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

XB/T 616.4-2012

**Chemical analysis methods of gadolinium ferroalloy-Part 4:
Determination of iron content-Potassium dichromate titrimetry
(English Translation)**

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3.13 Standard solution of potassium dichromate [$c(K_2Cr_2O_7) \sim 0.006 \text{ mol/L}$]

Foreword

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

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 4 of XB/T 616-2012.

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

The unit responsible for drafting this section. Ganzhou Institute of Nonferrous Metallurgy.

This section was drafted. Ganzhou Nonferrous Metallurgy Research Institute.

Participated in the drafting of this section. Hunan Rare Earth Metal Materials Research Institute, Ganzhou Qiangdong Industrial Group Co., Ltd., Beijing Nonferrous Metals General Research Institute, Ganzhou Chenguang Rare Earth New Materials Co., Ltd., Jiangmen Keheng Industrial Co., Ltd.

1 Scope

This part of XB/T 616 specifies the determination method of iron content in gadolinium-iron alloy.

This section applies to the determination of iron content in gadolinium-iron alloys.
Measuring range. 10.00%~40.00%.

2 Method principle

After the sample is dissolved in hydrochloric acid, sodium tungstate is used as an indicator, and the trivalent iron is reduced to divalent iron with titanium trichloride, and potassium dichromate solution is added dropwise to oxygen to remove excess trivalent titanium, add sulfur-phosphorus mixed acid, use sodium diphenylamine sulfonate as indicator, and titrate with potassium dichromate standard solution to purple as the end point.

3 Reagents and materials

3.1

Sulfuric acid ($\rho=1.84\text{g/mL}$).

3.2

Phosphoric acid ($\rho=1.70\text{g/mL}$).

3.3

Sulfur-phosphorus mixed acid. slowly add 100mL sulfuric acid (3.1) to 100mL phosphoric acid (3.2).

3.4

Hydrochloric acid (1 2).

3.5

Hydrochloric acid (1 3).

3.6

Hydrochloric acid (1 9).

3.7

Sodium tungstate solution (250g/L). Weigh 25g sodium tungstate in 95mL water, add 5mL phosphoric acid (3.2), and mix well.

3.8

Commercially available titanium trichloride solution (150g/L~200g/L).

3.9

Titanium trichloride solution (19). Dilute the commercially available titanium trichloride solution (3.8) with hydrochloric acid (3.6) by 10 times, and prepare it when used.

3.10

Potassium dichromate (0.1g/L).

3.11

Iron standard solution. Weigh 2.0000g pure metal iron wire (purity greater than 99.95%, wipe off the surface oxide layer before use), and place in a 250mL beaker, add 120mL of hydrochloric acid (3.4), put it on the boiling water bath until it is completely dissolved, cool it, and transfer it to a 1000mL volumetric flask.

Dilute to the mark with water and mix well. This standard solution 1mL contains 2.0mg iron.

3.12

Sodium diphenylamine sulfonate (10g/L).

3.13 Standard solution of potassium dichromate [$c(K_2Cr_2O_7) \sim 0.006mol/L$]

3.13.1

Preparation. Weigh 1.8g of potassium dichromate (3.10) into a 500mL beaker, add 200mL of water, until completely dissolved, transfer Dilute to the mark with water in a 1000mL volumetric flask and mix well.

3.13.2

Calibration. Pipette 20.00mL iron standard solution (3.11), place it in a 300mL conical flask, add 40mL hydrochloric acid (3.5), add Heat to near boiling, cool to room temperature, add water to a volume of about 150mL, add 1mL sodium tungstate solution (3.7), use titanium trichloride solution (3.9) Drop to blue, add potassium dichromate solution (3.10) to colorless, add 15mL sulfur-phosphorus mixed acid (3.3), 2 drops of sodium diphenylamine sulfonate the solution (3.12) is titrated with potassium dichromate standard solution (3.13.1) until it becomes purple and does not disappear for 30 seconds.

Calculate potassium dichromate standard solution according to formula (1).

$$c = \frac{m \times V_1}{V_2 - V_3} \times 6 \times 55.85$$