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

XB/T 616.5-2012

**Chemical analysis methods of gadolinium ferroalloy-Part 5:
Determination of silicon content-Molybdenum blue
spectrophotometry
(English Translation)**

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Foreword

XB/T 616-2012 "Gadolinium Ferroalloy Chemical Analysis Method" is divided into 5 parts.

---Part 1: Determination of the total amount of rare earths by gravimetric method;

---Part 2: Determination of rare earth impurities by inductively coupled plasma atomic emission spectrometry;

---Part 3: Determination of calcium, magnesium, aluminum and manganese content inductively coupled plasma atomic emission spectrometry;

---Part 4: Determination of Iron Content Potassium Dichromate Titration Method;

---Part 5: Determination of silicon content Silicon molybdenum blue spectrophotometric method.

This part is part 5 of XB/T 616.

This section was drafted in accordance with the rules given in GB/T 1.1-2009.

This part is under the jurisdiction of the National Rare Earth Standardization Technical Committee (SAC/TC229).

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1 Scope

This part of XB/T 616 specifies the method for determining the amount of silicon in gadolinium-iron alloys.

This section applies to the determination of silicon content in gadolinium-iron alloys.
Measuring range. 0.010%~0.20%.

2 Method principle

The sample is dissolved in dilute acid. In the dilute acid medium, silicon and ammonium molybdate form silicomolybdenum heteropoly acid, which decomposes phosphorus in sulfuric acid and oxalic acid medium, and arsenic is heteropoly Acid, use ascorbic acid to reduce the silicomolybdenum heteropoly acid into a blue low-valent complex, and measure the absorbance at a wavelength of 800 nm with a spectrophotometer.

The curve method finds the corresponding amount of silicon.

3 Reagents and materials

3.1

Mixed acid. Dilute 180mL of hydrochloric acid (excellent grade) and 60mL of nitric acid (excellent grade) with water to 1000mL.

3.2

Ammonia (1 1), excellent grade pure.

3.3

Sulfuric acid (1 5), pure superior grade.

3.4

Oxalic acid-sulfuric acid mixed acid. 1g oxalic acid is dissolved in 100mL sulfuric acid (3.3).

3.5

Ammonium molybdate solution (50g/L).

3.6

Ascorbic acid (10g/L).

3.7

Silicon standard storage solution. weigh 0.1070g of silicon dioxide ($w_{\text{SiO}_2} > 99.9\%$, bake at 120°C for 2h and cool to room temperature), and place it in a platinum crucible Add 5g of anhydrous sodium carbonate (excellent grade pure) and melt at $950^\circ\text{C}\sim 1000^\circ\text{C}$ to red and transparent. After it is slightly cold, it is leached out with hot water and cooled. will the

solution was transferred to a 500mL volumetric flask and diluted with water to the mark. Transfer to a plastic bottle immediately. This solution 1mL contains 100 μ g silicon.

3.8

Silicon standard solution. Pipette 10mL of silicon standard stock solution (3.7) into a 100mL plastic volumetric flask, dilute to the mark with water, and mix.

1mL of this solution contains 10 μ g of silicon.

3.9

p-nitrophenol solution (1g/L).

4 Instruments

Spectrophotometer.

5 Sample

The surface oxide layer should be removed from the metal sample and weighed immediately after sampling.