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

YS/T 896-2013

**Methods for chemical analysis of high purity niobium.
Determination of trace impurity element content. Inductively
coupled plasma mass spectrometry**

高纯铌化学分析方法痕量杂质元素的测定电感耦合等离子体质谱法

(English Translation)

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1 Scope

This Standard stipulates the method for the determination of lithium, beryllium, boron, magnesium, titanium, vanadium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, zirconium, tantalum, molybdenum, cadmium, tin, antimony, hafnium, tungsten, lead and bismuth in high purity niobium.

This Standard is applicable to the determination of trace impurity element content in high purity niobium. The determination range is 0.0001% ~ 0.010%.

2 Method Summary

Use nitric acid and hydrofluoric acid to dissolve the test portion, use rhodium internal standard for calibration, and conduct direct determination through an inductively coupled plasma mass spectrometer. In accordance with the working curve method, calculate the mass concentration of each element, and express the determination result as mass fractions.

3 Reagents

Unless otherwise stated, the reagents used in the analysis are guaranteed reagents; the water used is Grade-1 water.

3.1

Hydrofluoric acid ($\rho = 1.16 \text{ g/mL}$).

3.2

Nitric acid ($\rho = 1.42 \text{ g/mL}$).

3.3

Nitric acid (1 + 1).

3.4

Lithium, beryllium, boron, magnesium, titanium, vanadium, manganese, iron, cobalt, nickel, copper, zinc, gallium, arsenic, strontium, zirconium, tantalum, molybdenum, cadmium, tin, antimony, hafnium, tungsten, lead, bismuth and rhodium single-element standard storage solution (national standard sample / national reference substance), the mass concentration is

1,000 $\mu\text{g/mL}$.

3.5

Mixed standard solution A. respectively transfer-take 10.00 mL of each element of standard storage solution (except rhodium) (3.4) in 1,000 mL plastic volumetric flasks, add 2 mL of hydrofluoric acid (3.1) and 20 mL of nitric acid (3.3); use water to dilute to the scale and mix

5 Specimens

The specimens are processed into chips with a side length not exceeding 2 mm.

6.1 Test Portion

Weigh-take 0.10 g of specimen, accurate to 0.0001 g.

6.2 Number of Determinations

Independently carry out two determinations and take the average value.

6.3 Blank Test

Carry out a blank test along with the test portion.

6.4.1 Preparation of test portion solution

Place the test portion (6.1) in a 50 mL plastic beaker, add a small amount of water, 2 mL of nitric acid (3.2) and 2 mL of hydrofluoric acid (3.1), heat to completely dissolve the test portion, and cool it. Transfer to a 100 mL plastic volumetric flask, add 1.00 mL of rhodium internal standard solution (3.7), use water to dilute to the scale, and mix it well.

6.4.2 Preparation of standard series solutions

Transfer-take 0 mL, 0.20 mL, 0.50 mL, 1.00 mL, 2.00 mL, 5.00 mL and 10.00 mL of the mixed standard solution B (3.6) in a series of 100 mL plastic volumetric flasks. Respectively add 1.00 mL of the rhodium internal standard solution (3.7), use water to dilute to the scale and mix it well.

6.5 Measurement

6.5.1

On the inductively coupled plasma mass spectrometer, after the instrument stably operates, under the selected instrument operating conditions, in accordance with the recommended isotope mass numbers shown in Table 1, use the prepared standard series solutions (6.4.2) to standardize or calibrate the working curve. The correlation coefficient of the working curve of each element shall be above 0.999, otherwise, it is necessary to re-standardize or re-prepare standard series solutions for standardization.

6.5.2 Determine the analytical test solution (6.4.1) and blank test solution. The instrument

automatically processes data in accordance with the working curve, calculates and outputs the mass concentration of each element.

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