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Coke - Determination of Phosphorus Content - Reduced Molybdophosphate Spectrophotometric Method

焦炭 磷含量的测定

还原磷钼酸盐分光光度法

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Warning -- personnel who use this Standard shall have practical experience of working at a regular laboratory. This Standard does not point out all the possible safety questions. Users shall undertake the responsibility of adopting proper safety and health measures and guaranteeing the compliance with the conditions of relevant national laws and regulations.

1 Scope

This Standard specifies the determination of phosphorus content through reduced Molybdophosphate spectrophotometric method.

This Standard is applicable to the determination of phosphorus content in coke (metallurgical coke, foundry coke, ferroalloy coke, gasification coke and semi-coke, etc.). Coal for coking can take this as a reference. The range of determination (mass fraction): 0.0050% ~ 0.10%.

2 Normative References

The following documents are indispensable to the application of this Standard. In terms of references with a specified date, only versions with a specified date are applicable to this Standard. The latest version (including all the modifications) of references without a specified date is also applicable to this Standard.

GB/T 1997 Coke - Sampling and Preparation of Samples

GB/T 2001 Coke - Determination of Proximate Analysis

GB/T 6379.2 Accuracy (trueness and precision) of Measurement Methods and Results - Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 7729 Chemical Analysis of Metallurgical Products, General Rule for Spectrophotometric Methods

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

3 Principle

After ashing sample, use acid to dissolve the sample, which will emit perchloric acid smoke; completely oxidize phosphorus into orthophosphoric acid. Under the existence of bismuth salt, add ammonium molybdate to generate phosphorus molybdenum bismuth ternary heteropoly acid. Take ethanol as the stabilizer; use ascorbic acid to reduce into phosphorus bismuth molybdenum blue; measure its absorbance.

4 Reagents and Materials

Unless it is otherwise stated, analytical purity reagents that are acknowledged, and above Level-3 distilled water that complies with the stipulation in GB/T 6682 or water of equivalent purity shall be merely adopted in analysis.

4.1 Nitric acid, $\rho \approx 1.42$ g/mL.

4.2 Hydrofluoric acid, $\rho \approx 1.15$ g/mL.

4.3 Perchloric acid, $\rho \approx 1.67$ g/mL.

4.4 Sodium nitrite solution, 100 g/L.

4.5 Boric acid-anhydrous sodium carbonate flux, 1+2.

4.6 Sulfuric acid solution, 1+1.

4.7 Nitric acid-bismuth nitrate solution, 10 g/L. Use nitric acid (1+3) to prepare it.

4.8 Sodium thiosulfate-anhydrous sodium sulfite solution; 100 mL of solution contains 0.2 g of sodium thiosulfate and 1 g of anhydrous sodium sulfite solution.

4.9 Ascorbic acid ethanol solution, 20 g/L. Weigh -take 20 g of ascorbic acid; dissolve it in 1 L of ethanol solution (1+1).

4.10 Ammonium molybdate-potassium sodium tartrate solution; 100 mL of solution contains 3.5 g of ammonium molybdate and 6.5 g of potassium sodium tartrate.

4.11 Phosphorus standard solution: weigh-take 0.4394 g of potassium dihydrogen phosphate that is previously dried at 105 °C to a constant weight. Use a proper amount of water to dissolve it; add 10 mL of nitric acid, then, transfer it into a 1,000 mL volumetric flask. Use water to dilute to the constant volume, then, mix it up. Transfer 25.00 mL into a 500 mL volumetric flask; use water to dilute to the constant volume, then, mix it up. 1 mL of this solution contains 5.0 μg of phosphorus.

7.3.2 Alkali melting decomposition method

Add the sample (please refer to 7.1) to a platinum crucible, which already has 3 g ~ 4 g of boric acid-anhydrous sodium carbonate flux (please refer to 4.5); mix it up. Then, cover it with 2 g ~ 3 g of boric acid-anhydrous sodium carbonate flux (please refer to 4.5). Place it into a 950 °C ~ 1,000 °C Muffle furnace; start high-temperature melting for 10 min ~ 15 min. Take it down to cool it down, then, use water to rinse the external wall of the platinum crucible. Place the platinum crucible into a 250 mL beaker which holds 50 mL of water. Take 10 mL of sulfuric acid solution (please refer to 4.6), then, heat it up, start leaching; clean the crucible. Continue the heating, till it boils, then, cool it down to room temperature. Transfer it into a 100 mL volumetric flask; use water to dilute to the constant volume, then, mix it up.

7.4 Color Development and Determination

Divide-take 10.00 mL of solution (please refer to 7.3), then, place it into a 50 mL volumetric flask. Add 15 mL of nitric acid-bismuth nitrate solution (please refer to 4.7), 5 mL of sodium thiosulfate-anhydrous sodium sulfite solution (please refer to 4.8), 10 mL of ascorbic acid ethanol solution (please refer to 4.9) and 5 mL of ammonium molybdate-potassium sodium tartrate solution (please refer to 4.10). After placing it for 1 min ~ 2 min, use water to dilute to the constant volume, then, mix it up. Place it at room temperature for over 15 min. On a spectrophotometer, use a 1 cm ~ 3 cm colorimetric cuvette; at the wavelength of 700 nm, take blank test solution as a reference. Measure the absorbance of the color developing solution; obtain corresponding phosphorus content through the calibration curve.

NOTE: after adding various reagents in turn, thoroughly mix it up.

7.5 Draw a Calibration Curve

Respectively transfer-take 0 mL, 0.50 mL, 1.00 mL, 2.00 mL, 4.00 mL, 6.00 mL and 8.00 mL of phosphorus standard solution (please refer to 4.11); place it in a group of 50 mL volumetric flasks; comply with the operation (in 7.4). Take phosphorus-free standard solution as a reference; take the measured absorbance as the y-coordinate; take the mass of phosphorus in the standard series solution as the x-coordinate to draw a calibration curve.

8 Analysis Result Calculation and Expression

8.1 Calculation of Analysis Result

In accordance with Formula (1), calculate the content of phosphorus in coke; calculate by mass fraction; the numerical value shall be expressed in %.

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