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

GB/T 13747.17-2017

Replacing GB/T 13747.17-1992

**Methods for chemical analysis of zirconium and zirconium
alloys - Part 17: Determination of cadmium content -
Polarography**

锆及锆合金化学分析方法 第17部分：镉量的测定 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 13747.17-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 17: determination of cadmium 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 alloys cadmium determination. This section applies to sponge zirconium, zirconium and zirconium alloy determination of cadmium content. Determination of the range of 0.00002% to 0.00050%.

2 Method summary

The sample is dissolved in hydrofluoric acid and the zirconium is masked with ammonium citrate, extracted with dithizone-trichloromethane in an ammoniacal medium of pH 9 to pH 10 Cadmium was separated and the cathodic secondary derivative wave height of cadmium was measured by oscillopolarography in dilute hydrochloric acid at the bottom. When the lead content is more than 0.005%, it will interfere with the determination of cadmium. Add the same amount of lead as the sample solution to the standard series of solutions To eliminate interference.

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 absorption by distilled water purification.

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

0.5 mol/L].

3.6 triammonium citrate solution (300g/L). Weigh 150g of ammonium citrate dissolved in about 300mL of water, add 100mL boric acid (excellent Pure) saturated solution, diluted with water to 500mL volume, mix.

3.7 Dithizone - chloroform extract (0.5g/L), weighed 2.5g dithizone dissolved in about 400mL of chloroform, into 1000mL Separatory funnel. Add.200mL ammonia (1 9), shake 3min, stand stratification, the organic phase into another separatory bucket, water phase Keep in 500mL beaker. The organic phase was added.200mL ammonia (1 9) extracted once, the organic phase was discarded. The combined aqueous phase, with off Insoluble cotton filter 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 two 500mL Trichloromethane was extracted by shaking to dissolve the dithizone in the organic phase. The combined organic phase was washed once with 500 mL of water. Organic phase in brown bottle Sealed in the preservation. The organic phase 1L contains 5g of dithizone, when used with chloroform diluted to 0.5g/L.

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

3.9 Cadmium standard stock solution Weigh 0.1000g metal cadmium ($w_{Cd} \geq 99.9\%$) dissolved in 20mL hydrochloric acid (1 1) heated to cool down Into 1000mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 100 μg of cadmium.

3.10 cadmium standard solution Pipette 10.00mL cadmium standard stock solution (3.9) in 1000mL volumetric flask, diluted with water to the mark, mixed uniform. 1 mL of this solution contains 1 μg of cadmium.

3.11 lead standard solution Weigh 0.1000g lead ($w_{Pb} \geq 99.99\%$), 20mL nitric acid (12), dissolved by heating, after cooling Into a 1000mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 100 μg lead.