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

GB/T 5195.12-2016

**Fluorite - Determination of arsenic content - Atomic
fluorescence spectrometry**

萤石 砷含量的测定 原子荧光光谱法

(English Translation)

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Foreword

GB/T 5195 is divided into 12 parts.

- Part 1.Fluorite -- Determination of calcium fluoride content -- EDTA titration method and distillation-potentiometric titration method;
- Part 2.Fluorspar -- Determination of carbonate content;
- Part 3.Fluorspar -- Determination of loss in mass at 105°C -- Gravimetric method;
- Part 4.Fluorspar -- Determination of sulfide content -- Iodometric method;
- Part 5.Fluorspar -- Determination of total sulfur content -- The iodometric method after combustion;
- Part 6.Fluorspar -- Determination of phosphorus content;
- Part 7.Fluorspar -- Determination of zinc content -- Flame atomic absorption spectrophotometric method;
- Part 8.Fluorspar -- Determination of silicon dioxide content;
- Part 9.Fluorspar -- Determination of loss on ignition -- Gravimetric method;
- Part 10.Fluorspar -- Determination of iron content -- 1, 10-phenanthroline spectrophotometric method;

- Part 11. Fluorspar -- Determination of manganese content -- Periodate spectrophotometry and flame atomic absorption spectrometry;
- Part 12. Fluorite -- Determination of arsenic content -- Atomic fluorescence spectrometry.

This is Part 12 of GB/T 5195.

This Part was drafted in accordance with the rules given in GB/T 1.1-2009.

This Part was proposed by China Iron and Steel Association.

This Part shall be under the jurisdiction of National Technical Committee on Steel of Standardization Administration of China (SAC/TC 183).

The drafting organizations of this Part. Shanghai Entry-Exit Inspection and Quarantine Bureau Industrial Products and Raw Materials Testing Technology Center, Metallurgical Industry Information Standards Research Institute.

Fluorite - Determination of arsenic content - Atomic fluorescence spectrometry

WARNING -- Personnel using this part shall have practical experience in regular laboratory work. This part does not point out all possible safety issues. Users are responsible for taking appropriate safety and health measures and ensuring that the conditions specified in relevant national laws and regulations are met.

1 Scope

This Part of GB/T 5195 specifies the determination of arsenic content by hydride generation-atomic fluorescence spectrometry.

This Part applies to the determination of arsenic content in fluorite. Determination range (mass fraction). 0.00001%~0.010%.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6379.1, Accuracy (trueness and precision) of measurement methods and

results -- Part 1.General principles and definitions

GB/T 6379.2, Measurement methods and results -- Accuracy (trueness and precision)

-- Part 2.Determine the standard methods of measurement repeatability and reproducibility of the basic method

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

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

GB/T 12806, Laboratory glassware -- One-mark volumetric flasks

GB/T 12807, Laboratory glassware -- Graduated pipettes

GB/T 12808, Laboratory glassware -- One mark pipettes

GB/T 21191, Atomic fluorescence spectrometer

GB/T 22564, Fluorspar -- Sampling and sample preparation

3 Principle

The test material is dissolved with nitric acid and perchloric acid. In a dilute hydrochloric acid medium, a thiourea-ascorbic acid mixed solution is added to reduce arsenic (V) to arsenic (III). In the hydride generator, arsenic is reduced to hydride by potassium borohydride. Argon gas is introduced into the quartz furnace atomizer. The fluorescence intensity of arsenic is measured on an atomic fluorescence spectrometer. The arsenic content is calculated according to the standard curve method.

4 Reagents

Unless otherwise specified, only analytically pure reagents shall be used. Water shall be deionized water or water of equivalent purity, which shall comply with the requirements for grade two in GB/T 6682.

4.13 Potassium borohydride solution, 20 g/L. Weigh 10.0 g of potassium borohydride

and 2.5 g of sodium hydroxide. Dissolve in water and dilute to 500 mL. Prepare when needed.

4.14 Thiourea-ascorbic acid solution, 50 g/L. Weigh 5.0 g each of thiourea and ascorbic

acid. Dissolve them in water. Dilute to 100 mL. Prepare when needed.

4.15 Arsenic standard stock solution, 100 µg/mL. Accurately weigh 0.1320 g of

standard arsenic trioxide that has been pre-dried at 100°C~105°C for 1 h and cooled to room temperature in a desiccator into a 100 mL beaker. Add 10 mL of sodium hydroxide solution (4.11). Heat at low temperature to dissolve. Add 10 mL of water and 2 drops of phenolphthalein ethanol solution (4.12). Neutralize with hydrochloric acid (4.8) until the red color just disappears. Add another 5 mL in excess. Transfer to a 1000 mL volumetric flask. Dilute to the mark with water. Mix well. 1 mL of this solution contains

4.16 Arsenic standard solution, 1 µg/mL. Take 1.00 mL of arsenic standard stock

solution (4.15) in a 100 mL volumetric flask. Add 5 mL of nitric acid (4.6). Dilute to the mark with water. Shake well. This solution contains 1.00 µg of arsenic in 1 mL.

5.1 Atomic fluorescence spectrometer. equipped with hydride generator and sample

injection device, dedicated arsenic hollow cathode lamp, meeting the requirements of GB/T 21191.

5.7 Pipette. 0.5 mL~5.0 mL, 10 µL~1 000 µL, in accordance with the requirements of

GB/T 12807 and GB/T 12808.

NOTE: The polytetrafluoroethylene beakers and volumetric flasks used in the experiment are soaked in nitric acid (1+9) for more than 24 h, rinsed with water, dried and set aside for later use.

6 Specimen

Prepare chemical analysis specimens with a particle size of less than 63 µm according to GB/T 22564. Dry the specimens in an oven at 105°C±2°C for 2 h. Cool them to room temperature in a desiccator and set aside for later use.