

ICS 77.080.01

CCS H11



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

GB/T 223.59-2008

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**

钢铁及合金 磷含量的测定 铋磷钼蓝分光光度法和锑磷钼蓝分光光度法

Issued on: August 19, 2008

Implemented on: April 1, 2009

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

Table of Contents

- 1 Scope
- 2 Normative references
- 3 Method I. Bismuth Phosphomolybdate Blue
 - 3.2.14 Iron solution
 - 3.2.15 Phosphorus standard solution
 - 3.2.15.2 Phosphorus standard solution, 5.0 µg/mL. Take

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 Qihong, 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

GB/T 223.59-2008 is the Chinese national standard on iron, steel and alloy - determination of phosphorus content - bismuth phosphomolybdate blue spectrophotometric method and antimony phosphomolybdate blue spectrophotometric method, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 19 August 2008 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 April 2009. Classification: ICS 77.080.01, CCS H11. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

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 who 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 Repeatability and Reproducibility of a Standard Measurement Method (GB/T 6379.2-2004, ISO 5725-2. 1994, IDT)

GB/T 20066, Steel and Iron-Sampling and Preparation of Samples for the Determination of

Chemical Composition (GB/T 20066-2006, ISO 14284. 1996, IDT)

3 Method I. Bismuth Phosphomolybdate Blue

Spectrophotometric Method

3.1 Principle After the sample is subject to acid dissolution, perchloric acid smoke is given out so that all phosphorus is oxidized into orthophosphoric acid and the carbide is damaged. In sulfuric acid medium, the phosphorus, bismuth and ammonium molybdate form yellow complex compound; ascorbic acid is used to reduce bismuth phosphomolybdate yellow into bismuth phosphomolybdate blue. The absorbance shall be measured at 700nm wave length on spectrophotometer; the mass fraction of phosphorus shall be calculated out. If there are 150 μ g of titanium, 10 mg of manganese, 2 mg of cobalt, 5 mg of copper,

0.5 mg of vanadium, 10 mg of nickel, 500 μ g of chromium (III), 50 μ g of cerium, 5 mg of zirconium, 5 μ g of niobium and 10 μ g of wolfram in color reagent, the determination will not be affected. Arsenium has severe interference on the determination and may be removed with hydrobromic acid where treating samples.

3.2 Reagents and materials Unless otherwise specified, it can only use approved analytical reagent and distilled water or water with equivalent purity during the analysis.

3.2.1 Hydrofluoric acid, rho is about

3.2.2 Perchloric acid, rho is about

3.2.3 Hydrochloric acid, rho is about

3.2.4 Nitric acid, rho is about

3.2.5 Hydrobromic acid, rho is about

3.2.6 Sulfuric acid, rho is about

3.2.7 Sulfuric acid, 1+1. Slowly add sulfuric acid (3.2.6) into water while stirring and diluting into 1+1.

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.

3.2.14 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

3.2.15.1 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 (3.2.7), transfer into 1000 mL volumetric flask, dilute it with water to the scale and mix uniformly. This solution contains 100 µg of phosphorus per mL.

3.2.15.2 Phosphorus standard solution, 5.0 µg/mL. Take

50.00 mL of phosphorus stock solution (3.2.15.1), place it into 1000 mL volumetric flask, dilute it with water to the scale and mix uniformly. This solution contains 5.0 µg of phosphorus per mL.