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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 24583.1-2019

Replacing GB/T 24583.1-2009

Vanadium-nitrogen alloy - Determination of the vanadium content - Ammonium ferrous sulfate titration method

钒氮合金 钒含量的测定 硫酸亚铁铵滴定法

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Foreword

GB/T 24583 Vanadium-nitrogen alloy analysis method is divided into 8 parts: - GB/T 24583.1 Vanadium-nitrogen - Determination of vanadium content - Ammonium ferrous sulfate titration method; - GB/T 24583.2 Vanadium-nitrogen - Determination of nitrogen content - Thermal conductimetric method after fusion in a current of inert gas; - GB/T 24583.3 Vanadium-nitrogen - Determination of nitrogen content - Distillation-neutralization titration method; - GB/T 24583.4 Vanadium-nitrogen - Determination of carbon content - Infrared absorption method; - GB/T 24583.5 Vanadium-nitrogen - Determination of phosphorus content - Bismuth molybdenum blue spectrophotometric method; - GB/T 24583.6 Vanadium-Nitrogen alloy - Determination of sulfur content - The infrared absorption method; - GB/T 24583.7 Vanadium-nitrogen - Determination of oxygen content - Infrared absorption method; - GB/T 24583.8 Vanadium-nitrogen - Determination of silicon, manganese, phosphorus, aluminum content - Inductively coupled plasma atomic emission spectrometric method. This Part is Part 1 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 24583.1-2009 "Vanadium-Nitrogen alloy - Determination of vanadium content - The ammonium ferrous sulfate titration method". Compared with GB/T 24583.1-2009, the main technical changes in

this Part are as follows: - Modify "Normative references" (see Clause 2; Clause 2 of the 2009 edition); - ADD "Instruments" (see Clause 5); - Modify the concentration and calibration method of the standard titration solution of ammonium ferrous sulfate (see 4.11;

4.10 of the 2009 edition); - Modify the method of sampling and sample preparation (see Clause 6; Vanadium-nitrogen - Determination of vanadium content - Ammonium ferrous sulfate titration method Caution - The personnel using this Part shall have hands-on experience in formal laboratory work. This Part does not address all possible safety problems. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions set by the relevant national regulations.

1 Scope

China's national method for determining vanadium in vanadium-nitrogen alloy by titration with ammonium ferrous sulfate. It is Part 1 of GB/T 24583 and specifies the determination of the vanadium content of the alloy. Vanadium-nitrogen alloy is bought and sold on its vanadium content, and vanadium is the expensive part by a wide margin - so this determination settles the invoice. That is why the method is a titration rather than an instrumental technique: a titration is absolute, depending on the stoichiometry of a reaction and the purity of a standardised reagent rather than on a calibration built from reference materials, and for a determination with a large price attached at a concentration around eighty per cent, that traceability is the point. The chemistry uses vanadium's accessible oxidation states. The sample is dissolved and the vanadium is brought fully to the pentavalent state by oxidation, the excess oxidant is destroyed, and the vanadium is then titrated back to the tetravalent state with a standard solution of ammonium ferrous sulfate, with a redox indicator or a potentiometric endpoint. The reaction is clean and rapid and the endpoint is sharp. What the standard has to fix is everything around it: the decomposition of an alloy that is a hard nitride and does not dissolve readily, the complete oxidation and the removal of the excess oxidant, since any left over is titrated as vanadium and biases the result high, the interference from any iron present in the alloy, the standardisation of the titrant, and the precision - which for a commercial assay of this kind is stated tightly. Issued on 4 June 2019 and in force since 1 May 2020, it replaces GB/T 24583.1-2009.

This Part of GB/T 24583 specifies the determination of vanadium content in vanadium-nitrogen alloy by ammonium ferrous sulfate titration method. This Part applies to the determination of vanadium content in vanadium- nitrogen alloy. Measuring range (mass fraction): >=60.00%.

2 Normative references

The following documents are indispensable for the application of this document. For the dated references, only the editions with the dates indicated are applicable to this document. For the undated references, the latest edition (including all the amendments) are applicable

to this document.

GB/T 6379.1 Accuracy (trueness and precision) of measurement methods and results - Part 1: General principles and definitions

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 6682 Water for analytical laboratory use - Specification and test methods

GB/T 8170 Rules of rounding off for numerical values & expression and judgement of limiting values

GB/T 12805 Laboratory glassware - Burettes

GB/T 12806 Laboratory glassware - One-mark volumetric flasks DILUTE to the mark and mix well.

4.11 Standard titration solution of ammonium ferrous sulfate, about

0.07 mol/L.

4.11.1 Preparation WEIGH

27.45 g of ammonium ferrous sulfate [(NH4)2Fe(SO4)2 - 6H2O] in a beaker; DISSOLVE it in an appropriate amount of sulfuric acid (5+95); USE sulfuric acid (5+95) to dilute to 1000 mL; MIX well.

4.11.2 Calibration and indicator correction TAKE 3 parts of

5.00 mL of potassium dichromate standard solution (see 4.10); PLACE them into 500 mL conical flasks respectively. ADD 20 mL of sulfuric acid (see 4.4), 5 mL of phosphoric acid (see 4.2), 70 mL of water, 3 drops of N- benzo-o-aminobenzoic acid indicator solution (see 4.13) in sequence. USE the standard titration solution of ammonium ferrous sulfate (see 4.11) to titrate until the solution is changed from fuchsia to bright yellow-green as end point, regardless of the volume of the standard titration solution of ammonium ferrous sulfate consumed. After adding

20.00 mL (V) of potassium dichromate standard solution (see 4.10), USE the standard titration solution of ammonium ferrous sulfate (see 4.11.1) to titrate until the solution is changed from fuchsia to bright yellow-green as end point; RECORD the volume V1 of the standard titration solution of ammonium ferrous sulfate consumed. According to formula (1), calculate the concentration of the standard titration solution of ammonium ferrous sulfate. Where: c1 - The concentration of standard titration solution of ammonium ferrous sulfate, in moles per liter (mol/L); c - The concentration of potassium dichromate standard solution, in moles per liter (mol/L); V - The volume of the potassium dichromate standard solution taken

separately, in milliliters (mL); V1 - The volume of the standard titration solution of ammonium ferrous sulfate consumed by titrating the potassium dichromate standard solution, in milliliters (mL).

4.12 Vanadium pentoxide solution,

4.0 mg/mL. ammonium ferrous sulfate to titrate. When closing to the end point, TITRATE slowly until the solution is changed from fuchsia to bright yellow-green as the end point. The volume of the standard titration solution of ammonium ferrous sulfate consumed is V3. The sample analysis reagent blank V0=V2-V3.

7.4 Verification test The same type of standard sample is analyzed along with the test portion.

7.5.1 Potassium permanganate oxidation-ammonium ferrous sulfate titration method

7.5.1.1 Decomposition of test portion PLACE the test portion (see 7.2) in a 500 mL conical flask; along the wall of the flask, ADD a little water to wet the sample. ADD 5 mL of phosphoric acid (see 4.2), 10 mL of nitric acid (see 4.3), 40 mL of sulfuric acid (see 4.4); HEAT until the test portion is dissolved completely and produces sulfuric acid smoke for 2 min~3 min; REMOVE and cool. Along the wall, ADD about 100 mL of water; COOL to room temperature.

7.5.1.2 Oxidation of vanadium ADD 5 mL of ammonium ferrous sulfate solution (see 4.5) to the test solution (see 7.5.1.1) and place for 2 min; ADD dropwise potassium permanganate solution (see 4.6) until fuchsia is stable; LET stand for 5 min. ADD 10 mL of urea solution (see 4.7); ADD dropwise sodium nitrite solution (see 4.8) until red disappears and add 1 drop~2 drops; PLACE for 2 min.

7.5.1.3 Titration ADD 3 drops of N-benzo-o-aminobenzoic acid indicator (see 4.13); immediately USE the standard titration solution of ammonium ferrous sulfate (see 4.11) to titrate until fuchsia is changed to bright yellow-green as end point. The volume of the standard titration solution of ammonium ferrous sulfate consumed is V4.

7.5.2 Ammonium persulfate oxidation-ammonium ferrous sulfate titration method

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