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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 15076.13-2017

Replacing GB/T 15076.13-1994

Methods for chemical analysis of tantalum and niobium - Part 13: Determination of nitrogen content - Inert gas fusion thermal conductivity method

钽铌化学分析方法 第13部分：氮量的测定 惰气熔融热导法

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Foreword

GB/T 15076 "Chemical Analysis Method for Poria" is divided into 16 parts.

--- Part 1 . Determination of germanium content in helium inductively coupled plasma atomic emission spectrometry ;

--- Part 2. determination of yttrium in ytterbium extraction, chromatographic separation - DSPCF spectrophotometric method and chromatographic separation gravimetric method; -- Part 3 . Determination of copper content Carbon tetrachloride extraction Separation of sodium diethyldithiocarbamate spectrophotometry ;

--- Part 4. Determination of iron content 1,10-Phenanthroline spectrophotometric method; Part 5 . Determination of molybdenum content and tungsten content -- Inductively coupled plasma atomic emission spectrometry ;

---Part 6. Determination of silicon content in cesium - Anion exchange separation - Molybdenum blue spectrophotometric method and separation with 4-methyl-pentanone-[2] Molybdenum blue spectrophotometry;

--- Part 7. Determination of phosphorus content in barium 4-Methyl-pentanone-[2] Extraction separation Phosphorus-molybdenum blue spectrophotometric method;

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

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

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

--- Part 11. Determination of arsenic, antimony, lead, tin and antimony content in germanium by DC arc carrier fractionation atomic emission spectrometry; Part 12 .

Determination of phosphorus content in barium Ethyl acetate extraction separation of phosphomolybdic blue spectrophotometric method ;

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

--- Part 14. Determination of oxygen content Inert gas fusion infrared absorption method; Part 15 . Determination of hydrogen content ; Inert gas fusion thermal conductivity method ;

--- Part 16. Determination of sodium and potassium - Flame atomic absorption spectrometric method. This part is part 13 of GB/T 15076. This section was drafted in accordance with the rules given in GB/T 1.1-2009. This Part replaces GB/T 15076.13-1994 "Determination of nitrogen in cesium by chemical analysis methods". This section and GB/T 15076.13- Compared with.1994, the main technical content changes as follows:

--- Changed the method of determination, changed from spectrophotometry to inert gas fusion thermal conductivity method;

1 Scope

GB/T 15076.13-2017 is the Chinese national standard on methods for chemical analysis of tantalum and niobium - part 13: determination of nitrogen content - inert gas fusion thermal conductivity method, 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 August 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 specifies the method for the determination of nitrogen in thorium and thorium. This section applies to the determination of nitrogen in cesium and lanthanum. Measurement range. 0.0005% to 0.400%.

2 Method summary

The sample and the flux are added to a high-purity graphite crucible and heated and melted under the protection of an inert gas (helium gas), in which oxygen is combined with carbon in the crucible. A mixture of carbon monoxide and carbon dioxide is formed, and nitrogen is released as nitrogen. Carbon monoxide is partially oxidized to carbon dioxide, one of the gases The carbon oxides and carbon dioxide are absorbed and separated, and the remaining nitrogen enters the thermal conductivity detector with a carrier gas (helium gas). The output signal of the detector is calculated. The nitrogen content is calculated based on

the sample mass.

3 Materials

3.1 Helium. The volume fraction is not less than 99.995%.

3.2 Argon/nitrogen (power gas). The volume fraction is not less than 99.9%.

3.3 High purity nickel foil/nickel capsule. wN<=0.0002%, nickel foil thickness<=0.10mm.

3.4 High-purity graphite crucible.

3.5 Reference material/standard sample. The corresponding reference material should be selected. In principle, the chemical composition of the reference material and the analytical sample is similar.

4 Instrument devices

Inert gas fusion thermal conductivity detection system, including an electrode furnace, carrier gas purification and analysis gas flow conversion system, nitrogen thermal conductivity detector, computer And software control system.

5 Samples

5.1 The particle size of tantalum powder is less than 180μm; tantalum powder should be vacuum dried in advance at 80°C~150°C for 4 hours, cooled to room temperature, and used with composite aluminum foil bag. Empty packaging.

5.2 The powder sample is loaded into a pre-treated and weighed high-purity nickel foil/nickel capsule (3.3) and sealed to remove the air in the high-purity nickel foil/nickel capsule (3.3). The total mass of the sample and the high-purity nickel foil/nickel capsule (3.3) was again weighed to obtain the difference in the mass of the sample. The sample should be analyzed daily.

5.3 The bulk sample was removed from the epidermis and cut from the sample core and processed into pellets with a mass no greater than

0.03 g, weighed, and loaded with high-purity nickel foil/nickel Bags, seals, and purify air in high-purity nickel foil/nickel capsules (3.3).

5.4 The treated sample should not be contaminated prior to analysis and testing. Clean tweezers should be used during the test.