9\%$), which was previously ignited at 650°C for 1 hour and cooled to room temperature; dissolve it in a small amount of water;

transfer the solution into a 1000 mL volumetric flask; use water to dilute it to the mark;

mix well; store in a plastic bottle.

3.2.9.2

Fluoride standard solution

10.0 µg/mL.

Pipette 10.00 mL of the fluoride stock solution (3.2.9.1) into a 1000 mL volumetric flask; use water to dilute it to the mark; mix well; store in a plastic bottle.

3.2.10 Calcium standard solution: 0.01000 mol/L.

Weigh 1.0008 g of calcium carbonate (> 99.9%), which was previously dried at 105 °C

~ 110 °C for 2 h and cooled to room temperature in a desiccator, into a 400 mL beaker.

Cover with a watch glass; slowly add 25 mL of hydrochloric acid (3.2.1). Once the calcium carbonate has dissolved, add 100 mL of water; heat to boiling to expel the carbon dioxide; cool to room temperature. Transfer the solution into a 1000 mL volumetric flask; use water to dilute it to the mark; mix well.

3.2.11 EDTA standard titration solution: 0.01 mol/L.

3.2.11.1 Preparation

Weigh 3.72 g of disodium ethylenediaminetetraacetate ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_8\text{Na}_2 \cdot 2\text{H}_2\text{O}$, abbreviated as EDTA) into a 400 mL beaker. Add 200 mL of water; heat to dissolve;

cool to room temperature. Transfer the solution into a 1000 mL volumetric flask; use water to dilute it to the mark; mix well.

3.2.11.2 Calibration

Pipette three sets of 25.00 mL calcium standard solution (3.2.10) into separate 250 mL conical flasks; use water to dilute each to 100 mL. Add 5 mL of triethanolamine (3.2.4),

5 drops of magnesium sulfate solution (3.2.6), 20 mL of potassium hydroxide solution (3.2.5), a small amount of calcium indicator (3.2.8). Titrate with the standard EDTA titration solution (3.2.11), until the test solution changes color from red to pure blue, which is the endpoint. If the range of the volumes of standard EDTA titration solution consumed by the three sets of standard calcium solution does not exceed 0.10 mL, take the average value to calculate the concentration.

3.2.11.3 Calculation

Calculate the concentration of the standard EDTA titration solution using formula (1):

Where:

c - Concentration of the standard EDTA titration solution, in moles per liter (mol/L);

c₁ - Concentration of the standard calcium solution, in moles per liter (mol/L);

V - Average volume of standard EDTA titration solution consumed during the titration of the

three sets of standard calcium solution, in milliliters (mL);

V₀ - Volume of standard EDTA titration solution consumed during the blank test, in milliliters (mL).

3.3 Specimen

Grind the sample in an agate mortar, until it completely passes through a 0.063 mm sieve. Dry the sieved sample at 105 °C ± 2 °C for 2 hours; then cool it to room temperature in a desiccator.

3.4.1 Test material amount

Weigh 0.50 g of the specimen (3.3), accurate to 0.0001 g.

3.4.2 Blank test

Perform a blank test concurrently with the analysis of the test material.

3.4.3 Decomposition of test material

Place the test material (3.4.1) into a 250 mL beaker; add 20 mL of acetic acid (3.2.2); cover with a watch glass. Allow the mixture to stand at room temperature for 30 ~ 40 minutes, shaking it once every few minutes. Filter the leachate into a 100 mL volumetric flask using a slow-speed filter paper supplemented with paper pulp. Wash the beaker and the precipitate 4 times each with water. Dilute the filtrate with water to the mark; mix thoroughly.

Note: If the room temperature is below 20 °C, the beaker may be placed in a warm water bath at 25 °C ~ 30 °C for extraction.

3.4.4.1 Color-developing solution

Using a dry pipette to transfer 5.00 mL of the filtrate (3.4.3) into a 50 mL volumetric flask. Add 10.00 mL of the zirconium-hydrochloric acid solution (3.2.3) and 2.0 mL of the xylenol orange solution (3.2.7). Use water to dilute it to the mark; mix thoroughly; allow to stand for 10 min.

Note: After washing the pipette with water, allow it to dry vertically; then, use filter paper to absorb the water column remaining at the tip of the pipette, after which it may be used directly to pipette the filtrate.