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

GB/T 12690.18-2017

**Chemical analysis methods for non-rare earth impurity of rare
earth metals and their oxides - Part 18: Determination of
zirconium content**

稀土金属及其氧化物中非稀土杂质化学分析方法 第18部分：锆量的测定

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Foreword

GB/T 12690 "rare earth metals and oxides non-rare earth impurities chemical analysis method" is divided into 18 parts.

- Part 1. Determination of carbon, sulfur content High-frequency - infrared absorption method;
- Part 2. Determination of ignition loss in rare earth oxides gravimetric method;
- Part 3. Determination of water content in rare earth oxides gravimetric method;
- Part 4. Determination of oxygen and nitrogen content Pulsed - infrared absorption and pulse - thermal conductivity method;
- Part 5. Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium and iron content;
- Part 6. Determination of iron content potassium thiocyanate, 1,10-phenanthroline spectrophotometry;
- Part 7. Determination of silicon content molybdenum blue spectrophotometry;
- Part 8. Determination of sodium content; Flame atomic absorption spectrometry;
- Part 9. Determination of chlorine content silver nitrate turbidimetric method;
- Part 10. Determination of phosphorus content molybdenum blue spectrophotometry;
- Part 11. Determination of magnesium content by flame atomic absorption spectrometry;
- Part 12. Determination of thorium content;
- Part 13. Determination of molybdenum and tungsten content;

1 Scope

GB/T 12690.18-2017 is the Chinese national standard on chemical analysis methods for non-rare earth impurity of rare earth metals and their oxides - part 18: determination of

zirconium content, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 14 October 2017 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 May 2018. Classification: ICS 77.120.99, CCS H14. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This part of GB/T 12690 specifies the determination of zirconium content of rare earth metals and their oxides. This section applies to the determination of zirconium in rare earth metals and oxides, including two methods, method 1 inductively coupled plasma Emission spectrometry, Method

2 Inductively coupled plasma mass spectrometry. Measurement range for Method 1 > 0.010% to 0.20%; Method

2 Determination Range .0.0005% ~ 0.010%.

2.2 Reagents and materials

2.2.1 Hydrogen peroxide (30%).

2.2.2 Nitric acid (1 1).

2.2.3 Sulfuric acid (1 1).

2.2.4 zirconium standard stock solution Accurately weigh 1.0000g metal zirconium [w (Zr) $\geq$ 99.9%] in a Teflon beaker, add 20mL Water, add 3mL hydrofluoric acid ($\rho = 1.14\text{g/mL}$), heat, and add a small amount of nitric acid (2.2.2) dropwise until completely dissolved. After boiling the solution, take Beaker, cooled to room temperature, transferred to 1000mL volumetric flask, diluted with nitric acid (1 19) to the mark, and mix. This solution contains 1mL 1 mg zirconium.

2.2.5 zirconium standard solution Pipette 10.00mL zirconium standard stock solution (2.2.4) in 200mL volumetric flask, add 10mL nitric acid (2.2.2), diluted with water to the mark, mix well. This solution 1 mL contains 50 μg zirconium.

2.2.6 Rare Earth Base Solution Weigh 1.0000g of single rare earth oxide [w (REO) $\geq$ 99.5% after burning at 950 °C for 1h, rare earth phase For purity $\geq$ 99.9%, w (Zr) < 0.001%], placed in 100mL beaker, add 20mL of nitric acid (2.2.2), low temperature heating until complete dissolution, Remove the cooling. Into a 100mL volumetric flask, diluted with water to the mark, mix well. 1 mL of this solution contains 10 mg of a single rare earth oxide.

2.2.7 Argon [ϕ (Ar) $\geq 99.99\%$].

2.3 equipment Inductively Coupled Plasma Spectrometer with resolution $<$

0.006 nm (at 200 nm).

2.4.1 Preparation of rare earth oxide sample at 105 °C drying 1h, placed in a desiccator, cooled to room temperature.

2.4.2 Preparation of rare earth metal sample. Remove the surface oxide layer, immediately after sampling, weighing.

2.5 Analysis steps

2.5.1 Sample Weigh 0.10g sample (2.4), accurate to 0.0001g.