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Replacing GB/T 223.59-1987

Iron, steel and alloy - Determination of phosphorus content - Bismuth phosphomolybdate blue spectrophotometric method and antimony phosphomolybdate blue spectrophotometric method

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Issued by: General Administration of Quality Supervision, Inspection and Quarantine;

Standardization Administration Committee.

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Foreword

This Part of GB/T 223 replaces "Methods for Chemical Analysis of Iron, Steel and Alloy-The Reduced Molybdoantimonyl Phosphoric Acid Photometric Method for the Determination of Phosphorus Content" (GB/T 223.59-1987).

In this revision, this Part has been renamed as "Iron, Steel and Alloy-Determination of Phosphorus Content-Bismuth Phosphomolybdate Blue Spectrophotometric Method and Antimony Phosphomolybdate Blue Spectrophotometric Method"; it includes two analysis methods: bismuth phosphomolybdate blue spectrophotometric method (Method I) and antimony phosphomolybdate blue spectrophotometric method ((Method II)).

Method I is a new method.

While compared with GB/T 223.59-1987, Method II in this Part mainly has the following revisions in terms of technical contents:

- added the description content for reagents and water in the analysis, and revised the expression method for solution concentration;
- revised the expression for weighing specimen quantity;
- revised the expression of quantity in result calculation formulae and quantity units in formulae;
- specified the explanation for precision function formula.

Appendix A of this Part is informative.

This Part was proposed by China Iron and Steel Association.

This Part shall be under the jurisdiction of National Technical Committee on Iron and Steel of Standardization Administration of China.

The drafting organizations of this Part: Maanshan Iron & Steel Co., Ltd and Central Iron & Steel Research Institute.

Main drafters of this Part: Cheng Jianping, Long Rucheng, Cui Qiuhong, Xu Fenlan and Hua Jing.

Versions of standard substituted by this Standard are:

- Method IV in GB/T 223.3-1981;
- GB/T 223.59-1987.

Iron, steel and alloy - Determination of phosphorus content - Bismuth phosphomolybdate blue spectrophotometric method and antimony phosphomolybdate blue spectrophotometric method

WARNING: The personnel using this Part shall have practical experiences in regular laboratory work. This Part does not point out all the possible safety problems. Users have the responsibility to take appropriate safety and health measures, and ensure meeting the conditions specified in relevant regulations of the State.

1 Scope

This Part of GB/T 223 specifies the determination of phosphorus content with bismuth phosphomolybdate blue spectrophotometric method and antimony phosphomolybdate blue spectrophotometric method.

This Part is applicable to the determination of phosphorus content in pig iron, cast iron, iron powder, carbon steel, low alloy steel and alloy steel and is not applicable to niobium and wolfram steel. The determination scope of Method I is 0.005% ~0.300% mass fraction while the determination scope of Method II is 0.01% ~0.06% mass fraction.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Part of GB/T 223. For dated reference, subsequent amendments to (excluding correction to), or revisions of, any of these publications do not apply. However, the parties whose enter into agreement according to this Part are encouraged to research whether the latest editions of these documents are applied or not. For undated references, the latest edition of the normative document is applicable to this Part.

GB/T 6379.1, *Accuracy (Trueness and Precision) of Measurement Methods and Results-Part 1: General Principles and Definitions* (GB/T 6379.1-2004, ISO 5725-1: 1994, IDT)

GB/T 6379.2, *Accuracy (Trueness and Precision) of Measurement Methods and Results Part 2: Basic Method for the Determination of*

3.2.8 Acid mixture of hydrochloric acid and nitric acid, 2+1. Mix two shares of hydrochloric acid (3.2.3) and one share of nitric acid (3.2.4) uniformly.

3.2.9 Acid mixture of hydrobromic acid and hydrochloric acid, 1+2. Mix one share of hydrobromic acid (3.2.5) and two shares of hydrochloric acid (3.2.3) uniformly.

3.2.10 Ascorbic acid solution, 20 g/L. Weigh 2 g of ascorbic acid, place it into 100 mL beaker and add 50 mL of water to dissolve and dilute it until 100 mL, then mix uniformly. This solution shall be prepared immediately before use.

3.2.11 Ammonium molybdate solution, 30 g/L. Weigh 3 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$] dissolve it in water, dilute until 100 mL and mix uniformly.

3.2.12 Sodium nitrite solution, 100 g/L. Weigh 10g of sodium nitrite, dissolve in water, dilute until 100 mL and mix uniformly.

3.2.13 Bismuth nitrate solution, 10 g/L. Weigh 10 g of bismuth nitrate [$Bi(NO_3)_3\cdot 5H_2O$], Place it into 200 mL beaker, add 25 mL of nitric acid (3.2.4) , dissolve it in water, boil to remove nitrogen oxide, cool until room temperature, transfer it into 1000 mL volumetric flask, dilute it with water to the scale and mix uniformly.

3.2.14 Iron solution

3.2.14.1 Iron solution A, 5 mg/mL. Weigh 0.5000 g of pure iron (with mass fraction of phosphorus less than 0.0005%), dissolve it with 10 mL of hydrochloric acid (3.2.3) before dropwise adding nitric acid (3.2.4) for oxidation, add 3 mL of perchloric acid (3.2.2) for evaporating until giving out perchloric acid smoke and continue evaporating till taking on wet salt state, cool it with 20 mL of sulfuric acid (3.2.7) dissolution salt until room temperature, transfer into 100 mL volumetric flask, dilute it with water to the scale and mix uniformly. This solution contains 5 mg of iron per mL.

3.2.14.2 Iron solution B, 1 mg/mL. Weigh 0.1000 g of pure iron (with mass fraction of phosphorus less than 0.001%), dissolve it with 10 mL of hydrochloric acid (3.2.3) before dropwise adding nitric acid (3.2.4) for oxidation, add 3 mL of perchloric acid (3.2.2) for evaporating until giving out perchloric acid smoke and continue evaporating till taking on wet salt state, cool it with 20 mL of sulfuric acid (3.2.7) dissolution salt until room temperature, transfer into 100 mL volumetric flask, dilute it with water to the scale and mix uniformly. This solution contains 1 mg of iron per mL.

3.2.15 Phosphorus standard solution

4.2.6 Sulfuric acid, 1+5. Slowly add sulfuric acid (4.2.4) into water while stirring and diluting into 1+5.

4.2.7 Acid mixture of hydrochloric acid and nitric acid, 2+1. Mix two shares of hydrochloric acid (4.2.2) and one share of nitric acid (4.2.3) uniformly.

4.2.8 Acid mixture of hydrobromic acid and hydrochloric acid, 1+2. Mix one share of hydrobromic acid (4.2.5) and two shares of hydrochloric acid (4.2.2) uniformly.

4.2.9 Ascorbic acid solution, 30 g/L. Weigh 3 g of ascorbic acid, place it into 100 mL beaker and add 50 mL of water to dissolve and dilute it until 100 mL, then mix uniformly. This solution shall be prepared immediately before use.

4.2.10 Ammonium molybdate solution, 20 g/L. Weigh 2 g of ammonium molybdate [$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$], dissolve it into water, dilute until 100 mL and mix uniformly.

4.2.11 Potassium antimony tartrate solution, 2.7 g/L; this solution contains 1 mg of antimony per mL.

4.2.12 Sodium nitrite solution, 100 g/L. Weigh 10 g of sodium nitrite, dissolve in water, dilute until 100 mL and mix uniformly.

4.2.13 Starch solution, 10 g/L. Weigh 1g of soluble starch [if there is high phosphorus content in the starch, it shall be subject to fully agitator treating with hydrochloric acid (5+95), after settlement, pour forth acid solution and wash it with water till neutral]; after wetting it with a small amount of water, add 100 mL of boiling water while stirring it, mix uniformly and boil for a while. It shall heat it until the solution becomes transparent and then cool it until room temperature before use.

4.2.14 Iron solution, 4 g/L. Weigh 0.4 g of pure iron (with mass fraction of phosphorus less than 0.001%), dissolve it with 10 mL of hydrochloric acid (4.2.2) before dropwise adding nitric acid (4.2.3) for oxidation, add 3 mL of perchloric acid (4.2.1) for evaporating until giving out perchloric acid smoke and continue evaporating till taking on wet salt state, cool it with 20 mL of sulfuric acid (4.2.6) dissolution salt until room temperature, transfer into 100 mL volumetric flask, dilute it with water to the scale and mix uniformly.

4.2.15 Phosphorus standard solution

4.2.15.1 Phosphorus stock solution, 100 μg/mL. Weigh 0.4393 g of reference potassium dihydrogen phosphate (KH_2PO_4) dried in advance at 105°C till constant quantity, dissolve it with right amount of water, add 5 mL of sulfuric acid (4.2.6), transfer into 1000 mL volumetric flask, dilute it with water to the scale and mix uniformly. This solution contains 100 μg of phosphorus

to the scale and mix uniformly.

Take two shares of 10.00 mL of test solutions and place them into 25 mL volumetric flask respectively.

4.5.3.3 Add 2.0 mL of sulfuric acid (4.2.6), 0.3 mL of potassium antimony tartrate solution (4.2.11), 2 mL of starch solution (4.2.13) and 2 mL of ascorbic acid solution (4.2.9) (Each newly-added reagent shall be mixed uniformly; it may also mix the required sulfuric acid, potassium antimony tartrate and starch solution in proportion during color rendering and then add the mixed solution at a time).Add 5.0 mL of ammonium molybdate solution (4.7) to one share (add from the middle volumetric flask opening, the ammonium molybdate solution on the flask wall need be washed with water; otherwise, the ammonium molybdate on the flask wall will be reduced to blue due to low acidity, thus causing measurement error), dilute it with water to the scale and mix uniformly.

4.5.3.4 Add no ammonium molybdate solution to the other share, dilute it with water to the scale and mix uniformly.

4.5.3.5 After placing at 20°C~30°C for 10min, transfer it into 2cm ~ 3cm cuvettes, take the one share without ammonium molybdate solution as the reference, measure the absorbance at 700nm wave length on spectrophotometer, subtract the blank absorbance accompanied samples, and find out the corresponding phosphorus content from calibration curve.

4.5.4 Plotting of calibration curve

Take 0 mL, 1.00 mL, 2.00 mL, 4.00 mL, 6.00 mL and 8.00 mL of phosphonium standard solution (4.2.15.2), place them into 6 25-mL volumetric flask, add 5 mL of iron solution (4.2.14), and the following procedure shall be in accordance with 4.5.3.3. After placing at 20°C~30°C for 10min, transfer it into 2cm ~ 3cm cuvettes, take water as the reference, measure the absorbance at 700nm wave length on spectrophotometer, subtract the absorbance of reagent blank; take phosphorus content as the lateral coordinate, absorbance as the vertical coordinate and plot the calibration curve.

4.6 Result calculation

The mass fraction wp of phosphorus content, expressed in percentage (%), shall be calculated according to Formula (2):

$$w_P = \frac{m_1 \times V \times 10^{-6}}{m \times V_1} \times 100 \qquad \cdots\cdots(2)$$

where,

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