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

GB/T 13747.13-2017

Replacing GB/T 13747.13-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 13: Determination of lead content - Polarography**

锆及锆合金化学分析方法 第13部分：铅量的测定 极谱法

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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 13747.13-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 13: determination of lead content - polarography, 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 alloy determination of the amount of lead. This section applies to sponge zirconium, zirconium and zirconium alloy determination of the amount of lead. Measurement range. 0.0005% ~ 0.030%.

2 Method summary

The sample is dissolved in hydrofluoric acid, the zirconium is masked with ammonium citrate, and the mixture is extracted with dithizone-chloroform in an ammonia medium having a pH of 9 to

10 Lead, measured by linear sweep polarography in dilute hydrochloric acid at the bottom.

3 Reagents and materials

Unless otherwise noted, only reagents identified as superior pure and laboratory grade II water were used in the analysis.

3.2 Nitric acid ($\rho =$

3.3 Perchloric acid ($\rho = 1.68\text{g/mL}$).

3.4 Ammonia ($\rho = 0.9\text{g/mL}$) Liquid ammonia is absorbed and purified by distilled water.

3.5 Hydrochloric acid [$c(\text{HCl}) =$

0.5 mol/L].

3.6 triammonium citrate solution (300g/L). Weigh 150g of triammonium citrate dissolved in about 300mL water was added 100mL saturated boric acid Liquid, diluted with water to

500mL volume, mix.

3.7 Dithizone - chloroform extract (0.5g/L). Weigh 2.5g dithizone dissolved in about 400mL of chloroform, into 1000mL points Liquid funnel. Add.200mL ammonia (1 9), shake 3min. After stratification, the organic phase was placed in another separatory funnel and the aqueous phase was added 500mL beaker retained. The organic phase was added.200mL ammonia (1 9) extracted once, the organic phase was discarded. The combined aqueous phase, with degreasing Cotton filter to remove insoluble material. Hydrochloric acid was added dropwise to acidify the aqueous phase, and the dithizone precipitate was completely transferred to a separatory funnel. A total of 500mL three times in three The chloroform was extracted by shaking to dissolve the dithizone in the organic phase. The combined organic phases were washed once with 500 mL of water and the organic phase was in a brown bottle Save sealed. This organic phase 1L containing 5g dithizone, when used to dilute the preparation of chloroform.

3.8 Ammonium citrate - aqueous ammonia washing solution Each 100mL solution contains 3g ammonium citrate, 1mL ammonia (3.4).

3.9 Lead standard solution A. Weigh 0.1000g metal lead ($w_{Pb} \geq 99.9\%$) was dissolved in 30mL of nitric acid (1 2), heated to remove nitrogen oxygen Chemical, cooling. Into a 1000mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 100 μg lead.

3.10 Lead standard solution B. Pipette 50.00mL lead standard solution A (3.9) in 500mL volumetric flask, diluted with water to the mark, and mix. 1 mL of this solution contains 10 μg of lead.

3.11 nitrogen (volume fraction $\geq 99.99\%$).

Polarographic analyzer. Droplet mercury electrode, mercury pool anode or three-electrode system.