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NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 216-2003

Replacing GB/T 216-1996

Methods for determination of phosphorus content in coal

(ISO 622: 1981, Solid mineral fuels - Determination of phosphorus content –

Reduced molybdophosphate photometric method, NEQ)

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Foreword

This Standard corresponds to ISO 622:1981 *Solid mineral fuels - Determination of phosphorus content – Reduced molybdophosphate photometric method* (English edition).

This Standard is not equivalent to ISO 622. The main differences between this Standard and ISO 622 are as follows:

- Delete the wet oxidation process in decomposition method of samples. Only adopt dry oxidation process.
- Add method “directly measure the mass of coal sample, then re-ashing”.

This Standard replaces GB/T 216-1996 *Methods for determination of phosphorus content in coal*.

Compared with GB/T 216-1996, main changes of this Standard are as follows:

- Delete Appendix A “Ash decomposition methods for hydrochloric - perchlorate” (1996-edition).

This Standard was proposed by China National Coal Association.

This Standard shall be under the jurisdiction of National Technical Committee on Coal of Standardization of China.

Drafting organizations of this Standard: China Coal Research Institute-Coal analysis laboratory, and Team 143 of Yunnan Coalfield Exploration Company.

Main drafters of this Standard: Zhang Kerui, and Ma Zunmei.

This Standard replaces the following historical editions:

GB 216-63; GB 216-82; GB/T 216-1996.

Methods for determination of phosphorus content in coal

1 Scope

This Standard specifies the abstract of methods, reagents, instruments and equipment, determination procedure, result expression and precision of determination of phosphorus content in coal.

This Standard is applicable to determination of phosphorus content in lignite, bituminous coal, anthracite and coking coal.

2 Normative references

The articles contained in the following documents have become part of this Standard when they are quoted herein. For the dated documents so quoted, all the modifications (excluding corrections) or revisions made thereafter shall not be applicable to this Standard. For the undated documents so quoted, the latest editions shall be applicable to this Standard.

GB/T 212 Proximate analysis of coal (GB/T 212-2001, eqv ISO 1171:1997)

3 Abstract of methods

After ashing of the coal sample, decompose it with hydrofluoric acid-sulfuric acid. Remove silicon dioxide. Then add ammonium molybdate and ascorbic acid to generate phosphorus molybdenum blue. Determine the absorbance with spectrophotometer.

4 Reagents

4.1 Hydrofluoric acid (GB/T 620): 40% (mass fraction).

4.2 Sulfuric acid solution: $c(1/2H_2SO_4)=10\text{mol/L}$. Measure 278mL of concentrated sulfuric acid (GB/T 625). Put them into appropriate amount of water slowly. Stir it while adding. Then dilute with water to 1000mL.

4.3 Sulfuric acid solution: $c(1/2H_2SO_4)=7.2\text{mol/L}$. Measure 200mL of concentrated sulfuric acid. Put them into appropriate amount of water slowly. Stir it while adding. Then dilute with water to 1000mL.

A_{ad} — The ash content of air-dried coal sample, %.

6.2 Method B (weighing coal sample)

6.2.1 Sample treatment

6.2.1.1 Ashing of coal sample: Accurately measure 1g~0.5g of air-dried coal sample with particle size less than 0.2mm (ash content is around 0.05g~0.1g). Put it in an ash-pan, accurate to 0.0002g. Shake it gently to spread it evenly. Then put it into muffle. Half-open the furnace door. Raise the temperature from room temperature to $(815 \pm 10)^\circ\text{C}$ slowly. Keep burning at this temperature for at least 1h until there is no material containing carbon.

6.2.1.2 Acidolysis of ash: Transfer all ashing samples (6.2.1.1) into teflon or platinum crucible. Carry out acidolysis according to the provisions in 6.1.1.2.

6.2.2 Preparation of blank solution: the same as 6.1.2.

6.2.3 Determination procedure: the same as 6.1.3.

6.2.4 Result calculation

The mass fraction of phosphorus P_{ad} (%) in air-dried coal sample is calculated according to Formula (2):

$$P_{ad} = \frac{10m_1}{mV} \dots\dots\dots (2)$$

Where:

m_1 — The phosphorus content in separate test solution referred in working curve, mg;

V — The volume of test solution separated from total test solution, mL;

m — The mass of air-dried coal sample, g.

7 Precision

The precision of determination of phosphorus is specified in Table 1:

Table 1 Precision of determination of phosphorus

Mass fraction of phosphorus/%	Repeatability limit (P_{ad})/%	Critical difference of reproducibility (P_d)/%
<0.02	0.002 (absolute)	0.004 (absolute)
≥ 0.02	10% (relative)	20% (relative)

8 Test report

The test report shall include the following information:

- a) Sample No.;
- b) Test Standards;
- c) Methods used;
- d) Result calculation;
- e) Deviation from standard;
- f) Abnormalities observed in test;
- g) Test date.

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