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YS

**NONFERROUS METALS INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

YS/T 574.5-2009

Replacing YS/T 574.5-2006

**Methods for chemical analysis of zirconium powder for electro-vacuum
uses. Determination of calcium and magnesium content by inductively
coupled plasma atomic emission spectrometry**

电真空用锆粉化学分析方法电感耦合等离子体发射光谱法测定钙、镁量

(English Translation)

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Foreword

YS/T 574 "Methods for chemical analysis of zirconium powder for electro - vacuum" is divided into eight sections.

--- Determination of the total amount of zirconium and zirconium active YS/T 574.1 electric vacuum zirconium powder - Gravimetric method;

--- YS/T 574.2 electric vacuum Chemical analysis of zirconium powder sulfosalicylic acid spectrophotometric determination of iron content;

--- Determination of silicon content YS/T 574.3 electric vacuum chemical analysis of zirconium powder Molybdenum blue spectrophotometric method;

--- YS/T 574.4 electric vacuum zirconium powder chemical analysis of molybdenum blue spectrophotometric determination of phosphorus content;

Emission spectrometry measurement of calcium, magnesium content --- YS/T 574.5 electric vacuum zirconium powder chemical analysis by inductively coupled plasma;

--- YS/T 574.6 electric vacuum zirconium powder chemical analysis of Chromium Azure S spectrophotometric determination of aluminum content;

--- YS/T 574.7 electric vacuum Chemical analysis of zirconium powder with methylene blue spectrophotometric determination of sulfur content;

Determination of the amount of hydrogen heating thermal conductivity method --- YS/T 574.8 electric vacuum Chemical analysis of zirconium powder with an inert atmosphere.

This section YS/T 574 Part 5.

The partial replacement of YS/T 574.5 "power vacuum Chemical analysis of zirconium powder with atomic absorption spectrophotometry Ca, Mg" (formerly GB/T 3256.5-1982).

This portion of YS/T 574.5-2006 compared to the main changes are as follows.

--- Instead of the original by inductively coupled plasma atomic absorption spectrometry emission spectrometry;

--- Complements the precision and quality assurance and control provisions.

The non-ferrous metal part by the National Standardization Technical Committee and centralized.

This in part by Western Metal Materials Co., Ltd., China Nonferrous Metals Industry Institute of Standards Metrology and Quality is responsible for drafting.

This part by CITIC Jinzhou Ferroalloy Co., Ltd., Northwest Nonferrous Metal Research Institute participated in the drafting.

1 Scope

This section specifies the determination of electric vacuum zirconium powder, calcium and magnesium content.

This section applies to electric vacuum determination of zirconium powder, calcium and magnesium content. Measurement range (mass fraction). 0.004% to 1.00% magnesium, calcium 0.003% to 0.50%.

2 Principle of the method

Sample with hydrofluoric acid, nitric acid decomposition, in this medium, using inductively coupled plasma emission spectrometer to measure each element of a selected wavelength the amount of its concentration.

3 Reagents

3.1

nitrate ($\rho=1.42\text{g/mL}$), pure class distinctions.

3.2

hydrofluoric acid ($\rho=1.13\text{g/mL}$), pure class distinctions.

3.3

hydrochloric acid (1 + 1), pure class distinctions.

3.4

hydrochloric acid ($\rho=1.19\text{g/mL}$), pure class distinctions.

3.5

pure zirconium solution. Weigh 0.10g of high purity zirconium powder placed in a plastic beaker was added 5mL 100mL nitric acid (3.1) and hydrofluoric acid 3mL (3.2), heated to dissolve all cooled. Transferred to 250mL plastic flask, constant volume of water and shake.

3.6

Calcium standard stock solution. Weigh 2.4971g advance at 105 °C ~ 110 °C drying to constant calcium carbonate (reference reagent) to 300mL beaker, 20mL water was added, then a solution of hydrochloric acid (1 + 1) until completely dissolved, then was added 10mL of hydrochloric acid (3.4), boiled to remove Carbon dioxide, remove the cooled and transferred to 1000mL volumetric flask, dilute with water to volume, and mix. 1mL solution containing 1mg calcium.

3.7

Magnesium standard stock solution. Weigh 1.0000g of pure magnesium ($\geq 99.99\%$) in a group of 200mL beaker were added 15mL Nitric acid (1 + 1), heated to dissolve, the removal of nitrogen oxides was boiled and cooled. They were transferred to a 1000mL volumetric flask, diluted with water to the mark, and mix. This solution 1mL 1mg containing magnesium.

3.8

calcium, magnesium standard solution. Pipette 10.00mL were calcium, magnesium standard stock solution to a set 100mL flask, each added 10mL Nitric acid (1 + 1), diluted with water to volume, and mix. The solution 1mL each containing 100 μ g calcium, magnesium.

4 Instrument

Inductively coupled plasma emission spectrometer.

Measurement conditions.

Power (W). 1150 atomization pressure (psi). 27 auxiliary gas (lpm). 0.5 Pump speed (rpm). 100 longwave integration time (s). 20 shortwave integration time (s). 5 Observing way. Axialview (horizontal observation)

5.1 Sample

Table 1 sample was weighed, accurate to 0.0001g.