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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13747.25-2017

Methods for chemical analysis of zirconium and zirconium alloys - Part 25: Determination of niobium content - 2-(5-bromo-2-pyridylazo)-5-diethylam inophenol spectrophotometry and inductively coupled plasma atomic emission spectrometry

锆及锆合金化学分析方法 第25部分：铌量的测定 5-Br-PADAP分光光度法和电感耦合等离子体原子发射光谱法

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Foreword

GB/T 13747 "zirconium and zirconium alloy chemical analysis method" is divided into 27 parts.

--- Part 1. Determination of tin content potassium iodate titration and phenylfluorone - polyethylene glycol octyl phenyl ether spectrophotometry;

--- Part 2. Determination of iron content 1,10-phenanthroline spectrophotometry and inductively coupled plasma atomic emission spectrometry;

--- Part 3. Determination of nickel content dimethylglyoxime spectrophotometry and inductively coupled plasma atomic emission spectrometry;

--- Part 4. Determination of chromium content Diphenylcarbazide spectrophotometry and inductively coupled plasma atomic emission spectrometry;

--- Part 5. Determination of aluminum content chrome azure S-tetradecyl pyridine chloride spectrophotometry;

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--- Part 7. Determination of manganese content potassium periodate spectrophotometry and inductively coupled plasma atomic emission spectrometry;

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--- Part 9. Determination of magnesium content by flame atomic absorption spectrometry;

--- Part 10. Determination of tungsten content thiocyanate spectrophotometry;

--- Part 11. Determination of molybdenum content thiocyanate spectrophotometry;

--- Part 12. Determination of silicon content Molybdenum blue spectrophotometry;

--- Part 13. Determination of lead content polarographic method;

1 Scope

GB/T 13747.25-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 25: determination of niobium content - 2-(5-bromo-2-pyridylazo)-5-diethylam inophenol spectrophotometry and 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 29 September 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 April 2018. Classification: ICS 77.120.99, CCS H63. 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 section GB/T 13747 specifies the determination of zirconium and zirconium alloy niobium content. This section applies to sponge zirconium, zirconium and zirconium alloy niobium content determination. Method A determination of the range of 0.20% to 3.00%; Setting range .0.050% ~ 3.00%. Method two is the method of arbitration analysis.

2 Method A 5-Br-PADAP spectrophotometry

2.1 Method summary The sample was dissolved with sulfuric acid and ammonium sulfate. In the presence of tartaric acid and EDTA, niobium and 5-Br-PADAP were colored in an acidic medium Complex, absorbance was measured at a spectrophotometer wavelength of 610 nm.

2.2 Reagents Unless otherwise noted, only reagents identified as superior pure and laboratory grade II water were used in the analysis.

2.2.1 Ammonium sulfate.

2.2.2 Sulfuric acid (rho =

2.2.3 tartaric acid solution (240g/L).

2.2.4 tartaric acid solution (60g/L).

2.2.5 ammonia (1 1).

2.2.6 Sulfuric acid (1 19).

2.2.7 Disodium ethylenediaminetetraacetate (EDTA) solution (20g/L). 2.2.8 5-Br-PADAP [2-(5-Br-2-pyridylazo) -5-diethylaminophenol] ethanol solution (

0.3 g/L).

2.2.9 Zirconium base solution Weigh 0.10g of metal zirconium (wZr >= 99.95%) in a 100mL

beaker, add 1.0g ammonium sulfate (2.2.1) 2.0mL sulfuric acid (2.2.2), heated to dissolve, cooled, transferred to.200mL volumetric flask, cooled to room temperature, diluted with water to the mark, and mix. This solution 1 mL contains

0.5 mg zirconium.

2.2.10 Niobium standard stock solution Weigh 0.7153g niobium pentoxide (wNb205 >= 99.95%) in a platinum crucible, add 10g pyrosulfuric acid Potassium was heated and melted on a torch to dissolve completely, cooled, leached with a hot tartaric acid solution (100 g/L), placed in a 500 mL beaker, With constant stirring, the solution was clarified by heating, cooled, transferred to a 500 mL volumetric flask and diluted with tartaric acid solution (100 g/L) to the mark uniform. 1 mL of this solution contains

1.0 mg of niobium.

2.2.11 niobium standard solution Pipette 10.00mL niobium standard stock solution (2.2.10) in 1000mL volumetric flask to tartaric acid solution (2.2.4) diluted to the mark, mix well. 1 mL of this solution contains 10 μg of niobium.

2.2.12 p-nitrophenol solution (1g/L).