GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 73.040
D 21

GB/T 19226-2003

Determination of vanadium in coal

煤中钒的测定方法

Issued on: July 1, 2003 **Implemented on: November 1, 2003**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China

Table of Contents

Determination of vanadium in coal

1 Scope

This Standard specifies the method abstract, reagents and materials, instrument and apparatus, test procedures, calculation of results and method's precision for the determination of vanadium in coal.

This Standard is applicable to lignite, bituminous coal and anthracite.

2 Normative References

The provisions in the following documents become part of this Standard, through reference in this Standard. For the dated documents, the subsequent amendments (excluding corrections) or revisions shall not apply to this Standard. However, all parties who enter into agreement according to this Standard are encouraged to study if the latest versions of these documents are applicable. For undated documents, the latest version applies to this Standard.

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

3 Method Abstract

Perform ashing for the coal samples, then melt it with alkali and leach it with boiling water. Add masking agents into the leachate to eliminate the influence of interfering elements. In phosphoric acid medium -- pentavalent vanadium, 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (Br-PADAP) and hydrogen peroxide FORM the colored ternary complex; then, it shall perform the photometric measurement to determine the content of vanadium.

4 Reagents and Materials

4.1 Sodium peroxide: granular.

4.2 Sodium hydroxide (GB 629): granular.

4.3 Sodium hydroxide solution: c(NaOH)=1 mol/L.

Weigh and take 40 g of sodium hydroxide; dissolve it in the water of 1L.

4.4 Sulfuric acid solution: c ($1/2H_2SO_4$) = 1 mol/L.

6.2.1 Ashing of coal samples

Accurately weigh and take 1 g of air-dried coal samples of which the particle size is less than 0.2 mm (the accuracy of weigh shall be 0.0002 g); put it into the crucible (5.3); place it into the muffle furnace (5.1); heat it gradually from room temperature to (600±10°C); burn it at this temperature for at least 1 h until there is no black carbon particles; remove the crucible and cool it to room temperature.

Note: The amount of sample can be appropriately increased or decreased according to the content of vanadium.

6.2.2 Treatment of ash samples

In corundum crucible which contains ash sample, add 4 g of sodium hydroxide (4.2) and 1 g of sodium peroxide (4.1); place it in the muffle furnace. Under the condition of ensuring that the melt sample does not splatter or spill, gradually heat it from the room temperature until (650 ± 10) °C. Under this temperature, melt it for 10 min ~ 15 min; remove the crucible and cool it to room temperature.

Place the crucible in the beaker (5.6); add 50 mL ~ 60 mL of boiling water; boil for 1 min to 2 min on the electric furnace (5.8). Remove the beaker; wash it with hot water. Transfer the solution to 100 mL volumetric flask (5.5); cool it to room temperature; dilute it with water until the scale, shake it evenly; place it for overnight or use qualitative filter paper (4.13) to perform dry filtration.

Note: Dry filtration refers to the filtration under the condition that the using instruments and materials (e.g.: funnels, beakers and filter paper, etc.) are both dry and clean.

6.2.3 Preparation of sample blank solution

When decomposing a batch of samples, it shall prepare a sample blank solution at the same time. The preparation method shall be the same as 6.2.2, but the ash sample shall not be added.

6.2.4 Determination

Accurately pipette 10 mL of the upper clear solution or filtrate of the sample AND 10 mL of the sample blank solution (6.2.3); respectively place them into 50 mL volumetric flask; add 5 mL of mixed masking agent and 2 drops of p-nitrophenol indicator; use sulfuric acid solution (4.5) to carefully adjust it from yellow to colorless. Follow 6.1.2 and 6.1.3, except that – use sample blank solution as the reference to determine the absorbance of the sample solution. Find-obtain the content of vanadium pentoxide (μg) in sample solution from working curve.

Note: According to the content of vanadium in sample's total solution, the amount of liquid absorption can be appropriately increased or decreased.

This is an excerpt of the PDF (Some pages are marked off intentionally)

------ The End ------