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**Determination of total sulfur in coal
(English Translation)**

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1 Scope

This Document describes the principles, reagents and materials, instruments and equipment, test procedures, result calculation and expression, and precision of the Eschka method, coulometric titration method, infrared spectroscopy, and high-temperature combustion neutralization method for determining total sulfur in coal. The Eschka method is used in arbitration analysis.

This Document applies to dried samples of lignite, bituminous coal, anthracite, coke, and coal- water slurry.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in 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) is applicable to this Document.

GB/T 212 Proximate Analysis of Coal

GB/T 483 General rules for analytical and testing methods of coal

3 Terms and Definitions

For the purposes of this Document, the terms and definitions given in GB/T 483 apply.

4 Coal Samples

General analytical test coal samples with a particle size of less than 0.2 mm and in an air-dried state.

5.1 Principle

The coal sample was mixed with Eschka reagent and burned. The sulfur in the coal was converted into sulfate; and then the sulfate ions were precipitated as barium sulfate. The total sulfur content in the coal was calculated based on the mass of barium sulfate.

5.2 Reagents and materials

Unless otherwise specified, only reagents confirmed to be of analytical pure and distilled or deionized water or water of equivalent purity shall be used in the analysis.

5.2.1

Eschka reagent (hereinafter referred to as Aldrin). Evenly mix 2 portions by weight of chemically pure light magnesium oxide with 1 portion by weight of chemically pure

anhydrous sodium carbonate; grind them into substance with a particle size of less than 0.2 mm; and store in a sealed container. Commercially available Aldrin may also be used.

5.2.2 Hydrochloric acid solution. (1+1) (V+V), 1 volume of hydrochloric acid mixed evenly

with 1 volume of water.

5.2.3 Barium chloride solution. 100 g/L; take 10 g of barium chloride ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$); dissolve in water; and dilute to 100 mL.

5.2.4 Silver nitrate solution. 10 g/L; dissolve 1 g of silver nitrate in 100 mL of water; add 3~4 drops of nitric acid; and store in a brown bottle.

5.2.5 Methyl orange solution. 2 g/L; dissolve 0.2 g of methyl orange in 100 mL of water.

5.2.6 Porcelain crucibles. Capacities of 30 mL and 10~20 mL, respectively.

5.2.7 Filter paper. Medium-speed qualitative filter paper and dense ashless quantitative filter

paper.

5.3 Instruments and equipment

5.3.1

Analytical balance. Graduation value 0.1 mg.

5.3.2

Muffle furnace. Equipped with a temperature control device, capable of heating to 900 °C, temperature adjustable and ventilated.

5.4 Determination

5.4.1

Weigh (1.00 ± 0.01) g (accurate to 0.0002 g) of general analytical test coal sample and 2.0 g (accurate to 0.1 g) of Aldrin into a 30 mL porcelain crucible. Mix carefully and thoroughly;

then cover the top of the mixed coal sample with 1.0 g (accurate to 0.1 g) of Aldrin. Take 0.5 g of coal sample when the total sulfur content is 5%~10%; and take 0.25 g of coal sample when the total sulfur content is greater than 10%.

5.4.2

Transfer the crucible containing the coal sample into a well-ventilated muffle furnace.

Gradually heat the sample from room temperature to 800°C~850°C over 1h ~ 2h; and maintain this temperature for 1h ~ 2h.

5.4.3

Remove the crucible from the muffle furnace and allow it to cool to room temperature.

Carefully loosen and break up (if any unburned particles are found, continue igniting for 30 min) the ignited material in the crucible with a glass rod. Then transfer the ignited material to a 400 mL beaker. Rinse the inside of the crucible with hot water; collect the washings in the beaker; and add 100 mL~150 mL of freshly boiled water; stir thoroughly. If black particles are still floating on the surface of the liquid, then the determination is invalid.

5.4.4 Filter the solution using medium-speed qualitative filter paper via decantation. Rinse 3

times with hot water. Transfer the residue to the filter paper and wash thoroughly with hot water at least 10 times, producing a total washing solution volume of 250mL ~ 300mL.

5.4.5 Add 2 ~ 3 drops of methyl orange indicator to the filtrate. Neutralize with hydrochloric acid solution, adding an excess of 2 mL, to make the solution slightly acidic. Heat the solution to boiling and slowly titrate 10 mL of barium chloride solution while stirring constantly.

Maintain this gentle boiling state for approximately 2 h, until the final solution volume is approximately 200mL.

5.4.6 After cooling or allowing the solution to stand overnight, filter it using dense, ashless quantitative filter paper; and wash with hot water until no chloride ions are present (the solution shall not be turbid when tested with silver nitrate solution).

5.4.7 Transfer the filter paper with precipitate to a known-mass porcelain crucible. After low-temperature ashing of the filter paper, ignite it in a muffle furnace at 800°C~850°C for 20 min

~ 40 min. Remove the crucible; cool it slightly in air; and then place it in a desiccator to cool to room temperature before weighing.

5.5 Blank test

When preparing each batch of Aldrin or changing any other reagent, 2 or more blank tests shall be performed (without adding coal samples, follow 5.4). The mass range of barium sulfate precipitate in the blank tests shall be less than 0.0010 g. Take the arithmetic mean of the blank test results as the blank value.

5.6.1 Calculation of results

The determination results are calculated according to Formula (1).

Where.