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

XB/T 614.4-2011

**Chemical analysis method of gadolinium-magnesium alloy-Part
4: Determination of fluorine content-Water vapor distillation
spectrophotometry
(English Translation)**

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Foreword

XB/T 614 "Methods for Chemical Analysis of Gadolinium Magnesium Alloys" is divided into 6 parts.

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

---Part 2: Determination of magnesium content EDTA titration;

---Part 3: Determination of carbon content high frequency-infrared absorption method;

---Part 4: Determination of fluorine content by steam distillation spectrophotometry;

---Part 5: Determination of rare earth impurities;

--- Part 6: Determination of the amount of aluminum, calcium, copper, iron, nickel, and silicon inductively coupled plasma atomic emission spectrometry.

This part is part 4.

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 614 specifies the method for determining the amount of fluorine in gadolinium-magnesium alloys.

This section applies to the determination of fluorine content in gadolinium-magnesium alloys. The measurement range is 0.010%~0.50%.

2 Method principle

The fluorine in the sample is volatilized with perchloric acid fumes by water vapor distillation, separated from other elements, and condensed to be recovered and enriched. Using phenolphthalein as an indicator, use Sodium hydroxide and hydrochloric acid adjust the distillate to be colorless, use acetone as a stabilizer, alizarin ammonia carboxyl complex hydrazone (C₁₉H₁₅NO₈) as a color developer, and keep it in a water bath.

Temperature, measure its absorbance at a wavelength of 630nm.

3 Reagents and materials

3.1

Perchloric acid (excellent grade pure, $\rho=1.76\text{g/mL}$).

3.2

Acetone.

3.3

Saturated sodium hydroxide solution (excellent grade pure).

3.4

Sodium hydroxide solution (250g/L) (excellent grade pure).

3.5

Hydrochloric acid (1 1), (excellent grade pure).

3.6

Hydrochloric acid (5 95), (excellent grade pure).

3.7

Ammonia (1 1), (excellent grade pure).

3.8

Lanthanum oxide solution. Weigh 3.2589g lanthanum oxide (REO >99.5%, La₂O₃/REO >99.9%) into a 250mL beaker, add A small amount of water, add hydrochloric acid (3.5) until the lanthanum oxide just dissolves. Transfer the solution to a 500mL volumetric flask, dilute to the mark with water, and mix. Melt the liquid concentration is 0.04mol/L.

3.9

Acetic acid-sodium acetate solution. Weigh 120g of sodium acetate in a 250mL beaker, dissolve it with an appropriate amount of water, and transfer the solution to a 2500mL volume.

Add 30mL of acetic acid to the measuring flask, dilute to the mark with water, and mix. Solution pH~5.3.

3.10

Alizarin hydrazone color reagent. Weigh 0.0482g alizarin hydrazone into a 250mL beaker, add 1mL ammonia (3.7) Dissolve it. Add 125mL acetone (3.2), 6.25mL lanthanum oxide solution (3.8) and 50mL acetic acid-sodium acetate buffer sequentially Solution (3.9), transferred to a 250mL volumetric flask, dilute to the mark with water, and mix.

3.11

Fluorine standard storage solution. accurately weigh 0.5659g sodium fluoride (>99.95%, drying at 105°C for 1h) in a 250mL beaker, and use Dissolve an appropriate amount of water, transfer to a 250mL volumetric flask, dilute to the mark with water, and mix well. The concentration of this fluorine standard stock solution is 1mg/mL, guarantee Store in a plastic bottle.

3.12

Fluorine standard solution. accurately divide 10.00mL fluorine standard stock solution (3.11) into a 100mL volumetric flask and dilute to the mark with water.

Mix well. This fluorine standard solution has a concentration of 100µg/mL and is stored in a plastic bottle.

3.13

Phenolphthalein as indicator (10g/L). 1g phenolphthalein is dissolved in 100mL ethanol (32).