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

GB/T 13747.5-2019

Replacing GB/T 13747.5-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 5: Determination of aluminium content - Chronic
azurol-S-tetradrcylpyridine chloride spectrophotometry**

锆及锆合金化学分析方法 第5部分：铝量的测定 铬天青S-氯化十四烷基吡啶分光
光度法

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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 - Chrome 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 13747.5-2019 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 5: determination of aluminium content - chronic azurol-s-tetradecylpyridine chloride 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 May 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 aluminum content in zirconium and zirconium alloys. This Part is applicable to the determination of aluminum content in zirconium sponge, zirconium and zirconium alloys. The range of determination is: 0.0025% ~ 0.10%.

2 Principle

Use sulfuric acid and ammonium sulfate to dissolve specimen. Use N-benzoyl-N-phenylhydroxylamine trichloromethane solution to extract and separate the main element zirconium, and interference elements: hafnium, iron and titanium. After the separation, use o-phenanthroline and ascorbic acid to mask the residual iron. In acetic acid-sodium acetate medium (pH 5.8), aluminum and chronic azurol-S- tetradecylpyridine chloride form colored complex. At the wavelength of 630 nm, use a spectrophotometer to measure its absorbance. If the sample contains a small amount of uranium and thorium, sodium carbonate solution may be added during color- developing, so as to eliminate the influence.

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 Ammonium sulfate (excellent grade of purity).

3.2 Sulfuric acid (=

1.84 g/mL, excellent grade of purity).

3.3 Nitric acid (=

1.42 g/mL, excellent grade of purity).

3.4 Hydrochloric acid (=

1.19 g/mL, excellent grade of purity).

3.5 Trichloromethane.

6.4.2 In accordance with Table 1, transfer-take the solution. Place it in a 100 mL separating funnel. Add 10 mL of N-benzoyl-N-phenylhydroxylamine trichloromethane solution (3.13); violently oscillate it for 2 min. After placing it still for stratification, discard the organic phase, then, repeat extraction for three times. Add 10 mL of trichloromethane (3.5); start oscillation for

0.5 min. After placing it still for stratification, discard the organic phase. Place the aqueous phase into a 50 mL beaker, then, use a small amount of water to rinse the separating funnel. Combine the rinsing solution into the beaker. Conduct low-temperature heating, till it emits white smoke. Then, take it down. While it is still hot, along the wall of the beaker, add 20 drops of nitric acid (3.3). Continue to heat it up, till it emits dense white smoke, then, take it down. Cool it down, then, along the wall of the beaker, add a small amount of water, then, mix it up. Use micro-heat to dissolve salt. After cooling it down, use water to transfer it into a 50 mL volumetric flask; the volume of the solution shall be around 15 mL.

6.4.3 Add 2 drops of 2, 4-dinitrophenol solution (3.11). Use ammonia water (3.7) to adjust it, till the solution turns yellow. Then, add drops of hydrochloric acid (3.6), till such yellow disappears. Add 2 mL of o-phenanthroline solution (3.9), then, shake it up. Add 2 mL of ascorbic acid solution (3.10), then, shake it up. Along the wall of the beaker, add

5.0 mL of azulol-S-tetradecylpyridine chloride solution (3.8), then, shake it up. Add 5 mL of acetic acid-sodium acetate buffer solution (3.12). Use water to dilute to the scale, then, shake it up.

6.4.4 After placing the solution for 20 min, transfer part of the solution into a 1 cm absorption vessel. Take the blank solution of the accompany specimen as a reference. At the wavelength of 630 nm, use a spectrophotometer to measure its absorbance. Through the working curve, find the corresponding aluminum content.

6.5 Draw a Working Curve Accurately transfer-take 0 mL,

0.20 mL,

0.40 mL,

0.60 mL,

0.80 mL and

1.00 mL of aluminum standard solution B (3.16). Respectively place them in a group of 50 mL volumetric flasks. Add water to reach the volume of around 15 mL. The following analytical procedures shall comply with 6.4.3. After placing the solutions for 20 min, transfer them into 1 cm absorption vessel. Take reagent blank as a reference. At the wavelength of 630 nm, use a spectrophotometer to measure it. Take the aluminum content as the x-coordinate; take the absorbance as the y-coordinate; draw a working curve.