

HJ

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Determination of total sulfur in coal. Eschka-Ion chromatography method

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(English Translation)

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Foreword

In order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on the Prevention and Control of Atmospheric Pollution, Protection of human health, standardize the determination of total sulfur content in coal, the development of this standard.

This standard specifies the determination of total sulfur content in coal by escroine-ion chromatography.

This standard is the first release.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting of this standard. Liaoning Province Environmental Monitoring Experimental Center, Environmental Protection Department of Environmental Standards Institute.

The standard verification unit. Dalian Environmental Monitoring Center, Anshan City Environmental Monitoring Center Station, Fushun City Environmental Monitoring Center Station, Yingkou City Environmental Monitoring Center Station, Tieling City Environmental Protection Monitoring Station, Huludao City Environmental Protection Monitoring Center Station.

The Environmental Protection Department of this standard approves on November 20, 2015.

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This standard is explained by the Ministry of Environmental Protection.

Determination of Total Sulfur in Coal - Ash Chromatography - Ion Chromatography

1 Scope of application

This standard specifies the determination of total sulfur content in coal by escroine-ion chromatography.

This standard applies to the determination of total sulfur in anthracite, bituminous coal, lignite and coke.

When the sampling volume is 1.0 g, the calcination volume is 250 ml and the injection volume is 25 µl, the detection limit of this method is 0.02% the lower limit is 0.08%.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

Methods for industrial analysis of coal - Determination of moisture content GB/T Method for preparing GB/T 474 coal samples GB/T 475 Method for artificial extraction of commercial coal samples GB/T.1997 Coke Specimen Preparation and Preparation

3 Principle of the method

The coal sample and Aishi card reagent mixed burning, coal sulfur sulfate, with alkaline eluent into the anion the column was separated in the form of sulfate (SO_4^{2-}) and detected by conductivity detector. According to the quality of sulfate Total sulfur content in coal.

4 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents conforming to national standards are used in the analysis, and the test water is freshly prepared.

Ion water.

4.1

Light Magnesium Oxide (MgO). Analytical purity.

4.2

anhydrous sodium carbonate (Na_2CO_3). analytical grade.

4.3

Aceka Reagent. Mix 2 parts by weight of light magnesium oxide (4.1) with 1 part by mass of anhydrous sodium carbonate (4.2) Grinding to a particle size of less than 0.2 mm, the room temperature stored in a closed dryer.

4.4

Sulfuric acid standard stock solution. $\rho(\text{SO}_4^{2-}) = 1\,000\ \mu\text{g/ml}$.

Weigh 1.814 0 g potassium sulfate ($105 \pm 5\ ^\circ\text{C}$ drying 2 h) dissolved in water, into the 1 000 ml volumetric flask, water volume, Shake well Stored in polyethylene bottles, $0 \sim 4\ ^\circ\text{C}$ frozen storage for 6 months. You can also purchase commercially available standard solutions.

4.5

Sulfuric acid standard liquid. $\rho(\text{SO}_4^{2-}) = 100\ \mu\text{g/ml}$.

Remove 10.00 ml of sulfate standard stock solution (4.4), in 100 ml volumetric flask, with water volume, shake. Stored in Polyethylene bottles, temporary with the current allocation.

4.6

filter paper. the speed of qualitative filter paper.

4.7

Filter. A pore size of $0.45\ \mu\text{m}$ acetate fiber microporous membrane (can be used with the syringe).

4.8

syringes. 10 ml.

4.9

Eluent stock solution. $C(\text{KOH}) = 0.20\ \text{mol/L}$.

Weigh 5.611 g potassium hydroxide ($105 \pm 5\ ^\circ\text{C}$ drying 2 h) dissolved in water, into the 500 ml volumetric flask, water volume, shake uniform. Stored in polyethylene plastic bottles.

4.10

Rinse the use of liquid. $C(\text{KOH}) = 0.02\ \text{mol/L}$.

Remove the eluent stock solution (4.9) 100.00 ml in a 1 000 ml volumetric flask, shake with water, shake.

Note 1: Prepare the eluent according to the instrument type and column conditions. If the instrument model supports automatic generation of eluent, it can be automatically generated.

5 Instruments and equipment

Unless otherwise stated, all Class A glass gauges conforming to national standards are used for analysis.

5.1

ion chromatograph. with conductivity detector.

5.2

Columns. Anion Separation Columns and Anion Protection Columns.

5.3

muffle furnace. with temperature test instrument temperature, can be heated to 900 °C, adjustable temperature and ventilation.

5.4

porcelain crucible. capacity 30 ml.

5.5

Analytical Balance. Accuracy of 0.000 1 g.

5.6

Common laboratory equipment and equipment.

6.1 Collection and storage of samples

In accordance with the relevant provisions of GB/T 475 for coal sample collection, in accordance with the relevant provisions of GB/T.1997 for coke collection.

Samples should be stored in a non-absorbent, airtight sealed container and placed in a cool place.

6.2 Preparation of samples

In accordance with the relevant provisions of GB/T 474 for the preparation of coal samples, in accordance with the relevant provisions of GB/T.1997 coke preparation.

6.3 Preparation of the sample

6.3.1

Weigh coal sample 1 g (nominal to 0.000 2 g), Escherichia reagent (4.3) 2 g (nominal to 0.01 g), placed in 30 ml Porcelain crucible (5.4) after mixing, and then 1 g (called quasi to 0.01 g) Eskka reagent (4.3) covered.