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CCS H63



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

GB/T 13747.11-2017

Replacing GB/T 13747.11-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 11: Determination of molybdenum content -
Thiocyanate spectrophotometry**

锆及锆合金化学分析方法 第11部分：钼量的测定 硫氰酸盐分光光度法

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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 13747.11-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 11: determination of molybdenum content - thiocyanate 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 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 Determination of the amount of molybdenum. This section applies to sponge zirconium, zirconium and zirconium molybdenum content determination. Measurement range. 0.0025% to 0.025%.

2 Method summary

The sample is decomposed with sulfuric acid-ammonium sulfate, and the molybdenum is reduced to pentavalent with tin dichloride in the presence of iron (III) in a sulfuric acid medium to react with thiocyanate Orange-red complex. Extract with ethyl acetate and measure its absorbance at a wavelength of 470 nm of the spectrophotometer.

3 Reagent

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

3.1 Ammonium sulfate, analytical grade.

3.2 Sulfuric acid ($\rho = 1.84/\text{mL}$), analytical grade.

3.3 Sulfuric acid (1 4).

3.4 ammonium ferric sulfate (5g/L).

3.5 Potassium thiocyanate (50 g/L).

3.6 Stannous chloride solution (100g/L) Weigh 50g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$), dissolved in 50mL of hydrochloric acid ($\rho = 1.19\text{g/mL}$) In water, dilute with water to 500mL, mix well.

3.7 Ethyl acetate, analytical grade.

3.8 Molybdenum standard stock solution Weigh 0.5000g metal molybdenum ($w_{\text{Mo}} \geq 99.9\%$), placed in 400mL beaker, add 50mL sulfuric acid (11), in the case of heating, dropping nitric acid ($\rho = 1.42\text{g/mL}$) dissolved, continue heating to take sulfuric acid smoke. Cool, add 20mL water, add Heat dissolves salts. Moved into 500mL volumetric flask, diluted with water to the mark, mix well. This solution 1 mL contains

1.0 mg molybdenum.

3.9 Molybdenum standard solution Pipette 10.00mL molybdenum standard stock solution (3.8), in 1000mL volumetric flask, diluted with water to the mark, mixed uniform. 1 mL of this solution contains 10 μg of molybdenum.

4 Instruments

Spectrophotometer.

5 Sample

5.1 Sampling Sponge zirconium, zirconium and zirconium alloy sampling should be in accordance with the promulgation of the corresponding standard methods.