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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 15076.5-2017

Replacing GB/T 15076.5-1994

Methods for chemical analysis of tantalum and niobium - Part5:Determination of molybdenum and tungsten contents - Inductively coupled plasma atomic emission spectrometry

钽铌化学分析方法 第5部分：钼量和钨量的测定 电感耦合等离子体原子发射光谱法

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Foreword

GB/T 15076 "tantalum and niobium chemical analysis method" is divided into 16 parts.

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--- Part 2. Determination of niobium in tantalum, extraction, chromatography - DSPCF spectrophotometry and chromatography - gravimetric method;

--- Part 3. Determination of copper content CTC extraction and separation of sodium diethyl dithiocarbamate spectrophotometry;

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

--- Part 5. Determination of molybdenum content and tungsten content Inductively coupled plasma atomic emission spectrometry;

--- Part 6. Determination of silicon content in tantalum anion exchange separation molybdenum silicate blue spectrophotometry and 4-methyl-pentanone - [2] extraction separation Silicon molybdenum blue spectrophotometry;

--- Part 7. Determination of the amount of phosphorus in niobium 4 - methyl - pentanone - [2] extraction and separation of phosphomolybdate blue spectrophotometry;

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

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

--- Part 12. Determination of phosphorus in tantalum; Ethyl acetate extraction;

Phosphomolybdate blue spectrophotometry;

--- Part 13. Determination of nitrogen; inert gas melting heat conduction method;

--- Part 14. Determination of oxygen; inert gas melting infrared absorption method;

1 Scope

GB/T 15076.5-2017 is the Chinese national standard on methods for chemical analysis of tantalum and niobium - part5:determination of molybdenum and tungsten contents - 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.

GB/T 15076 provisions of this part of tantalum, niobium molybdenum content and tungsten content of the determination. This section applies to tantalum, niobium and its hydroxides, oxides, carbides and potassium fluotitanate potassium molybdenum and tungsten content determination. Measurement range. 0.0005% ~ 3.00%.

2 Method summary

The sample is dissolved with nitric acid and hydrofluoric acid. In a dilute acid medium, argon plasma is used as the ionization source, and the inductively coupled plasma Atomic Emission Spectrometry.

3 Reagents and materials

Unless otherwise indicated, the reagents used in this section are in line with national standards or industry standards excellent grade pure reagent, the water used for two water.

3.1 Nitric acid (p =

1.42 g/mL). Hydrofluoric acid (rho =

3.3 sodium hydroxide solution (200g/L).

3.4 tantalum matrix. WTa >= 99.99%, wW <= 0.00001%, wMo <= 0.00001%.

3.5 niobium matrix .wNb>=99.99%, wW<=0.00001%, wMo<=0.00001%.

3.6 Tungsten standard stock solution Weigh 1.2611g of tungsten oxide pre-dried at 105 °C for 1h and placed in a desiccator to cool to room temperature (wWO3 >= 99.99%) in 250mL Teflon beaker, add 20mL sodium hydroxide solution (3.3), heated to dissolve, cooled, Into a 1000mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 1 mg of tungsten. Store in polyethylene volumetric flask.

3.7 Tungsten standard solution Pipette 10.00mL tungsten standard stock solution (3.6) In a 100mL volumetric flask, add 1.8mL sodium hydroxide solution Liquid (3.3), diluted with water to the mark, mix well. 1 mL of this solution contains 100 μg of tungsten. Store in polyethylene volumetric flask.

3.8 Molybdenum standard stock solution Weigh 1.8403g ammonium molybdate [w (NH4) Mo7O24 · 4H2O >= 99.99%] in a 250mL beaker with a small amount of water Dissolved, into 1000mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 1 mg of molybdenum.

3.9 Molybdenum standard solution Pipette 10.00mL molybdenum standard stock solution (3.8) in 100mL volumetric flask, diluted with water to the mark, and mix. This solution 1 mL contains 100 μg molybdenum.

3.10 Argon, the volume fraction of not less than 99.99%.

4.1 Inductively Coupled Plasma Atomic Emission Spectrometer with Hydrofluoric Acid Injection System, Resolution <

0.006 nm (at.200 nm).

4.2 Recommended elements of the line of choice in Table 1.