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YS

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

YS/T 574.4-2009

Replacing YS/T 574.4-2006

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

电真空用锆粉化学分析方法钼蓝分光光度法测定磷量

(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;

Determination of calcium and magnesium content --- YS/T 574.5 electric vacuum zirconium powder chemical analysis by inductively coupled plasma atomic emission spectrometry;

--- 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 Part 4 574.

The partial replacement of YS/T 574.4 "power vacuum spectrophotometric determination of

zirconium powder with a chemical analysis of molybdenum blue phosphor" (formerly GB/T 3256.4-

1982).

This portion of YS/T 574.4-2006 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 the determination of phosphorus content in zirconium powder.

This section applies to electric vacuum measuring phosphorus content of zirconium powder. Measurement range (mass fraction). 0.001% to 0.010%.

2 Principle of the method

Sample with nitric acid, hydrofluoric acid decomposition, masking boric acid fluoride, phosphoric acid with potassium permanganate oxidation of the sub-phosphate. In nitric acid medium, phosphorus Acid ions with ammonium molybdate forming yellow phosphomolybdic heteropoly acid, n-butanol - a mixed solution of chloroform extraction, stannous chloride reduction of the phosphorus molybdenum yellow phosphorus Molybdenum blue and back-extracted into the aqueous phase. Spectrophotometer measuring the absorbance at 700nm.

Zirconium matrix without interference. Test solution containing arsenic, titanium, niobium each 1000µg not interfere with the determination. Copper, nickel do not interfere with each of the following test 800µg set.

3 Reagents

3.1

hydrofluoric acid (rho1.13g/mL), pure class distinctions.

3.2

hydrochloride (rho1.19g/mL).

3.3

saturated solution of boric acid.

3.4

nitric acid (1 + 1). nitrate (ρ 1.42g/mL) boiling 3min ~ 5min, formulated after the flooding exhausted nitrogen oxides.

3.5

potassium permanganate solution (40g/L).

3.6

sodium nitrite solution (10g/L).

3.7

ammonium molybdate solution (10g/L).

3.8

stannous chloride solution (10g/L). Weigh 1.0g of stannous chloride in a dry beaker, added 8mL hydrochloric acid (3.2) dissolved, Diluted with water to 100mL, and mix. The time is now equipped with.

3.9

n-butanol - chloroform mixed solution (1 + 3).

3.10

P standard stock solution. Weigh 0.4394g advance at 105 °C ~ 110 °C drying 2h potassium dihydrogen phosphate (excellent pure) placed 250mL beaker, dissolved in water and transferred to 1000mL volumetric flask, dilute with water to volume, and mix. This solution contains per milliliter 100 μ g phosphorus.

3.11

Phosphorus standard solution. Pipette 10.00mL phosphorus standard stock solution (3.10), placed in 100mL volumetric flask, dilute to the mark, Mix well. This solution containing 10 μ g per ml of phosphorus.

4 Instrument

Spectrophotometer.

5.1 Sample

Weigh 0.20g sample, accurate to 0.0001g.

5.2 Blank test

Along with sample blank test.