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

GB/T 13747.2-2019

Replacing GB/T 13747.2-1992

**Methods for chemical analysis of zirconium and zirconium alloys - Part 2:
Determination of iron content - 1,10-phenanthroline spectrophotometry and
inductively coupled plasma atomic emission spectrometry**

锆及锆合金化学分析方法 第2部分：铁量的测定 1,10-二氮杂菲分光光度法和电感
耦合等离子体原子发射光谱法

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Foreword

GB/T 13747 "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 13747.2-2019 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 2: determination of iron content - 1,10-phenanthroline 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 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 method for determination of iron content in zirconium and zirconium alloys. This section applies to the determination of iron content in sponge zirconium, zirconium and zirconium alloys. Measurement range. 0.010% ~ 0.40%. Method one for arbitration method.

2 Method 1. 1,10-diazaphenanthrene spectrophotometry

2.1 Principle The sample was decomposed with sulfuric acid-ammonium sulfate, zirconium was complexed with citric acid, and Fe³ was reduced to Fe² with hydroxylamine hydrochloride. At around pH 6, Fe² and 1,10-Diazaphenanthrene formed a red complex, and its absorbance was measured at a spectrophotometer wavelength of 510 nm.

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

2.2.1 Hydrochloric acid (1 1).

2.2.2 Ammonium sulfate.

2.2.3 sulfuric acid ($\rho = 1.84\text{g/mL}$).

2.2.4 Hydroxylamine hydrochloride solution (100g/L).

2.2.5 Citric acid solution (500g/L).

2.2.6 Acetic acid-ammonium acetate buffer solution. Weigh 500g of ammonium acetate to dissolve in water, add 20mL of glacial acetic acid, and dilute to 1000mL with water. Mix well.

2.2.7 Ammonia (1 1). 2.2.8 1,10-Diazaphenanthrene solution (5g/L). Weigh 0.5g of 1,10-diazaphenanthrene and dissolve it in 20mL absolute ethanol, and dilute with water to

100mL, mix well and store in brown bottle.

2.2.9 Iron standard storage solution. Weigh 1.000g of metallic iron ($w_{Fe} \geq 99.99\%$), place it in a 200mL beaker, and add 30mL hydrochloric acid (2.2.1), heat to complete decomposition at low temperature, cool, transfer to 1000mL volumetric flask, dilute to the mark with water, and mix well. 1mL of this solution contains 1mg iron.

2.2.10 Iron Standard Solution. Transfer 10.00mL iron standard storage solution (2.2.9) into a 100mL volumetric flask, dilute to the mark with water, and mix uniform. 1 mL of this solution contained 100 μ g of iron.

2.3 Instrument Spectrophotometer.

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