ICS 77.120.99
CCS H14

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 15076.2-2019

Replacing GB/T 15076.2-1994

Methods for chemical analysis of tantalum and niobium - Part 2:Determination of niobium content in tantalum - Inductively coupled plasma atomic emission spectrometry and stratography gravimetry

钽铌化学分析方法 第2部分:钽中铌量的测定 电感耦合等离子体原子发射光谱法和色层分离重量法

Issued on: December 31, 2019 **Implemented on: November 1, 2020**

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

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Foreword

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

--- Part 1. Determination of tantalum in niobium by inductively coupled plasma atomic emission spectrometry;

--- Part 2. Determination of the amount of niobium in tantalum by inductively coupled plasma atomic emission spectrometry and chromatogravimetry;

--- Part 3. Determination of copper content by flame atomic absorption spectrometry;

--- Part 4. Determination of iron content 1,10-diazaphenanthrene spectrophotometry;

--- Part 5. Determination of the amount of molybdenum and tungsten; inductively coupled plasma atomic emission spectrometry;

--- Part 6. Determination of silicon content by inductively coupled plasma atomic emission spectrometry;

--- Part 7. Determination of phosphorus content in niobium 4-methyl-pentanone- [2] Extraction and separation of phosphorus molybdenum blue spectrophotometry and inductively coupled plasma Bulk atomic emission spectroscopy;

--- Part 8. Determination of carbon and sulfur content High-frequency combustion infrared absorption method;

--- Part 9. Determination of iron, chromium, nickel, manganese, titanium, aluminum, copper, tin, lead and zirconium in tantalum by DC arc atomic emission spectrometry;

--- Part 10. Determination of iron, nickel, chromium, titanium, zirconium, aluminum and manganese in niobium by DC arc atomic emission spectrometry;

--- Part 11. Determination of arsenic, antimony, lead, tin and bismuth in niobium by DC arc atomic emission spectrometry;

--- Part 12. Determination of phosphorus content in tantalum Phosphomolybdenum blue

spectrophotometric method for extraction and separation of ethyl acetate;

--- Part 13. Determination of nitrogen content; inert gas fusion thermal conductivity method;

1 Scope

GB/T 15076.2-2019 is the Chinese national standard on methods for chemical analysis of tantalum and niobium - part 2:determination of niobium content in tantalum - inductively coupled plasma atomic emission spectrometry and stratography gravimetry, 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 31 December 2019 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 November 2020. 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.

1 Inductive Coupling Plasma Atomic Emission Spectroscopy "(see Chapter 3, Chapter 1 of the.1994 edition);

--- Expanded the measurement range, the measurement range of Method 1 was changed from "0.0010% ~ 1.50%" to "0.0010% ~ 2.00%"; method The measurement range of the second was changed from " > 1.50% ~ 6.00%" to " > 2.00% ~ 20.00%" (see Chapter 1, Chapter 1 of the.1994 edition) And chapter 9);

--- Removed the reference standard (see Chapter 2 and Chapter 10 of the.1994 edition);

--- Added sample clauses (see Chapter 2);

--- Added precision clauses (see

--- Modify the amount of hydrofluoric acid and nitric acid in the method 2 developing agent from "8mL and 4mL" to "6mL and 6mL" (see 4.2.8,

12.4 of the.1994 edition);

--- Change the amount of sample in Method 2 from "0.1g" to "0.10g and 0.05g" according to different mass fractions (see 4.4.1,.1994 version 14.2);

--- Added test report clauses (see Chapter 5). This section is proposed by China Nonferrous Metals Industry Association. This section is under the jurisdiction of the National Nonferrous Metals Standardization Technical Committee (SAC/TC243). This section was drafted by: Ningxia Dongfang Tantalum Co., Ltd., Guangdong Guangsheng Rare Metal Optoelectronic New Material Co., Ltd., Northwest Rare Metal Materials Research Institute Ningxia Co., Ltd., Xi'an Hantang Analysis and Testing Co., Ltd., Jiujiang Nonferrous Metal

Smelting Co., Ltd., Guangdong Industry Analysis and Testing Center. The main drafters of this section. Zhang Junfeng, Huang Shuang, Yu Hongyan, Chen Hong, Yang Junhong, Xuan Hongjian, Liu Baili, Zhang Tianyi, Hao Wenting, and Wu Zhuokui. The previous versions of the standards replaced by this section are.

--- GB/T 15076.2-1994. Chemical analysis of tantalum and niobium Part 2. Determination of niobium in tantalum Inductively coupled plasma atomic emission spectrometry and Chromatographic separation gravimetric method

2 Samples

2.1 The particle size of tantalum powder is less than 700 μm; the particle size of tantalum carbide is less than 150 μm.

2.2 Tantalum powder should be vacuum dried in advance at 80 °C ~ 150 °C for 4h, cooled to room temperature, and vacuum packed with composite aluminum foil bag; tantalum hydroxide and oxygen Tantalum and potassium fluorotantalate should be dried in advance at 105 °C ~ 110 °C for 2h, placed in a desiccator and cooled to room temperature, and then used.

2.3 The tantalum ingot is sampled from the top 10cm to the middle of the ingot after removing the skin with a planer, and the size of the debris is uniform. Uniform, and the particles should be as small as possible.

3 Method 1-Inductively coupled plasma atomic emission spectrometry

3.1 Principle The sample was dissolved with nitric acid and hydrofluoric acid. In a dilute acid medium, argon plasma was used as the ionization source to directly conduct inductively coupled plasma. Atomic emission spectrometry.

3.2 Reagents and materials Unless otherwise stated, only reagents and laboratory secondary water identified as analytically pure were used in the analysis.

3.2.1 Hydrofluoric acid (rho = 1.14g/mL).

3.2.2 Nitric acid (rho = 1.42g/mL).

3.2.3 Metal tantalum (wTa>=99.99%, wNb<=0.0001%).

3.2.4 Niobium standard storage solution. Weigh 1.000g of metal niobium (wNb>=99.99%) into a 250mL polytetrafluoroethylene beaker, add 10mL hydrofluoric acid (3.2.1), add 10mL nitric acid (3.2.2) dropwise, slowly heat to dissolve until clear, cool to room temperature, transfer to 1000mL In a volumetric flask, dilute to the mark with water and mix well. 1 mL of this solution contains 1 mg of niobium. Store in polyethylene volumetric flasks.

3.2.5 Niobium standard solution. Transfer 10.00mL of niobium standard storage solution

(3.2.4) into a 100mL volumetric flask, and add 2mL of hydrofluoric acid (3.2.1), dilute to the mark with water and mix well. 1 mL of this solution contained 100 μg of niobium. Store in polyethylene volumetric flasks.

3.2.6 Argon (volume fraction is not less than 99.99%).

3.3 Equipment

3.3.1 Inductively coupled plasma atomic emission spectrometer with hydrofluoric acid resistant sampling system, resolution < 0.006nm (at.200nm).