

ICS 77.100
CCS H 11



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

GB/T 24583.3-2019

Replacing GB/T 24583.3-2009

**Vanadium-nitrogen alloy - Determination of the nitrogen content
- Distillation and neutralisation titration method**

钒氮合金 氮含量的测定 蒸馏-中和滴定法

Issued on: June 4, 2019

Implemented on: May 1, 2020

**Issued by: State Administration for Market Regulation;
Standardization Administration of China**

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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 third 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 24583.3-2009 "Determination of Nitrogen Content of Vanadium Nitrogen Alloys by Distillation - Neutralization Titration". This part and Compared with GB/T 24583.3-2009, the main technical changes are as follows:
 - Change the measurement range (mass score) to "8.00%~20.00%" (see Chapter 1, Chapter 1 of the.2009 edition);

- Modified "normative references" (see Chapter 2, Chapter 2 of the.2009 edition);
- Revised the method of preparation and preparation of the sample (see Chapter 6, Chapter 6 of the.2009 edition);
- Modified by "heating and dissolving on a low temperature electric furnace" to "heating on an electric furnace until smoke is dissolved" (see 7.4, 7.4 of.2009 edition);

1 Scope

China's national method for determining nitrogen in vanadium-nitrogen alloy by distillation and neutralisation titration. It is Part 3 of GB/T 24583 and specifies the determination of the nitrogen content of the alloy. This part determines the same quantity as Part 2 and does it in an entirely different way, which is why both exist. The inert gas fusion method of Part 2 is fast and needs no reagents, but it is an instrumental method: it measures a signal and converts it through a calibration built on certified reference materials, so its accuracy depends on those materials and on the instrument being right. The distillation method is classical and absolute. The sample is decomposed so that the nitride nitrogen is converted to ammonium, the solution is made alkaline, and the ammonia is distilled out and collected in a measured excess of standard acid; the acid not neutralised is then back-titrated, and the nitrogen follows from the difference by stoichiometry alone. There is no calibration curve and no reference material between the sample and the answer - only the purity of the standard acid and the accuracy of a burette. That makes it the referee method: slower and more laborious, requiring careful work to avoid losing ammonia or carrying alkali over, and used to settle a dispute over a shipment, to validate the instrumental method, or in a laboratory that does not have an analyser. The standard fixes the decomposition, which for a hard nitride is the difficult step, the distillation apparatus and the conditions that ensure the ammonia is quantitatively transferred, the absorption and back-titration, the indicator or endpoint, the blank and the precision. Issued on 4 June 2019 and in force since 1 May 2020, it replaces GB/T 24583.3-2009.

This part of GB/T 24583 specifies the determination of nitrogen in vanadium-nitrogen alloys by distillation-neutralization titration. This section applies to the determination of nitrogen content in vanadium-nitrogen alloys. Measuring range (mass fraction). 8.00%~20.00%.

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 20567 vanadium nitrogen alloy

3 Principle

The sample is decomposed in a sulfur-phosphorus mixed acid medium, wherein the nitrogen is converted into an ammonium salt, and under the action of excess alkali, the generated ammonia gas is accompanied by water vapor. It is separated by distillation, and the vapor is condensed to form ammonia water. Absorbing ammonia water with boric acid solution, using sulfamic acid standard titration solution Titration.

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.

4.2 Phosphoric acid, $\rho =$

4.3 Boric acid solution, 25g/L.

4.4 sodium hydroxide solution, 500g/L. Weigh 500g of sodium hydroxide, dissolve it in 800mL of water, add a few grains of zinc, heat and boil for 10min, remove the cooling, remove the residual zinc particles, Dilute to 1000 mL with water, mix and store in plastic bottles.

Note. Slowly add sodium hydroxide to the beaker with water and stir constantly. If the exotherm is large, it can be formulated in a water-cooled environment.

4.5 Methyl red-methine blue mixed indicator.

0.125 g of methyl red and

0.083 g of methylene blue were weighed, dissolved in 100 mL of ethanol, stored in a brown bottle, and mixed.