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CCS H 71

**YS**

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

**YS/T 574.3-2009**

Replacing YS/T 574.3-2006

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**Methods for chemical analysis of zirconium powder for  
electro-vacuum uses. The molybdenum blue  
spectrophotometric method for determination of silicon**

电真空用锆粉化学分析方法钼蓝分光光度法测定硅量

**(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 3.

The partial replacement of YS/T 574.3 "power vacuum spectrophotometric determination of zirconium powder with a chemical analysis of silicon molybdenum blue" (original GB/T 3256.3-

1982).

This portion of YS/T 574.3-2006 compared to the main changes are as follows.

--- 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 the amount of silicon electric vacuum zirconium powder.

This section applies to electric vacuum measuring silicon content of zirconium powder. Measurement range (mass fraction). 0.01% to 0.15%.

## 2 Principle of the method

The sample is dissolved with hydrofluoric acid, boric acid masking fluoride ion. Silicic acid ammonium molybdate was added thereto to generate ions Si - Mo heteropoly acid was added grass Acid - sulfuric acid mixture to eliminate phosphorus, arsenic interference. Ferrous ammonium sulfate to restore the silicon molybdenum yellow silicon molybdenum blue, measured in a spectrophotometer at 700nm Absorbance.

## 3 Reagents

Unless otherwise indicated in the analysis using only recognized as analytical reagents and secondary deionized water.

### 3.1

boric acid, pure class distinctions.

### 3.2

pure zirconium, silicon content less than 0.002%.

### **3.3 Hydrofluoric acid (commercially available 40%). pure class distinctions**

3.4 sulfate ( $\rho$ 1.84g/mL).

3.5 sulfate (1 + 4).

3.6 oxalic acid - sulfuric acid mixture. Weigh 25g of oxalic acid, placed in 500mL beaker, add water 200mL, 100mL sulfuric acid (3.4) to make it Dissolution, cooled to room temperature, diluted with water to 1000mL, and mix.

3.7 potassium permanganate solution (1g/L).

3.8 ammonium molybdate solution (50g/L).

3.9 ferrous ammonium sulfate solution (60g/L). Weigh 6g ferrous ammonium sulfate, placed in 200mL beaker, add 2mL of sulfuric acid (3.5), with Diluted with water to 100mL, stirred to dissolve, use (valid for 3 days) and filtered.

3.10 Silicon Storage standard solution. Weigh 0.2139g pre-burning 1h silica at 1000 °C, placed in a platinum crucible, added 2g ~ 3g of anhydrous sodium carbonate, and mix. 900 °C high temperature furnace melting at 5min ~ 7min, then cool to room temperature. Placed in 400mL burn Cup, 200mL water was added and heated to dissolve the melt. Remove the crucible was washed with water, cooled to room temperature, transferred to 1000mL volumetric flask Diluted with water to volume, and mix. Immediately transferred to a polyethylene bottle save. This solution 1mL containing 100 $\mu$ g silicon.

3.11 Silicon standard solution. Pipette 100.00mL silicon standard stock solution (3.10) into 500mL volumetric flask, dilute to the mark, Mix well. Immediately transferred to a polyethylene bottle save. 1mL solution containing 20 $\mu$ g silicon.

## **4 Instrument**

Spectrophotometer.

### **5.1 Sample**

Weigh 0.10g sample, accurate to 0.0001g.

### **5.2 Blank test**

Along with sample blank test.