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

GB/T 13747.1-2017

Replacing GB/T 13747.1-1992

**Methods for chemical analysis of zirconium and zirconium alloys - Part 1:
Determination of tin content - Potassium iodate titration and
phenylfluorone-polyethylene glycoloctyl phenyl ether spectrophotometry**

**锆及锆合金化学分析方法 第1部分：锡量的测定 碘酸钾滴定法和苯基荧光酮-聚乙
二醇辛基苯基醚分光光度法**

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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 13747.1-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 1: determination of tin content - potassium iodate titration and phenylfluorone-polyethylene glycol octyl phenyl ether 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 tin content. This section applies to sponge zirconium, zirconium and zirconium alloy determination of tin content. Potassium iodate titration range 0.50% ~ 4.00%; phenyl Fluorone - polyethylene glycol octyl phenyl ether spectrophotometric determination of the range of 0.0010% to 0.045%.

2 Methods Potassium iodate titration

2.1 Method summary The sample is decomposed with ammonium sulfate-ammonium sulfate, and the impurity elements are masked with fluoride ions in a hydrochloric acid medium. Tin (IV) is reduced to tin (II) using aluminum flakes, To starch solution as an indicator, titration with potassium iodate standard titration solution until the solution showed a blue end.

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

2.2.1 Ammonium sulfate.

2.2.2 Ammonium fluoride.

2.2.3 aluminum (wAl $\geq$ 99.5%).

2.2.5 Hydrochloric acid (p =

2.2.6 saturated sodium bicarbonate solution.

2.2.7 tin standard solution Weigh 1.0000g metal tin (wSn $\geq$ 99.95%) placed in 250mL beaker, add 100mL hydrochloric acid (1 1) Dissolve and dissolve in low temperature, after

cooling, transfer to 1000mL volumetric flask, dilute with water to the mark, and mix 1mL of this solution with 1.0mg tin.

2.2.8 potassium iodate standard titration solution [$c(1/6KIO_3) = 0.02\text{mol/L}$].

2.2.8.1 Preparation Weigh 1g sodium hydroxide, 10g potassium iodide was dissolved in 200mL of water, weighed 0.7133g potassium iodate was dissolved in the solution, Into 1000mL volumetric flask, diluted with water to the mark, mix well. (The water needs to be boiled before use.)

2.2.8.2 Calibration. Pipette 3 parts 20.00mL tin standard solution (2.2.7), were placed in three 500mL conical flask, each added 5g sulfur Acid ammonium (2.2.1), 10mL sulfuric acid (2.2.4), the following 2.4.4.2 ~ 2.4.4.4. Parallel calibration of potassium iodate consumed standard titration solution Liquid (2.2.8) The volume of the difference should not exceed 0.10mL, whichever is the average.

2.2.8.3 with the calibration to do a blank test. Calculate the titration coefficient of tin for potassium iodate standard titration solution (2.2.8) according to formula (1). $f = m_1 V_1 - V_0$
(1) In the formula. f

--- potassium iodate standard titration solution (2.2.8) titration coefficient of tin in units of grams per milliliter (g/mL);