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Replacing GB/T 16415-1996

Determination of selenium in coal - Hydride generation-atomic absorption method

煤中硒的测定方法 氢化物发生原子吸收法

(ISO 11723:2004, Solid mineral fuels - Determination of arsenic and selenium - Eschka's mixture and hydride generation method, MOD)

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Standardization Administration of PRC.

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Determination of selenium in coal - Hydride generation-atomic absorption method

1 Scope

This standard specifies the reagents and materials, instruments and equipment, analytical procedures, result calculation, method precision and test report for the determination of selenium in coal.

This standard applies to lignite, bituminous coal and anthracite.

2 Normative references

The clauses in the following documents become clauses of this standard through reference in this standard. For dated referenced documents, all subsequent amendments (excluding errata) or revisions are not applicable to this standard. However, parties who reach an agreement based on this standard are encouraged to investigate whether the latest versions of these documents are applicable. For undated referenced documents, the latest versions apply to this standard.

GB/T 483 General rules for analytical and testing methods of coal (GB/T 483-2007, ISO 1213-2:1992, Solid mineral fuels - Vocabulary - Part 2: Terms relating to sampling, testing and analysis, NEQ)

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696:1987, Water for analytical use - Specification and test methods, MOD)

3 Method summary

The coal sample is mixed with Aldrin, burned at 800 °C, dissolved with hydrochloric acid, and heated to reduce hexavalent selenium to tetravalent selenium. Then, sodium borohydride is used to reduce tetravalent selenium to selenium hydride. The mixture is introduced into a quartz tube atomizer using nitrogen as a carrier gas and determined by atomic absorption method.

4 Reagents and materials

The water used in this standard shall comply with the requirements for Grade 3 water

in GB/T 6682.

4.1 Aldrin: Take 2 parts by mass of light calcined magnesium oxide and 1 part by mass of anhydrous sodium carbonate, grind to a particle size of less than 0.2 mm, mix well, and store in a sealed container.

4.2 Hydrochloric acid: with a relative density of 1.18.

4.3 Sodium borohydride solution: 18 g/L. Weigh 1.8 g of sodium borohydride and dissolve it in 100 mL of 5 g/L sodium hydroxide solution. Prepare the solution before use.

4.4 Selenium standard stock solution: 1 mg/mL. Weigh 0.1000 g of high-purity selenium into a 100 mL beaker, add 5 mL of nitric acid, heat at low temperature until dissolved, continue heating to drive out nitrogen oxides, cool, transfer to a 100 mL volumetric flask, dilute to the mark with water, and shake well.

Commercially available certified reference materials can also be used as selenium standard stock solutions.

4.5 Selenium standard intermediate solution: 10 μg/mL. Pipette 1 mL of the selenium standard stock solution (4.4) into a 100 mL volumetric flask, dilute to the mark with the blank solution (6.2.2), and shake well.

4.6 Selenium standard working solution: 0.2 μg/mL. Pipette 1 mL of the selenium standard intermediate solution (4.5) into a 50 mL volumetric flask, dilute to the mark with the blank solution (6.2.2), and shake well.

4.7 Nitrogen: The purity is above 99.9%.

4.8 Acetylene: High-purity acetylene.

5 Instruments and equipment

5.1 Analytical balance: The sensitivity is 0.1 mg.

5.2 Porcelain crucible: 30 mL. The inner surface of the enamel shall be intact.

5.3 Muffle furnace: The temperature can be controlled at 800 °C±20 °C; the ventilation is good.

5.4 Atomic absorption spectrophotometer: with absorption peak area integration and peak height measurement functions.

5.5 Light source: Selenium hollow cathode lamp or selenium electrodeless discharge lamp.

5.6 Automatic hydride generator: It is capable of automatic washing, liquid metering and liquid addition, with an accuracy of 0.5%.

5.7 Electric heating plate: It can maintain the temperature at 60 °C~90 °C.

6 Analysis steps

6.1 Preparation of sample solution

6.1.1 Accurately weigh 1 g of air-dried coal sample with a particle size of less than 0.2 mm (accurate to 0.0002 g) (when A_d of the coal sample is greater than 40.00% or $S_{t,d}$ is greater than 8.00%, or Se_d is greater than 15 μg/g, weigh 0.5 g of coal sample), place it in a porcelain crucible pre-filled with 1.5 g of Aldrin, mix the coal sample and aldrin evenly, and then evenly cover it with about 1.5 g of Aldrin.

6.1.2 Place the crucible in a cold muffle furnace, slowly raise the temperature to 500 °C and heat at this temperature for 1 hour. Then raise the temperature to 800 °C and heat at this temperature for another 3 hours. Remove the crucible and cool to room temperature.

6.1.3 Crush the calcined sample and transfer it to a 150 mL beaker containing 20~30 mL of hot water. Add 5 mL of hydrochloric acid to the crucible to dissolve the remaining residue, then pour it into the beaker. Wash the crucible with 15 mL of hydrochloric acid three times (5 mL each time), and transfer the wash solution to the beaker. Stir the solution. After cooling, transfer the entire solution to a 100 mL volumetric flask, dilute to the mark with water, and shake well.

NOTE: Some samples may contain solid residue, but it will not affect the test results.

6.2 Preparation of blank solution

6.2.1 Weigh 15 g of aldrin into a 100 mL evaporating dish. Place the dish in a cold muffle furnace, slowly raise the temperature to 500 °C, and heat at this temperature for 1 hour. Then raise the temperature to 800 °C and continue heating for 3 hours. Remove the evaporating dish and cool to room temperature.

6.2.2 Transfer the ignited aldrin to a beaker containing 100~150 mL of hot water. Dissolve the residue in the dish with 25 mL of hydrochloric acid, transfer it to the beaker, and rinse the residue into the beaker with water. Rinse the evaporating dish with 75 mL of hydrochloric acid three times (25 mL each time) and transfer the solution to the beaker. Stir until the aldrin is completely dissolved. Cool to room temperature, transfer to a 500 mL volumetric flask, dilute to the mark with water, and shake well. Transfer it to a plastic bottle for storage.

6.3 Preparation of standard solutions

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