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

GB/T 13747.12-2019

Replacing GB/T 13747.12-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 12: Determination of silicon content - Molybdenum
blue spectrophotometry**

锆及锆合金化学分析方法 第12部分：硅量的测定 钼蓝分光光度法

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Foreword

GB/T 13747 "Chemical analysis methods for zirconium and zirconium alloys" is planned to be divided into 27 parts.

- Part 1. Determination of tin content potassium iodate titration method and phenylfluorone-polyethylene glycol octylphenyl ether spectrophotometry;
- Part 2. Determination of iron content 1,10-diazaphenanthrene spectrophotometry and inductively coupled plasma atomic emission spectrometry;
- Part 3. Determination of nickel content Butadione oxime 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 Chromazurol S-tetradecylpyridine chloride spectrophotometry;
- Part 6. Determination of copper content 2,9-dimethyl-1,10-diazaphenanthrene spectrophotometry;
- Part 7. Determination of manganese content potassium periodate spectrophotometry and inductively coupled plasma atomic emission spectrometry;
- Part 8. Determination of the amount of drilled nitroso R salt spectrophotometric method;
- Part 9. Determination of magnesium content by flame atomic absorption spectrometry;
- Part 10. Determination of tungsten content thiocyanate spectrophotometric method;
- Part 11. Determination of molybdenum content thiocyanate spectrophotometry;
- Part 12. Determination of silicon content Molybdenum blue spectrophotometry;
- Part 13. Polarographic determination of lead content;

1 Scope

GB/T 13747.12-2019 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 12: determination of silicon content - molybdenum blue spectrophotometry, 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.

This part of GB/T 13747 specifies the determination method of silicon content in zirconium and zirconium alloys. This section applies to the determination of silicon content in sponge zirconium, zirconium and zirconium alloys. Measurement range. 0.0010% ~ 0.040%.

2 Principle

The sample was dissolved with hydrofluoric acid and nitric acid. Fluoride was masked with boric acid. At about pH 1.5, silicon and ammonium molybdate generate yellow silicon molybdenum heteropoly acid, After reducing it to silicon molybdenum blue with a reducing agent, the absorbance was measured at a wavelength of 800 nm by a spectrophotometer.

3 Reagents

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

3.1 Hydrofluoric acid (1 4), excellent grade pure.

3.2 Nitric acid (1 1), excellent grade pure.

3.3 Saturated boric acid solution.

3.4 Potassium permanganate solution (1g/L).

3.5 Ammonium molybdate solution (100g/L). Weigh 50g of ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$ and dissolve it in 500mL of water.

3.6 Tartaric acid solution (200g/L).

3.7 Reducing agent solution. Weigh 20g of sodium sulfite and dissolve it in 160mL of water, and then add 0.4g of 1-amino-2-naphthol-4-sulfonic acid. After adding 40mL of water, it is

now ready to use.

3.8 Silicon standard storage solution. Weigh 1.0697 g of dioxide that was previously burned at 1000 ° C for 30 min and cooled to room temperature in a desiccator. Put silicon (excellent grade) in a platinum crucible, add 5g sodium carbonate and 5g potassium carbonate, and mix well. Melt in 1000 °C high temperature furnace for 20min, take out cold. However, in a polytetrafluoroethylene beaker, immerse the frit with hot water, cool it, and transfer it to a 500mL volumetric flask. Dilute to the mark with water, mix, and store. In a polyethylene bottle. 1 mL of this solution contained 1 mg of silicon.

3.9 Silicon standard solution. Transfer 10.00mL silicon standard storage solution (3.8) to a 1000mL volumetric flask, dilute to the mark with water, mix well, store. Store in a polyethylene bottle. 1 mL of this solution contained 10 µg of silicon.

4 Instruments

Spectrophotometer.

5 Samples

The sample was processed into chips with a length of not more than 5 mm.