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Methods for chemical analysis of tantalum and niobium - Part 1: Determination of tantalum content in niobium - Inductively coupled plasma atomic emission spectrometry

钽铌化学分析方法 第1部分：铌中钽量的测定 电感耦合等离子体原子发射光谱法

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Foreword

GB/T 15076 Methods for Chemical Analysis of Tantalum and Niobium is divided into 16 parts:

---Part 1: Determination of Tantalum Content in Niobium - Inductively Coupled Plasma Atomic Emission Spectrometry;

---Part 2: Determination of Niobium Content in Tantalum - Inductively Coupled Plasma Atomic Emission Spectrometry and Stratography Gravimetry;

---Part 3: Determination of Copper Content - Flame Atomic Absorption Spectrometry;

---Part 4: Determination of Iron Content - 1,10-Phenanthroline Spectrophotometry;

---Part 5: Determination of Molybdenum and Tungsten Contents - Inductively Coupled Plasma Atomic Emission Spectrometry;

---Part 6: Determination of Silicon Content - Inductively Coupled Plasma Atomic Emission Spectrometry;

---Part 7: Determination of Phospherus Content in Niobium - 4-Formyloxy-pentyl Keton- [2] Extraction Separation Phosphomolybdate Blue Spectrophotometry and Inductively Coupled Plasma Atomic Emission Spectrometry;

---Part 8: Determination of Carbon and Sulphur Contents;

---Part 9: Determination of Iron, Chromium, Nickel, Manganese, Titanium, Aluminum, Copper, Tin, Lead and Zirconium Contents in Tantalum;

---Part 10: Determination of Iron, Nickel, Chromium, Titanium, Zirconium, Aluminum and Manganese Contents in Niobium - Direct Current Arc Atomic Emission Spectrometry;

---Part 11: Determination of Arsenic, Antimony, Lead, Tin and Bismuth Contents in Niobium - Direct Current Arc Atomic Emission Spectrometry;

---Part 12: Determination of Phosphorus Content in Tantalum;

---Part 13: Determination of Nitrogen Content - Inert Gas Fusion Thermal Conductivity Method;

1 Scope

GB/T 15076.1-2017 is the Chinese national standard on methods for chemical analysis of tantalum and niobium - part 1: determination of tantalum content in niobium - inductively coupled plasma atomic emission spectrometry, 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 14 October 2017 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 May 2018. Classification: ICS 77.120.99, CCS H14. 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 15076 stipulates the determination method for tantalum content in niobium. This Part is applicable to the determination of tantalum content in niobium, niobium hydroxide and niobium oxide. The determination range is 0.0050% ~ 3.00%.

2 Method Summary

Use nitric acid and hydrofluoric acid to dissolve the test portion. In a dilute acid medium, use argon plasma as the ionization source, and directly conduct the determination through inductively coupled plasma atomic emission spectrometry.

3 Reagents and Materials

Unless otherwise stated, the reagents used in this Part are all guaranteed reagents that comply with national standards or industry standards, and the water used is Grade-2 water.

3.2 Nitric acid (=

3.3 Niobium matrix: wNb 99.99%, wTa 0.0001%.

3.4 Tantalum standard storage solution: accurately weigh-take 1.0000 g of pure tantalum (wTa > 99.99%) in a 250 mL polyethylene beaker, use a polyethylene lid to cover it, and add 10 mL of hydrofluoric acid (3.1). Dropwise add 10 mL of nitric acid (3.2) and dissolve it on 80 C water bath. After complete dissolution, transfer it to a 1,000 mL volumetric flask, use water to dilute it to the scale and mix it well. 1 mL of this solution contains 1 mg of tantalum. Store it in a polyethylene volumetric flask.

3.5 Tantalum standard solution: transfer-take

10.00 mL of tantalum standard storage solution (3.4) into a 100 mL volumetric flask, add 2 mL of hydrofluoric acid (3.1), use water to dilute to the scale, and mix it well. 1 mL of this solution contains 100 g of tantalum. Store it in a polyethylene volumetric flask.

3.6 Argon: volume fraction is not less than 99.99%.

6.4.2 In accordance with Table 1, add hydrofluoric acid (3.1). After the violent reaction stops, dropwise add 4 mL of nitric acid (3.2), heat at a low temperature, until the test portion is completely dissolved, then remove and cool it to room temperature. When the oxide is not easily dissolved to clarity by this method, a hot-pressure digestion device can be used to dissolve the sample. Transfer the solution into a 100 mL polyethylene volumetric flask, use water to dilute to the scale, and mix it well.

6.5 Drawing of Working Curve 6.5.1 0.0050% wTa < 0.050% In accordance with Table 1, weigh-take 6 portions of the niobium matrix (3.3) equivalent to the test portion, and respectively place them in six 100 mL polytetrafluoroethylene beakers. Respectively add 0 mL,

0.25 mL,

0.50 mL,

3.00 mL of the tantalum standard solution (3.5), and follow

6.4.2 for the following steps. 6.5.2 0.050% < wTa 0.50% In accordance with Table 1, weigh-take 6 portions of the niobium matrix (3.3) equivalent to the test portion, and respectively place them in six 100 mL polytetrafluoroethylene beakers. Respectively add 0 mL,

15.00 mL of the tantalum standard solution (3.5), and follow

6.4.2 for the following steps. 6.5.3 0.50% < wTa 3.00% In accordance with Table 1, weigh-take 6 portions of the niobium matrix (3.3) equivalent to the test portion, and respectively place them in six 100 mL polytetrafluoroethylene beakers. Respectively add 0

mL,

0.25 mL,

0.50 mL,

3.00 mL of the tantalum standard storage solution (3.4), and follow

6.4.2 for the following steps.