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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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Replacing GB/T 13747.4-1992

Methods for chemical analysis of zirconium and zirconium alloysPart 4: Determination of chromium content - Diphenylcarbazide spectrophotometry and inductively coupled plasma atomic emission spectrometry

锆及锆合金化学分析方法 第4部分：铬量的测定 二苯卡巴肼分光光度法和电感耦合等离子体原子发射光谱法

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Foreword

GB/T 13747 "Methods for Chemical Analysis of Zirconium and Zirconium Alloys" is planned to be divided into 27 parts.

1.Determination of the amount of tin potassium iodate titration and phenylfluorone-polyethylene glycol octylphenyl ether spectrophotometry;

2.Determination of iron content 1,10-phenanthroline spectrophotometry and inductively coupled plasma atomic emission spectrometry;

3.Determination of nickel content diacetyl oxime spectrophotometry and inductively coupled plasma atomic emission spectrometry;

4.Determination of chromium content Diphenylcarbazide spectrophotometry and inductively coupled plasma atomic emission spectrometry;

5.Determination of aluminum content Chromazurol S-tetradecylpyridinium chloride spectrophotometric method;

6.Determination of copper content 2,9-dimethyl-1,10-phenanthroline spectrophotometry;

7.Determination of manganese content, potassium periodate spectrophotometry and inductively coupled plasma atomic emission spectrometry;

8.Determination of Drill Volume Nitroso R Salt Spectrophotometry;

9.Determination of magnesium content by flame atomic absorption spectrometry;

10.Determination of Tungsten Content Thiocyanate Spectrophotometric Method;

11.Determination of molybdenum content thiocyanate spectrophotometric method;

12.Determination of silicon content molybdenum blue spectrophotometry;

13.Determination of lead content by polarography;

1 Scope

GB/T 13747.4-2020 is the Chinese national standard on methods for chemical analysis of zirconium and zirconium alloyspart 4: determination of chromium content - diphenylcarbazide spectrophotometry and inductively coupled plasma atomic emission spectrometry, 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 6 March 2020 by the China Nonferrous Metals Industry Association, and has been in force since 1 February 2021. 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 specifies the method for determining the chromium content in zirconium and zirconium alloys. This section applies to the determination of chromium content in sponge zirconium, zirconium and zirconium alloys. Measuring range. 0.0020%~0.20%. Method two is arbitration Analytical method.

2 Method-Diphenylcarbazide spectrophotometry

2.1 Principle The sample was dissolved with sulfuric acid and ammonium sulfate. Use potassium permanganate to oxidize chromium. In the presence of urea, excess potassium permanganate is separated with sodium nitrite. It generates a red-purple complex with diphenylcarbazide and measures its absorbance at a wavelength of 540nm in a spectrophotometer.

2.2 Reagents Unless otherwise specified, only reagents and laboratory secondary water confirmed to be analytically pure are used in the analysis.

2.2.1 Ammonium sulfate.

2.2.2 Sulfuric acid (rho=1.84g/mL).

2.2.3 Sulfuric acid (1 4).

2.2.4 Potassium permanganate solution (30g/L).

2.2.5 Urea solution (100g/L).

2.2.6 Sodium nitrite solution (10g/L).

2.2.7 Diphenylcarbazide ethanol solution. Weigh 0.50g of diphenylcarbazide, dissolve it in 100mL ethanol, and store it in a brown bottle (within one week) effective).

2.2.8 Chromium standard storage solution. Weigh 0.2829g of potassium dichromate (reference reagent) that has been dried at 105°C for 2 hours, dissolve it in water, and transfer it into In a 1000mL volumetric flask, dilute to the mark with water and mix well. This solution 1mL contains 100μg chromium.

2.2.9 Chromium standard solution. Pipette 10.00mL chromium standard stock solution (2.2.8) into a 100mL volumetric flask, dilute to the mark with water, and mix uniform. This solution 1mL contains 10μg chromium.

2.3 Apparatus Spectrophotometer.

2.4 Sample Process the sample into chips with a length not greater than 5mm.

2.5 Test procedure

2.5.1 Sample Weigh the sample (2.4) according to Table 1, accurate to 0.0001g.

2.5.2 Parallel test Do two tests in parallel and take the average value.

2.5.3 Blank test Do a blank test with the sample.

2.5.4 Determination

2.5.4.1 Place the sample (2.5.1) in a 150mL beaker, add 6g of ammonium sulfate (2.2.1) and 10mL of sulfuric acid (2.2.2), cover with a watch glass, Heat to complete decomposition, cool to room temperature, add about 30mL of water to dissolve the salts, transfer to a 100mL volumetric flask, dilute to the mark with water, Mix well.

2.5.4.2 When the chromium content is 0.0020%~0.020%, pipette 10.00mL test solution into a 150mL beaker, add 1.0mL sulfuric acid (2.2.3) and 39mL water; when the chromium content is greater than 0.020%~0.20%, pipette 5.00mL test solution into a 150mL beaker, add 2.0mL Sulfuric acid (2.2.3) and 43mL water.

2.5.4.3 Add potassium permanganate solution (2.2.4) dropwise until the solution turns into a stable red and excess 2 to 3 drops, then slowly boil for a few minutes to make the chromium oxide Completely. After cooling to room temperature, add 20 mL of urea solution (2.2.5), shake well, add dropwise sodium nitrite solution (2.2.6) to make potassium permanganate The magenta disappears, shake until the bubbles stop, transfer to a 100mL volumetric flask, cool to room temperature with running water, add 5mL diphenylcarbazide

ethanol Solution (2.2.7), dilute to the mark with water, mix well, and place for 5 min.

2.5.4.4 Pipette part of the solution into a 1cm cuvette, use the blank solution with the sample as a reference, and use the spectrophotometer with a wavelength of 540nm At the place, measure its absorbance. Find the corresponding chromium content from the working curve.

2.5.5 Drawing of working curve

2.5.5.1 Pipette 0mL, 0.50mL, 1.00mL, 3.00mL, 5.00mL, 7.00mL, 10.00mL chromium standard solution (2.2.9), Do not place it in a set of 150mL beakers, add 4.0mL sulfuric acid (2.2.3), and dilute with water to about 50mL volume. Follow

2.5.5.2 Pipette part of the solution into a 1cm cuvette, use the reagent blank solution as a reference, and measure at the wavelength of 540nm on the spectrophotometer. Its absorbance. Use chromium content as the abscissa and absorbance as the ordinate to draw a working curve.

2.6 Test data processing The chromium content is calculated as the mass fraction wCr of chromium, calculated according to formula (1).

2.7 Precision

2.7.1 Repeatability The measured values of two independent test results obtained under repeatability conditions are within the range of the average value given in Table 2. The absolute difference does not exceed the repeatability limit (r), and the case of exceeding the repeatability limit (r) does not exceed 5%. Repeatability limit (r) adopts the line according to the data in Table

2 Obtained by sexual interpolation or extension method.

2.7.2 Reproducibility The measured value of the independent test result obtained under the reproducibility condition is within the range of the average value given in Table 3. The difference does not exceed the reproducibility limit (R), and the reproducibility limit (R) does not exceed 5%. Reproducibility limit (R) is linear according to the data in Table 3. Obtained by interpolation or extension.

3 Method two inductively coupled plasma atomic emission spectrometry

3.1 Principle Dissolve the sample with hydrochloric acid and hydrofluoric acid. Add nitric acid dropwise for oxidation. Use inductively coupled plasma atomic emission spectrometry to determine Calculate the mass concentration of chromium by curve method, and express the measurement result by mass fraction.

3.2 Reagents Unless otherwise specified, only reagents and laboratory secondary water