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

GB/T 24583.5-2019

Replacing GB/T 24583.5-2009

**Vanadium-nitrogen alloy - Determination of the phosphorus
content - Bismuth phosphomolybdenum blue
spectrophotometry**

钒氮合金 磷含量的测定 铋磷钼蓝分光光度法

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Foreword

The analysis method of GB/T 24583 vanadium nitrogen alloy is divided into 8 parts.

- GB/T 24583.1 Determination of vanadium content of vanadium-nitrogen alloys - Ammonium sulphate titration method;
- GB/T 24583.2 Determination of nitrogen content of vanadium-nitrogen alloys - Inert gas fusion heat conduction method;
- GB/T 24583.3 Determination of nitrogen content of vanadium-nitrogen alloys - Distillation - Neutralometric titration;
- GB/T 24583.4 Determination of carbon content of vanadium-nitrogen alloys - infrared absorption method;
- GB/T 24583.5 Determination of phosphorus content of vanadium-nitrogen alloys - bismuth phosphorus molybdenum blue spectrophotometric method;
- GB/T 24583.6 Determination of sulfur content of vanadium-nitrogen alloys by infrared absorption method;
- GB/T 24583.7 Determination of oxygen content of vanadium-nitrogen alloys - infrared absorption method;
- GB/T 24583.8 Determination of silicon, manganese, phosphorus and aluminium content of vanadium-nitrogen alloys by inductively coupled plasma atomic emission spectrometry. This part is the fifth part of GB/T 24583. This part is drafted in accordance with the rules given in GB/T 1.1-2009. This part replaces GB/T 24583.5-2009 "Determination of phosphorus content of vanadium-nitrogen alloys by bismuth phosphomolybdenum blue spectrophotometry". This part and Compared with GB/T 24583.5-2009, the main technical changes are as follows:
 - Modified "normative references" (see Chapter 2, Chapter 2 of the.2009 edition);
 - Added "instrument" (see Chapter 5);

--- Revised the method of preparation and preparation of the sample (see Chapter 6, Chapter 6 of the.2009 edition);

--- Modified the drawing of the calibration curve (see 7.6, 6.5 of the.2009 edition);

1 Scope

China's national method for determining phosphorus in vanadium-nitrogen alloy by bismuth phosphomolybdenum blue spectrophotometry. It is Part 5 of GB/T 24583 and specifies the determination of the phosphorus content of the alloy. Phosphorus in a ferroalloy is a residual, and it is controlled because the steel it goes into cannot get rid of it. Phosphorus is not removed in the ladle by any of the ordinary refining operations - dephosphorisation happens in the converter, before the alloy additions are made - so every gram of phosphorus that arrives with an alloy stays in the steel. And phosphorus causes cold shortness: it segregates and embrittles, and the specification on the finished steel is correspondingly tight. A microalloying addition made near the end of the process is therefore assayed for phosphorus with as much attention as for its active element. The determination is at a low concentration in a difficult matrix, and the method is the classical molybdenum blue chemistry adapted to it. Phosphate forms a heteropoly acid with molybdate, which is then reduced to an intensely coloured blue complex whose absorbance is proportional to the phosphorus; the bismuth variant is used because bismuth stabilises the complex and improves both the sensitivity and the reproducibility. What the standard has to handle is the vanadium matrix itself: vanadium is coloured in solution, it is a redox-active element that interferes with the reduction step, and silicon and arsenic form the same kind of heteropoly complex and are determined as phosphorus unless they are dealt with. So the decomposition, the separation or masking of vanadium, silicon and arsenic, the acidity and the reduction conditions, the wavelength and the calibration are all fixed. Issued on 4 June 2019 and in force since 1 May 2020, it replaces GB/T 24583.5-2009.

This part of GB/T 24583 specifies the determination of phosphorus in vanadium-nitrogen alloy by bismuth phosphomolybdate blue spectrophotometry. This section applies to the determination of phosphorus content in vanadium-nitrogen alloys. Measuring range (mass fraction). 0.010%~0.100%.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only dated versions apply to this article. Pieces. For undated references, the latest edition (including all amendments) applies to this document.

GB/T 6379.1 Accuracy of measurement methods and results (accuracy and precision) Part

1. General and definition

GB/T 6379.2 Accuracy of measurement methods and results (accuracy and precision) Part

2. Determination of standard method of measurement Basic method of sex and reproducibility

GB/T 6682 Analytical laboratory water specifications and test methods

GB/T 8170 Numerical Rounding Rules and Representation and Determination of Limit Values

GB/T 12806 laboratory glass instrument single standard line volumetric flask

GB/T 12807 laboratory glass instrument indexing pipette

GB/T 12808 laboratory glass instrument single standard line pipette

GB/T 20567 vanadium nitrogen alloy

3 Principle

The sample was decomposed with nitric acid and the phosphorus was oxidized to orthophosphoric acid with potassium permanganate. Phosphorus and phosphorus in a medium of

0.8 mol/L to

1.2 mol/L and ammonium molybdate form a complex, which is reduced to ascorb blue with ascorbic acid, and its absorbance is measured at 700 nm of a spectrophotometer.

4 Reagents and materials

In the analysis, unless otherwise stated, only approved analytical reagents and distilled water of Grade III and above in accordance with GB/T 6682 are used. Or deionized water or water of equivalent purity.

4.1 Nitric acid, 1 1.

4.2 Potassium permanganate solution, 20g/L.

4.3 sodium nitrite solution, 20g/L.

4.4 bismuth nitrate solution, 10g/L. 10 g of bismuth nitrate $[\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}]$ was weighed and dissolved in nitric acid (12) and diluted to 1 L.

4.5 Phosphorus coloring solution, weigh 40g of sodium potassium tartrate and 50g of ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ dissolved in water, diluted Up to 1000mL.

4.6 Ascorbic acid solution, 20g/L. Formulated when used.