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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 13747.14-2017

Replacing GB/T 13747.14-1992

Methods for chemical analysis of zirconium and zirconium alloys - Part 14: Determination of uranium content - Polarography

锆及锆合金化学分析方法 第14部分：铀量的测定 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 13747.14-2017 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloys - part 14: determination of uranium 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 Determination of uranium content. This section applies to sponge zirconium, zirconium and zirconium alloy determination of uranium content. Measuring range. 0.00010% to 0.00050%.

2 Method summary

Hydrofluoric acid was used to dissolve the sample, and the nitric acid was added dropwise to dissolve the insoluble carbides or nitrides. The mixture was extracted with tributyl phosphate-toluene in a nitric acid medium Separation of uranium. In the presence of copper and iron reagents, oscillopolarographic uranium adsorption catalytic wave was measured.

3 Reagents and materials

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

3.1 Hydrofluoric acid (rho = 1.15g/mL), excellent grade pure.

3.2 Nitric acid (rho = 1.42g/mL), excellent grade pure.

3.3 perchloric acid (rho = 1.68g/mL), excellent grade pure.

3.4 Nitric acid (1 3).

3.5 Nitric acid (1 1).

3.6 Disodium ethylenediaminetetraacetate solution (100g/L).

3.7 Ammonium nitrate solution (700g/L).

3.8 Tributylphosphate (TBP) - toluene extractant 60 mL of TBP was mixed with 500 mL of toluene in a 1000 mL separatory funnel each time Add 120mL ammonium nitrate solution

(50g/L), extracted twice, add 120mL water extraction once, add 120mL nitric acid (3.4) extraction once. Each extraction oscillation 3min, standing stratification, discard the aqueous phase. The organic phase is stored in grinding mouth reagent bottles.

3.9 washing solution Weigh 13g ethylenediaminetetraacetic acid disodium dissolved in 500mL hot water, add 135g of ammonium nitrate, then add 60mL nitric acid (3.2), diluted with water to 1000mL volume, mix.

3.10 Mixed base solution Weigh 47.6g sodium acetate ($NaCH_3COO \cdot 3H_2O$), 12.5g disodium ethylenediaminetetraacetate, 3g ammonium oxalate, add Hot in 400mL of water, after cooling add 20mL glacial acetic acid, diluted with water to 500mL volume, and mix.

3.11 copper and iron reagent solution (0.5g/L). Weigh 0.05g copper and iron reagent was dissolved in 100mL of water, with the time available.

3.12 uranium standard stock solution Weigh 0.1179g After the 105 °C drying 1h and cooled to room temperature of trisodium uranium (U_3O_8) in 100mL beaker, add 10mL nitric acid (3.5) heated until completely dissolved, cooled and moved into 1000mL volumetric flask, diluted with water to Scale, mix well. 1 mL of this solution contains 100 μg of uranium.

3.13 uranium standard solution Pipette 10.00mL uranium standard stock solution (3.12) in 1000mL volumetric flask, diluted with water to the mark, mixed uniform. 1 mL of this solution contains 1 ug of uranium.

4 Instruments

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