

ICS 77.120.99

CCS H63



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

GB/T 13747.8-2017

Replacing GB/T 13747.8-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 8: Determination of cobalt content - Nitroso-R-salt
spectrophotometry**

锆及锆合金化学分析方法 第8部分：钴量的测定 亚硝基R盐分光光度法

Issued on: September 29, 2017

Implemented on: April 1, 2018

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

Table of Contents

- 1 Scope
- 2 Method summary
- 3 Reagent
 - 3.1 Metal zirconium (wZr 99.95%, wCo < 0.0001%). Hydrofluoric acid ($\rho =$
 - 3.3 Nitric acid ($p =$
 - 3.4 Sulfuric acid ($p =$
 - 3.5 Ammonia ($p =$

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 13747.8-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 8: determination of cobalt content - nitroso-r-salt 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.

This section GB/T 13747 specifies the determination of cobalt content in zirconium and zirconium alloys. This section applies to sponge zirconium, zirconium and zirconium alloy determination of cobalt content. Measurement range. 0.0005% ~ 0.0050%.

2 Method summary

The sample was dissolved with hydrofluoric acid and nitric acid. In the presence of citric acid, pH8 or so, alpha-nitroso-beta-naphthol and chloroform extraction of cobalt. In sodium acetate medium cobalt and nitroso R salt red complex was measured at a spectrophotometer wavelength 520nm absorbance measured.

3 Reagent

Unless otherwise specified, only reagents that are identified as analytically pure and laboratory-grade water are used in the analysis.

3.5 Ammonia (p =

0.90 g/mL).

3.6 trichloromethane.

3.7 Hydrochloric acid (1 1).

3.8 Nitric acid (1 1).

3.9 Ammonia (1 1).

3.10 Ammonium citrate solution Weigh 50g ammonium citrate dissolved in 100mL water, add 70mL ammonia (3.5).

3.11 mixed acid in a 100mL volumetric flask by adding 10mL hydrochloric acid (3.7), 1mL nitric acid (3.8), diluted with water to the mark, and mix.

3.12 sodium acetate solution Weigh 50g of sodium acetate ($\text{NaCH}_3\text{COO} \cdot 3\text{H}_2\text{O}$) was dissolved in 100mL of water.

3.13 nitrosyl R salt solution (2g/L). Weigh 0.2g nitrosyl R salt was dissolved in 100mL of water. 3.14 alpha-nitroso-beta-naphthol solution (2g/L) Weigh 0.2g alpha-nitroso beta-naphthol dissolved in 5mL sodium hydroxide solution (40g/L) In the insoluble matter was filtered, diluted with water to 100mL.

3.15 cobalt standard stock solution Weigh 0.1000g cobalt metal in a 150mL beaker, with 25mL water and 10mL nitric acid (3.3) solution Solution, cooling. Into a 1000mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 100 μg of cobalt.

3.16 cobalt standard solution Pipette 25.0mL cobalt standard stock solution (3.15) in 250mL volumetric flask, diluted with water to the mark, and mix. 1 mL of this solution contains 10 μg of cobalt.