

ICS 77.120.99

CCS H14



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

GB/T 13747.6-2019

Replacing GB/T 13747.6-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 6: Determination of copper content -
2,9-Dimethyl-1,10-phenanthroline spectrophotometry**

锆及锆合金化学分析方法 第6部分：铜量的测定 2,9-二甲基-1,10-二氮杂菲分光光度法

Issued on: June 4, 2019

Implemented on: January 1, 2020

**Issued by: State Administration for Market Regulation;
Standardization Administration of China**

Table of Contents

- 1 Scope
- 2 Principle
- 3 Reagents
 - 3.1 Hydrofluoric acid (=
 - 3.2 Nitric acid (=
- 4 Instrument and Equipment...
- 5 Samples...
- 6 Test Procedures...
 - 6.4 Determination
 - 6.5 Draw a Working Curve
- 7 Test Data Processing
- 8 Precision...
- 9 Test Report...

Foreword

GB/T 13747 Methods for Chemical Analysis of Zirconium and Zirconium Alloys is divided into 27 parts:

- Part 1: Determination of Tin Content - Potassium Iodate Titration and Phenylfluorone-polyethylene Glycoloctyl Phenyl Ether Spectrophotometry;
- Part 2: Determination of Iron Content - 1,10-phenanthroline Spectrophotometric Method;
- Part 3: Determination of Nickel Content - Dimethylglyoxime Spectrophotometric Method;
- Part 4: Determination of Chromium Content - Diphenylcarbazide Spectrophotometric Method;
- Part 5: Determination of Aluminum Content - Chronic Azurol-S- tetradecylpyridine Chloride Spectrophotometry;
- Part 6: Determination of Copper Content - 2,9-Dimethyl-1,10-phenanthroline Spectrophotometry;
- Part 7: Determination of Manganese Content - Potassium Periodate Spectrophotometric Method;
- Part 8: Determination of Cobalt Content - Nitroso-R-salt Spectrophotometry;

- Part 9: Determination of Magnesium Content - Flame Absorption Spectrometric Method;
- 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 Spectrophotometric Method;
- Part 13: Determination of Lead Content - Polarography;

1 Scope

GB/T 13747.6-2019 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 6: determination of copper content - 2,9-dimethyl-1,10-phenanthroline 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 4 June 2019 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 January 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 stipulates the determination method of copper content in zirconium and zirconium alloys. This Part is applicable to the determination of copper content in zirconium sponge, zirconium and zirconium alloys. The range of determination is: 0.0010% ~ 0.025%.

2 Principle

Use hydrofluoric acid to dissolve specimen. Use tartaric acid to form zirconium complex. Use hydroxylamine hydrochloride to reduce copper into cuprous ion state. At pH 4 ~ 6, use trichloromethane to extract copper and colored complex, which is formed with 2,9-dimethyl-1,10-phenanthroline. At the wavelength of 460 nm, use a spectrophotometer to measure its absorbance.

3 Reagents

Unless it is otherwise stipulated, the analysis shall merely adopt reagents that are confirmed to be analytically pure and secondary laboratory water.

3.1 Hydrofluoric acid (=

1.15 g/mL, excellent grade of purity).

3.2 Nitric acid (=

1.42 g/mL, excellent grade of purity).

3.3 Trichloromethane.

3.4 Absolute ethanol.

3.5 Ammonia water (1 + 1).

3.6 Boric acid saturated solution.

3.7 Tartaric acid solution (200 g/L).

6.2 Parallel Test Conduct two parallel tests. Take the average value.

6.3 Blank Test Conduct blank test of the accompanying specimen.

6.4 Determination

6.4.1 Place the specimen (6.1) in a 100 mL plastic beaker, then, add 15 mL of water. Add 2 mL of hydrofluoric acid (3.1) in several times. Wait till the specimen completely dissolves, then, respectively add 1 mL of nitric acid (3.2), 25 mL of boric acid saturated solution (3.6) and 10 mL of tartaric acid solution (3.7). Use ammonia water (3.5) to adjust the pH of the solution to be 4 ~ 6. Then, transfer it into a 100 mL volumetric flask. Use water to dilute to the scale, then, shake it up. Transfer-take

20.00 mL of test solution, then, place it in a 100 mL separating funnel.

6.4.2 Add 5 mL of hydroxylamine hydrochloride solution (3.8), then, place it for 15 min. Add

10.0 mL of neocuproine ethanol solution (3.9); add

10.0 mL of trichloromethane (3.3). Conduct extraction for 30 s. After placing it still for stratification, transfer the organic phase into a dry 25 mL colorimetric tube. Add

3.0 mL of absolute ethanol (3.4), then, mix it up.

6.4.3 Transfer part of the organic phase into a 2 cm absorption vessel. Take the blank solution of the accompanying specimen as a reference. At the wavelength of 460 nm, use a spectrophotometer to measure its absorbance. Obtain the corresponding copper content through working curve.

6.5 Draw a Working Curve

6.5.1 Accurately transfer-take 0 mL,

0.20 mL,

0.50 mL,

2.00 mL of copper standard solution (3.11). Respectively place them in a group of 100 mL separating funnels. Add 5 mL of boric acid saturated solution (3.6),

0.4 mL of hydrofluoric acid (3.1) and 2 mL of tartaric acid solution (3.7). Use ammonia water (3.5) to adjust the pH of the solution to be 4 ~ 6; add water to 20 mL. The following analytical procedures shall comply with 6.4.2.

6.5.2 Transfer part of the organic phase into a 2 cm absorption vessel. Take reagent blank as a reference. At the wavelength of 460 nm, use a spectrophotometer to measure its absorbance. Take copper content as the x-coordinate; take its absorbance as the y-coordinate; draw a working curve.