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YS

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

YS/T 574.7-2009

Replacing YS/T 574.7-2006

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

电真空用锆粉化学分析方法次甲基蓝分光光度法测定硫量

(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 7.

The partial replacement of YS/T 574.7-2006 "power vacuum spectrophotometric determination of zirconium powder Chemical analysis of methylene blue sulfur" (formerly GB/T 3256.7-1982).

This portion of YS/T 574.7-2006 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 provides electrical measuring method vacuum zirconium powder sulfur content.

This standard applies to the determination of sulfur content of zirconium powder with a power vacuum. Measurement range (mass fraction). 0.010% to 0.100%.

2 Principle of the method

Flux calcined sample mixed with the flooding, with hydroiodic - hypophosphite as a reducing agent, is heated in a nitrogen stream of hydrogen sulfide was distilled off with Zinc acetate solution was absorbed, was added N, N- dimethyl-p-phenylenediamine hydrochloride solution, ferric chloride solution so that generation of methylene blue, in the spectrophotometer Meter measuring the absorbance at 680nm.

3 Reagents

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

3.1

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

3.2

HCl (7 + 3).

3.3

nitrogen (99.5%). oxygen scavenger such as oxygen-required oxygen.

3.4

mixed flux. Weigh 1g of anhydrous sodium carbonate, 2g of magnesium oxide, 0.1g ammonium nitrate, finely ground in a mortar and mix.

3.5

Potassium hydroxide solution (200g/L).

3.6

reducer. Take hydroiodic 500mL, 125mL of glacial acetic acid and 75g of sodium hypophosphite, and mix. Moved purification distiller, a nitrogen-plus Heat to boiling and maintain micro-boiling state under reflux for more than 4h.

3.7

absorption solution. Weigh 50g zinc acetate $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ and 12.5g of sodium acetate $[\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}]$ was dissolved Diluted with water, washed with water to 1000mL, and mix.

3.8

N, N- dimethyl-p-phenylenediamine hydrochloride solution (3g/L). Weigh 1.5gN, N- dimethyl-p-phenylenediamine hydrochloride, was dissolved in 500mL Hydrochloric acid (3.2) in.

3.9

ferric chloride solution (50g/L). Weigh 5g of ferric chloride $[\text{FeCl}_3 \cdot 6\text{H}_2\text{O}]$, 1mL was added hydrochloric acid (3.2), diluted with water to 100mL, and mix.

3.10

potassium permanganate - mercuric chloride solution. Weigh 2g and 5g of potassium permanganate mercuric chloride were dissolved in water and diluted to 50mL. The two A mixed solution well before use. In use, such as discoloration or precipitation discovery should be replaced.

3.11

sulfur standard stock solution. Weigh 0.5435g previously dried potassium 105 °C dissolved in water and transferred to 1000mL volumetric flask Diluted with water to volume, and mix. This solution containing 100μg per milliliter of sulfur.

3.12

sulfur standard solution. Pipette 50.00mL sulfur standard solution (3.11), placed in 500mL volumetric flask, dilute with water to volume, and mix.

This solution containing 10μg per ml of sulfur.

4 Instrument

4.1

purified by distillation (see Figure 1).