

ICS 73.040
CCS D 21

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**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 5256-2020

**Determination of phosphorus content in coal microwave
ashing-spectrophotometric method**

煤炭中磷含量的测定 微波灰化-分光光度法

(English Translation)

Issued on: August 27, 2020

Implemented on: March 1, 2021

Issued by: General Administration of Customs

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was proposed and managed by the General Administration of Customs of the People's Republic of China.

Drafting organization of this standard. Taiyuan Customs of the People's Republic of China.

1 Scope

This standard specifies the microwave ashing-spectrophotometric method to determine the phosphorus content in coal.

This standard applies to the determination of phosphorus content in lignite, bituminous coal, anthracite and coke. The measurement range (mass fraction) is 0.005%~0.7%.

2 Normative references

The following documents are indispensable for the application of this document. For dated reference documents, only the dated version applies to this article Pieces. For undated reference documents, the latest version (including all amendments) is applicable to this document.

GB/T 212 Coal Industrial Analysis Method GB/T 474 Coal sample preparation method
GB/T 1274 Chemical reagent potassium dihydrogen phosphate GB/T 6379.2 Accuracy of
measurement methods and results (accuracy and precision) Part 2 Determine the
repeatability of standard measurement methods Basic methods of sex and reproducibility
GB/T 6682 Analytical laboratory water specifications and test methods

3 Method summary

After microwave ashing, the coal sample is decomposed with hydrofluoric acid-sulfuric acid to remove silicon dioxide, and ammonium molybdate, potassium antimony tartrate and ascorbic acid are added.

The mixed solution produces phosphomolybdenum blue, and the absorbance is measured with a spectrophotometer, and the standard curve method is used for quantification.

4 Reagents and materials

Unless otherwise specified, the reagents used in this standard are of analytical grade, and the test water is the secondary water specified in GB/T 6682.

4.1

Hydrofluoric acid. $\rho(\text{HF})=1.15 \text{ g/mL}$, analytically pure.

4.2

Sulfuric acid solution. $c(12 \text{ H}_2\text{SO}_4) = 10 \text{ mol/L}$, measure 278 mL of concentrated sulfuric acid, slowly add an appropriate amount of water, stir while adding, and then use the volume of water is adjusted to 1 000 mL.

4.3

Sulfuric acid solution. 14.Measure.200 mL of concentrated sulfuric acid and slowly add an appropriate amount of water, stirring while adding, and then dilute the volume to 1 000 mL with water.

4.4

Ammonium molybdate-sulfuric acid solution. Dissolve 17.2 g of ammonium molybdate in an appropriate amount of sulfuric acid solution (4.3), and dilute with sulfuric acid solution (4.3) to 1 000 mL.

4.5

Ascorbic acid solution. Weigh 5 g of ascorbic acid, dissolve it in 1,000 mL of water, and make it ready for use.

4.6

Potassium antimony tartrate solution. Weigh 0.34 g of potassium antimony tartrate and dissolve it in 250 mL of water.

4.7

Mixed solution. add 10 mL of ascorbic acid solution (4.5) and 5 mL of potassium antimony tartrate to 35 mL of ammonium molybdate-sulfuric acid solution (4.4) Solution (4.6), mix well and prepare before use.

4.8

Phosphorus standard stock solution (0.1 mg/mL). accurately weigh the premium grade pure potassium dihydrogen phosphate dried at 110 °C for 1 h (see GB/T 1274) 0.439 2 g is dissolved in water and diluted with water to 1000 mL. A solution of certified reference materials can also be used.

4.9

Phosphorus standard series working solution. Draw the phosphorus standard stock solution (4.8) 0 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL in a 100 mL volumetric flask, dilute to the mark with water, the phosphorus content of the standard series of working solutions are 0.00 mg/L, 1.00 mg/L, 2.00 mg/L, 3.00 mg/L, 4.00 mg/L, ready to use.

5 Apparatus and equipment

5.1

Analytical balance. Sensitivity 0.1 mg.

5.2

Microwave ashing furnace. with program temperature adjustment device, can maintain the temperature (815±10) °C.

5.3

Spectrophotometer.

5.4

Electric heating plate. adjustable temperature.

5.5

Porcelain boat or quartz crucible. Porcelain boat, 45 mmx22 mmx14 mm; quartz crucible, 35 mmx15 mm or equivalent.

5.6

Platinum or PTFE crucible. the capacity is 25 mL~30 mL.

6.1 Sample preparation

According to GB/T 474, prepare a general analysis test coal sample with a particle size less than 0.2 mm.

6.2 Sample preparation

6.2.1

Microwave ashing. take an appropriate amount of coal sample and set the microwave ashing furnace program according to the program shown in Table 1 (it can be appropriate according to different microwave ashing furnaces).

Adjust the ashing procedure), until the sample is completely ashed, grind the fine ash sample until all the ash samples pass through a 0.1 mm sieve for use.

Table 1 Microwave ashing procedure

No.	Heating time/min	Power/W	Set temperature/°C	Holding time/min
1	30	1200	500	5
2	20	1200	815	10

6.2.2

Acid hydrolysis of ash. accurately weigh the above ash sample (6.2.1) 0.05 g~0.1 g (accurate to 0.0002 g) in a platinum or PTFE crucible (5.6) Add 2 mL of sulfuric acid (4.2) and 5 mL of hydrofluoric acid (4.1). Place it on a hot plate and slowly heat and evaporate (temperature is about 100 °C) until the hydrofluoric acid the sour white smoke is exhausted. Cool, add 0.5 mL of sulfuric acid (4.2), increase the temperature and continue heating to evaporate until the sulfuric acid emits white smoke (but do not dry up).

Cool, add a few drops of cold water and shake, then add 20 mL of hot water and continue heating to near boiling. Wash the contents of the crucible into a 100 mL volume with water.

Wash the crucible in a measuring flask, cool to room temperature, dilute to the mark with water, mix well, clarify or filter for later use.

6.3.1 Working curve

Respectively draw 10 mL of phosphorus standard series working solution (4.9) into a 50 mL volumetric flask, add 5 mL of mixed solution (4.7), Dilute with water to the mark, mix well, put it at room temperature (above 10 °C) for 1 h, and then transfer it into a 10 mm cuvette. In the light splitting on the meter, use a wavelength of 650 nm and use the standard blank solution as a reference to measure its absorbance. Take the phosphorus content as the abscissa and the absorbance as the vertical axis Plot the working curve.