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

GB/T 12690.16-2010

**Chemical analysis methods of non-rare earth impurities in rare
earth metals and the oxides - Part 16: Determination of fluorine
content - Ion selective electrode analysis**

稀土金属及其氧化物中非稀土杂质化学分析方法 第16部分：氟量的测定 离子选
择性电极法

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Foreword

GB/T 12690 "non - rare earth impurities in rare earth metals and their oxides chemical analysis method" is divided into 17 parts.

- Part 1. Carbon and sulfur content was measured high-frequency infrared method;
- Part 2. Determination of the weight loss on ignition;
- Part 3. rare earth oxide in water content - Gravimetric method;
- Part 4. oxygen, nitrogen pulse measurement - infrared absorption method and pulse - heating thermal conductivity;
- Part 5. aluminum, chromium, manganese, iron, cobalt, nickel, copper, zinc, lead content was measured;
- Part 6. Determination of iron content of potassium thiocyanate, 1,10-phenanthroline spectrophotometry;
- Part 7. Determination of silicon content - Molybdenum blue spectrophotometric method;
- Part 8. Determination of sodium content by flame atomic absorption spectrometry;
- Part 9. Determination of chlorine content of silver nitrate turbidimetry;
- Part 10. Determination of phosphorus content - Molybdenum blue spectrophotometric method;
- Part 11. Determination of magnesium content - Flame atomic absorption spectrometry;
- Part 12. Determination of the amount of thorium;
- Part 13. Determination of tungsten, molybdenum content;

1 Scope

GB/T 12690.16-2010 is the Chinese national standard on chemical analysis methods of non-rare earth impurities in rare earth metals and the oxides - part 16: determination of

fluorine content - ion selective electrode analysis, 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 January 2011 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 November 2011. 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.

GB/T 12690 provisions of this part of the determination of rare earth metals and oxides of fluorine content. This section applies to the determination of fluorine content of rare earth metals and oxides. Measurement range. 0.010% to 0.50%.

2 Principle of the method

After the sample is dissolved in dilute nitric acid by adding an excess of sodium hydroxide, the separation of fluorine and iron, rare earth and so on. In the solution of pH6.5 ~ 7.0, fluorine ion selective electrode as indicator electrode, a saturated calomel electrode as the reference electrode, the electrode potential value measured balance between the two poles, with Grignard mapping Obtained fluorine content.

3 Reagents

3.1 Hydrogen peroxide (30%).

3.2 nitric acid (13).

3.3 Sodium hydroxide solution (300g/L).

3.4 Sodium hydroxide solution (50g/L).

3.5 sodium citrate buffer (300g/L).

3.6 fluoro standard stock solution. Weigh 2.2101g superior grade pure sodium fluoride (120 °C drying to constant weight) in 250mL plastic beaker, add water After dissolution transferred to 1000mL volumetric flask, dilute with water to volume, and mix immediately stored in a plastic bottle containing the solution 1mL 1mg fluorine.

3.7 fluoro standard solution. Pipette 10.00mL fluoro standard stock solution (3.6) in 100mL volumetric flask, dilute to the mark with water, mix, Now stored in plastic bottles. This solution 1mL containing 100µg fluorine.

3.8 fluoro standard solution. Pipette 20.00mL fluoro standard solution (3.7) in 100mL

volumetric flask, dilute with water to volume, and mix immediately Stored in plastic bottles.
This solution 1mL containing 20 μ g fluorine.

3.9 bromocresol green indicator (1g/L). Weigh 0.1g bromocresol green dissolved in 20mL of ethanol, diluted with water to 100mL, and mix.

4 Equipment

4.1 potential measuring instrument, precision 0.1mV.

4.2 fluoride ion selective electrode. fluoride ion detection limit should be less than 2.5×10^{-4} mg/mL, the electrodes before use in 1×10^{-3} mol/L NaF solution soak for more than 1h, so that activation, and then washed with water until the determination can be carried out after the electrode potential of not greater than -370mV.

4.3 pairs of junction saturated calomel electrode.