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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 13747.22-2017

Replacing GB/T 13747.22-1992

**Methods for chemical analysis of zirconium and zirconium alloys - Part
22: Determination of oxygen and nitrogen contents - Inert gas
fusion-infrared absorption and thermal conductivity method**

锆及锆合金化学分析方法 第22部分：氧量和氮量的测定 惰气熔融红外吸收法/热
导法

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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;
- Part 6. Determination of copper content 2,9-dimethyl-1,10-phenanthroline spectrophotometry;
- Part 7. Determination of manganese content potassium periodate spectrophotometry and inductively coupled plasma atomic emission spectrometry;
- Part 8. Determination of cobalt content Nitroso R salt spectrophotometry;
- 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 13747.22-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 22: determination of oxygen and nitrogen contents - inert gas fusion-infrared absorption and 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 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.

GB/T 13747 provisions of this part of zirconium and zirconium alloys oxygen and nitrogen determination. This section applies to sponge zirconium, zirconium and zirconium alloy oxygen and nitrogen determination. Measurement range Oxygen 0.010% ~ 0.50%; Nitrogen 0.002% to 0.020%.

2 Method summary

The sample and flux were added to the graphite crucible, heated and melted under the protection of inert gas (helium), in which oxygen was combined with the carbon in the crucible CO and a small amount of CO₂, nitrogen released as N₂. Mixed gas carrier gas carried into the infrared detector, CO or direct detection or conversion After CO₂ was detected, CO₂ direct detection, the detector output signal, the calculation system based on sample quality calculation of oxygen content, the results in mass Number display. Gas in the CO, CO₂ are absorbed in the form of CO₂ separation, the remaining nitrogen with the carrier gas (helium) into the thermal conductivity detector, detection Output signal, the calculation system calculates the nitrogen content according to the sample quality, and the result is shown in mass fraction.

3 Reagents and materials

3.1 acetone or other organic reagents.

3.2 graphite crucible.

3.3 nickel basket.

3.4 anhydrous magnesium perchlorate.

3.6 rare earth copper oxide.

3.7 pure copper wire.

3.8 Helium (volume fraction $\geq 99.99\%$).

3.9 pickling solution. Nitric acid (18) and hydrofluoric acid (1 32) equal volume mixing.

3.10 Standard Substance/Sample Select standard or similar standard/sample with similar composition, oxygen and nitrogen content.

4 Instrument installation

Inert gas melting Infrared - thermal conductivity detection system (including electrode furnace, vacuum devices, gas purification and analysis of gas conversion system, carbon monoxide red External detector and carbon dioxide infrared detector, nitrogen thermal conductivity detector, computer and software control system).

5.1 According to

b) method of processing the surface of the sample.