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

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**Iron, steel and alloy - Determination of vanadium content -
Titrimetric method and spectrophotometric method
(English Translation)**

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1 Scope

This document the methods for determining vanadium content using ferrous ammonium sulfate titration and tantalum reagent extraction spectrophotometry.

This document applies to the determination of vanadium content in steel and alloys.

Method One is the ferrous ammonium sulfate titration method, applicable to the determination of vanadium content in steel and alloys with a mass fraction of 0.1% to 10.0%. It is not applicable to the determination of vanadium content in the following samples. a) specimens containing more than 20% cobalt (mass fraction); b) specimens containing more than 0.01% cerium (mass fraction); c) specimens containing more than 20% chromium, more than 20% manganese, or more than 18% tungsten (mass fraction), with a vanadium content less than 0.4%. Method Two is the N-BPHA (tantalum reagent) extraction spectrophotometric method, applicable to the determination of vanadium content in steel and cast iron with a mass fraction of 0.005%~0.50%. Method Three is the tantalum reagent extraction spectrophotometric method, applicable to the determination of vanadium content in steel and alloys with a mass fraction of

0.005%~0.50%.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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, Measurement methods and results -- Accuracy (trueness and precision)

-- Part 2. Determine the standard methods of measurement repeatability and reproducibility of the basic method (GB/T 6379.2-2004, ISO 5725-2.1994, IDT)

GB/T 6682, Water for analytical laboratory use -- Specification and test methods (GB/T 6682-2008, ISO 3696.1987, MOD)

GB/T 7729, Chemical analysis of metallurgical products -- General rule for spectrophotometric methods

GB/T 12805, Laboratory glassware -- Burettes (GB/T 12805-2011, ISO 385.2005, NEQ)

GB/T 12806, Laboratory glassware -- One-mark volumetric flasks (GB/T 12806-2011, ISO 1042.1998, NEQ)

GB/T 12808, Laboratory glassware - One mark pipettes

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)

ISO 648, Laboratory glassware -- One-mark pipettes

3 Terms and definitions

This document does not contain any terms or definitions that need to be defined.

4.1 Principle

After the test material is dissolved in a suitable acid, vanadium is oxidized with potassium permanganate at room temperature in a sulfuric acid-phosphoric acid medium. Excess potassium permanganate is reduced with sodium nitrite. Excess sodium nitrite is decomposed with urea. Vanadium is titrated with a standard ferrous ammonium sulfate solution using benzoic acid as an indicator.

4.2 Reagents and materials

In the analysis, unless otherwise specified, only analytically pure reagents and Grade II water or water of equivalent purity as specified in GB/T 6682 shall be used.

4.2.1

Hydrochloric acid. ρ is approximately 1.19 g/mL.

4.2.2

Nitric acid. ρ is approximately 1.42 g/mL.

4.2.3

Sulfuric acid (5+95). diluted with sulfuric acid (rho approx. 1.84 g/mL).

4.2.4

Phosphoric acid. rho is approximately 1.69 g/mL.

4.2.5 Sulfuric acid-phosphoric acid mixture. while stirring, slowly add 200 mL of

sulfuric acid (rho is approximately 1.84 g/mL) to 400 mL of water. Let cool slightly. Add 100 mL of phosphoric acid (4.2.4). Mix well.

4.2.6 Potassium permanganate solution. 3 g/L.

4.2.7 Sodium nitrite solution. 20 g/L.

4.2.8 Urea solution. 200 g/L.

4.2.9 Ferrous ammonium sulfate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$ solution. 40 g/L.

Weigh 40.0 g of ferrous ammonium sulfate hexahydrate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$.

Dissolve in sulfuric acid (4.2.3) and dilute to 1000 mL. Mix well.

4.2.10 Benzyl anthranilic acid solution. 2 g/L.

Weigh 0.2 g of benzo (o-aminobenzoic acid) into a 300 mL beaker. Add 0.2 g of anhydrous sodium carbonate. Add 20 mL of water and heat to dissolve. Dilute with water to 100 mL. Mix well.

4.2.11 Sodium arsenite solution. 5 g/L.

Weigh 0.38 g of arsenic trioxide. Dissolve in 50 mL of sodium hydroxide solution (50 g/L). Neutralize with sulfuric acid (rho is approximately 1.84 g/mL, 1+1) until the solution is neutral. Dilute with water to 100 mL. Mix well.

Alternatively, sodium arsenite can be used. Weigh 0.5 g of sodium arsenite and dissolve it in water. Dilute to 100 mL. Mix well.

4.2.12 Potassium dichromate standard solution $[\text{c}(1/6\text{K}_2\text{Cr}_2\text{O}_7) = 0.01000 \text{ mol/L}]$.

Weigh 0.4903 g of potassium dichromate, which has been pre-dried at 150°C for 1 h and cooled to room temperature in a desiccator. Place it in a 300 mL beaker. Dissolve it in water. Transfer the solution to a 1000 mL volumetric flask. Dilute to the mark with water. Mix well.

4.2.13 Ferrous ammonium sulfate standard solution $\{\text{c}[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}] = 0.01$

mol/L}.

4.2.13.1

Preparation.

Weigh 4.0 g of ferrous ammonium sulfate hexahydrate $[(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}]$.

Dissolve in sulfuric acid (4.2.3) and dilute to 1000 mL. Mix well.

4.2.13.2

Calibration and indicator calibration.

Take three 250 mL Erlenmeyer flasks. Add 15 mL of sulfuric acid (ρ is approximately 1.84 g/mL) and 10 mL of phosphoric acid (4.2.4) to each. Heat to evaporate until sulfuric acid fumes are emitted. Let cool slightly. Add 50 mL of water. Cool to room

The specimens used, the raw data of the precision tests, and the statistical results are listed in Tables C.1, C.2, and C.3 of Annex C. A graph of the precision data is shown in

Figure C.1.

The repeatability limit (r) and reproducibility limit (R) are obtained according to the equations given in Table 2.

Under repeatability conditions, the absolute difference between two independent test results shall not exceed the repeatability limit (r), provided that the number of cases exceeding the repeatability limit (r) does not exceed 5%.

Under reproducibility conditions, the absolute difference between two independent test results shall not exceed the reproducibility limit (R), provided that the number of cases exceeding the reproducibility limit (R) does not exceed 5%.

5 Method Two -- N-BPHA (tantalum reagent) extraction

spectrophotometric method

5.1 Principle

Dissolve the test material in a suitable acid. Add phosphoric acid to the solution, so as to eliminate interference from iron. Add potassium permanganate to oxidize vanadium to V(V). Excess permanganate is selectively reduced with sodium nitrite. In the presence of urea, add N-BPHA (tantalum reagent) and hydrochloric acid to form a complex. The mixture is then extracted with chloroform. Measure the absorbance at 535 nm.