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Replacing GB/T 6730.18-1986

**Iron ores - Determination of phosphorus content -
Molybdenum blue spectrophotometric method**

铁矿石 磷含量的测定 钼蓝分光光度法

(ISO 4687-1:1992, MOD)

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Iron ores - Determination of phosphorus content - Molybdenum blue spectrophotometric method

WARNING - Persons using this Part of GB/T 6730 shall have practical experience in formal laboratory work. This Part does not address all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This Part of GB/T 6730 specifies the molybdenum blue spectrophotometric method for the determination of phosphorus content.

This method is applicable to the determination of phosphorus content in natural iron ores, iron concentrates, agglomerates, including sintered products. The presence of arsenic, barium, titanium does not affect the determination results. The determination range (mass fraction) is 0.003% ~ 2.00%.

2 Normative references

The provisions in following documents become the provisions of this Part of GB/T 6730 through reference in this Part. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Part; however, parties who reach an agreement based on this Part are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682 Water for laboratory use - Specifications (GB/T 6682-1992, neq ISO 3696:1987)

GB/T 6730.1 Methods for chemical analysis of iron ores - Preparation of pre-dried test samples for chemical analysis (GB/T 6730.1-1986, eqv ISO 7764:1985)

GB/T 10322.1 Iron ores - Sampling and sample preparation procedures (GB/T 10322.1-2000, idt ISO 3082:1998)

GB/T 12806 Laboratory glassware - One-mark volumetric flasks (GB/T 12806-1991, neq ISO 1042:1983)

GB/T 12808 Laboratory glassware - One-mark pipettes (GB/T 12808-1991, neq ISO

4.7 Sodium sulfite solution, 100 g/L.

The solution must be prepared immediately for use; a sufficient amount is prepared at one time, according to the number of tests to be done.

4.8 Hydrazine sulfate solution, 2 g/L.

The solution must be prepared immediately for use; a sufficient amount is prepared at one time, according to the number of tests to be done.

4.9 Bottom fluid

Weigh 1.6 g of sodium carbonate (4.1) and 0.8 g of sodium tetraborate (4.2), into a 250 mL beaker. Add 40 mL of water. Carefully add 70 mL of hydrochloric acid (4.4), while stirring it continuously. Heat and boil for 1 min. After cooling, transfer to a 200 mL one-mark volumetric flask. Use water to dilute to the mark. Mix well.

4.10 Ammonium molybdate solution, 20 g/L.

Dissolve 20 g of ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 500 mL of water. After dissolution, slowly add 250 mL of 72% (mass fraction) perchloric acid or 325 mL of 60% (mass fraction) perchloric acid (4.5). After mixing and cooling, transfer to a 1000 mL one-mark volumetric flask. Use water to dilute it to the mark.

4.11 Phosphorus standard solution, 20 $\mu\text{g/mL}$.

Dry the standard potassium dihydrogen phosphate (99.99%) at 110 °C to a constant weight. Put it in a desiccator, to cool it to room temperature. Weigh 0.2197 g and dissolve in water. Transfer to a 250 mL one-mark volumetric flask. Use water to dilute to the mark. Mix well. Then pipette 25.0 mL of this solution into a 250 mL one-mark volumetric flask. Use water to dilute to the mark. Mix well.

5 Instrumentation

One-mark pipettes and one-mark volumetric flasks shall comply with the provisions of GB/T 12806 and GB/T 12808, respectively.

General laboratory instruments and:

5.1 Platinum or platinum alloy crucible, the volume of which is not less than 25 mL.

5.2 Muffle furnace, which can provide a temperature of 1020 °C.

5.3 Heating plate with magnetic stirring function.

5.4 Stirring rod, which is sealed with polytetrafluoroethylene plastic, 1.0 cm long.

5.5 Water bath device, which can be used at boiling temperature.

5.6 Spectrophotometer, which is suitable for measuring absorbance at 820 nm.

6 Sampling and specimen preparation

6.1 Laboratory samples

Take samples and prepare specimen, according to GB/T 10322.1. Generally, the specimen particle size shall be less than 100 μm . If the content of hydrated water or easily oxidized substances in the specimen is high, the particle size shall be less than 160 μm .

Note 1: Please refer to GB/T 6730.1, for specific instructions on hygroscopic moisture or easily oxidized compounds.

6.2 Preparation of pre-dried specimens

Mix the laboratory samples thoroughly. Take samples by fractional division method. According to the requirements in GB/T 6730.1, dry the specimen at a temperature of $105\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

7 Analytical procedures

7.1 Number of measurements

According to Appendix A, for the same pre-dried specimen, at least 2 independent measurements are carried out.

Note 2: The term "independent" means that the results of any repeated and subsequent determinations are not affected by the results of previous determinations. In this analytical method, this condition implies repeated determinations by the same operator at different times or by different operators, including with appropriate recalibration.

7.2 Sample

Weigh several parts of about 0.50 g pre-dried specimens (6.2), accurate to 0.0002 g.

Note 3: The sample shall be weighed quickly, to avoid moisture absorption again.

7.3 Blank test and calibration test

For each test, a blank test shall be done in parallel with the specimen under the same conditions. A standard sample similar to the specimen shall be analyzed at the same time. The standard samples used must also be dried, according to Clause 6.2.

Note 4: The standard sample used shall be of the same type as the specimen to be analyzed. The components of the standard sample shall be as close as possible to the specimen, to ensure that the analysis process does not change significantly.

When analyzing several samples at the same time, if the operation process is the same and the reagents used are all from the same reagent bottle, a blank can be shared.

When analyzing several samples of the same type at the same time, only one standard sample can be analyzed.

7.4 Determination

7.4.1 Decomposition of samples

Add 0.8 g of sodium carbonate (4.1) to platinum or platinum alloy crucible (5.1). Add the weighed sample (7.2). Use a platinum rod or stainless steel rod, to mix thoroughly. Then add 0.4 g of sodium tetraborate (4.2). Then use a metal rod to mix well. Put the crucible into the muffle furnace (5.2), to melt at 1020 °C for 30 min. Take out the crucible. Turn it gently, until the melt solidifies. Cool down.

7.4.2 Preparation of test solution

Put the stirring rod (5.4), which is sealed with polytetrafluoroethylene plastic, into the cooled crucible. Then place the crucible, obliquely into a 150 mL low-profile beaker. Add 25 mL of water and 35 mL of hydrochloric acid (4.4). Cover with a watch glass. Heat on a heating plate (5.3), which has magnetic stirring function, until the melt is completely dissolved.

Note 5: The crucible needs to be rotated during the heating process, to ensure that the melt can be immersed in the solution.

After cooling down, take out the crucible and wash it. Transfer it into a 100 mL one-mark volumetric flask. Use water to dilute to the mark. Mix well (this is the test solution).

7.4.3 If the phosphorus content in the specimen is less than 0.2% (mass fraction), proceed according to 7.4.3.1.

If the phosphorus content in the specimen is greater than or equal to 0.2% (mass fraction), proceed according to 7.4.3.2.

7.4.3.1 The phosphorus content is less than 0.2% (mass fraction)

Pipette equal volumes of test solution (7.4.2) and blank test solution, according to Table 2. Place them in two 250 mL tall beakers, respectively. Add corresponding volume of bottom solution (4.9) (see Table 2).

Pipette an equal volume of the test solution (7.4.2), according to Table 2. Put it in a 250

Add 2.0 mL of hydrazine sulfate solution (4.8). Mix well. Transfer to the boiling water bath (5.5). Make the liquid level in the beaker submerged below the water surface of the water bath. Keep it boiling for 20 min. Take it out. Cool it to room temperature under running water. Then transfer it into 100 mL one-mark volumetric flask. Use water to dilute to the mark. Mix well (Note 8).

Using water as a reference, to measure the absorbance of the solution, using a 1 cm cuvette, near the absorption peak at 820 nm. Subtract the absorbance of the corresponding blank solution or diluted blank solution, to obtain the absorbance of the test solution or diluted test solution (Note 9). If an iron compensation test is performed, the absorbance of the iron compensation solution shall also be subtracted, from the absorbance of the test solution.

Note 8: It has been proved that the solution is stable for at least 5 h, after color development.

Note 9: If the absorbance of the blank solution is greater than 0.025, a reagent with higher purity shall be used instead.

7.4.5 Preparation of calibration curve

7.4.5.1 Preparation of phosphorus standard spare calibration solution, 5 µg/mL.

Weigh 0.8 g of sodium carbonate (4.1) and 0.4 g of sodium tetraborate (4.2), into a 250 mL beaker. Add 30 mL of water. Carefully add 35 mL of hydrochloric acid (4.4), under constant stirring. Cover with a watch glass. Heat and boil for 1 min. After cooling, add 25.0 mL of phosphorus standard solution (4.11). Mix well. Transfer to a 100 mL one-mark volumetric flask. Use water to dilute to the mark. Mix well.

7.4.5.2 Calibration curve

Add 0 mL, 5.0 mL, 10.0 mL, 20.0 mL of phosphorus standard spare calibration solution (7.4.5.1), into four 250 mL tall beakers, respectively. Then add 20.0 mL, 15.0 mL, 10.0 mL of bottom solution (4.9), respectively, to the first three beakers. The subsequent operations follow 7.4.4.

Using water as a reference, to measure the absorbance of the solution, using a 1 cm cuvette near the absorption peak at 820 nm. Subtract the absorbance of 0 mL of phosphorus solution, to obtain the net absorbance of the standard solution. Draw a calibration curve, using micrograms of phosphorus as the abscissa and net absorbance as the ordinate.

Note 10: The expected absorbance of 100 µg phosphorus is 0.870 ± 0.03 .

8 Calculation of results

8.1 Calculation of phosphorus content

arithmetic mean of the final results (8.2.5) of the two laboratories;

σ_d - The standard deviation of repeated determinations within the laboratory;

σ_L - The standard deviation between laboratories;

R_d - The tolerance for repeated determinations within the laboratory (repeatability);

P - Tolerance between laboratories.

Note 12: The minimum value refers to the actual value, which is obtained by measuring the specimen, which has the lowest content during the international test. When the specimen with a very low level of content is measured AND the linear regression equation is used for calculation, negative values may be generated. In this case, use the minimum value.

8.2.2 Determination of analysis results

Follow the steps in Appendix A; calculate the independent repeated measurement results, according to the formula (1); compare them with the repeated measurement tolerance (R_d), to determine the analysis results.

8.2.3 Interlaboratory precision

Interlaboratory precision is used, to evaluate the consistency between the final results reported by two laboratories. After both laboratories report the results following the same procedure specified in 8.2.2, make calculation as follows:

$$\mu_{12} = \frac{\mu_1 + \mu_2}{2} \dots\dots\dots (6)$$

Where:

μ_1 - The final result reported by laboratory 1;

μ_2 - The final result reported by laboratory 2;

μ_{12} - Average of the final result.

If $|\mu_1 - \mu_2| \leq P$ (see 8.2.1), the final result is consistent.

8.2.4 Acceptance of analytical values

Acceptance of analytical values is verified, using certified reference samples. The steps are the same as described above. After confirming the precision, the laboratory final result is compared with the standard value A_c , for example:

- a) $|\mu_c - A_c| \leq C$, there is no significant difference between the measured value and the standard value.

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