

SY

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

SY/T 7774-2024

**Test method for sulfur isotope of source rock, oil, natural gas
and water**

(English Translation)

Issued on: September 24, 2024

Implemented on: March 24, 2025

Issued by: National Energy Administration

Table of Contents

Foreword...	3
1 Scope...	4
2 Normative references...	4
3 Terms and definitions...	4
4 Reagents and materials...	4
5 Instruments and equipment...	5
6 Sample pretreatment...	5
7 Sulfur isotope determination...	7
8 Precision...	8

1 Scope

This document describes the sample pretreatment and test method for sulfur isotope of source rocks, oil, natural gas, and water.

This document applies to the determination for sulfur isotope of source rocks, oil, natural gas, and water.

2 Normative references

This document has no normative references.

3 Terms and definitions

This document does not contain any terms or definitions that need to be defined.

4 Reagents and materials

4.1

Chromium trichloride. analytically pure.

4.2

Hydrochloric acid. mass fraction is 36%~38%.

4.3

Arsenic-free zinc powder. purity is greater than 99.99%; particle size is 5 μ m~10 μ m.

4.4

Silver nitrate. analytically pure.

4.5

Barium chloride. analytically pure.

4.6

Nitrogen. purity is greater than 99.99%.

4.7

Helium. purity is greater than 99.99%.

4.8

Oxygen. purity is greater than 99.99%.

4.9

Sulfur dioxide. purity is greater than 99.99%.

4.10

Silver sulfide standard sample.

4.11

Centrifuge tube.

4.12

Polytetrafluoroethylene (PTFE) tubing.

4.13

Flat-bottomed flask.

4.14

Erlenmeyer flasks. 250 mL, 500 mL, with two-hole rubber stoppers.

4.15

Condenser.

4.16

pH test paper.

4.17

Filter membrane. cellulose acetate membrane; pore size is 0.45 μm .

4.18

Filter paper. qualitative rapid filter paper.

5 Instruments and equipment

5.1

Isotope mass spectrometer. equipped with a continuous flow interface, it can be connected to an elemental analyzer.

5.2 Elemental analyzer. high-temperature nitriding furnace with a maximum

temperature rise of not less than 1200°C.

5.3 Electromagnetic heating stirrer. the maximum temperature rise is not less than 100°C; the rotation speed is not less than 1200 r/min.

5.4 Drying oven. the maximum temperature is not less than 120°C.

5.5 Centrifuge. the rotation speed is not less than 2000 r/min.

5.6 Analytical balance. the sensitivity is 0.1 mg.

6.1 Pyrite in source rocks transforms into silver sulfide

6.1.1

Weigh at least 40 g of source rock sample. Grind to a particle size no larger than 0.18 mm. Dry in an oven at 100°C for 1 h.

6.1.2

Add 50 mL of silver nitrate solution to the conical flask.

6.1.3

Insert one hole of the two-hole rubber stopper into a PTFE tube filled with nitrogen. Insert the other hole into a condenser tube. Connect the top of the condenser tube to a PTFE tube and immerse it in a silver nitrate solution.

6.1.4

Place the dried source rock sample into a flat-bottomed flask. Then add 400 mL of 1 mol/L chromium trichloride solution.

6.1.5

Fit the flat-bottomed flask with a two-hole rubber stopper. Open the nitrogen valve.

Adjust the nitrogen flow rate until the silver nitrate solution produces a uniform bubbling rate of 1 bubble/s ~ 3 bubbles/s.

6.1.6

The flat-bottomed flask is placed on an electromagnetic stirrer. Heated to 100°C, it is rotated at 1200 r/min. The silver nitrate solution reacts with pyrite to produce hydrogen sulfide. The reaction time is no less than 2 h.

6.1.7

Filter the solution in the conical flask using filter paper. Filter the silver sulfide onto the filter paper. Wash the silver sulfide with deionized water until the test paper shows neutral. Collect the resulting silver sulfide.

6.1.8

Place the silver sulfide in an oven and dry at a temperature not lower than 100°C for 2 h. Store away from light.

6.2 Removal of pyrite from kerogen

6.2.1

Use X-ray diffraction (XRD) to analyze and measure the content of pyrite in kerogen.

6.2.2

If the pyrite content in kerogen is less than 0.5% (mass fraction), sulfur isotope analysis can be performed directly.

6.2.3

If the pyrite content in the kerogen is greater than or equal to 0.5% (mass fraction), the pyrite shall be removed using method 6.1.

6.2.4

Dry the kerogen that has been through pyrite removal in an oven at 100°C for 12 h.

6.3 Conversion of hydrogen sulfide in natural gas into silver sulfide

6.3.1

If the volume percentage of hydrogen sulfide in natural gas is less than or equal to 0.1%, the hydrogen sulfide in the natural gas shall be converted into silver sulfide.

6.3.2

Add 300 mL of silver nitrate solution to a two-hole rubber-stoppered conical flask.

6.3.3

One hole of the rubber stopper is inserted into a PTFE tube. One end of the PTFE tube is connected to a natural gas cylinder via a stainless-steel pressure reducing valve.

The other end is immersed in a silver nitrate solution. The other hole of the rubber stopper