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

GB/T 12690.6-2017

Replacing GB/T 12690.6-2003

Chemical analysis methods for non-rare earth impurity of rare earth metals and their oxides - Part 6: Determination of iron content - Kslium thiocyanate, 1,10-Phenanthroline spectrophotometric method

稀土金属及其氧化物中非稀土杂质化学分析方法 第6部分：铁量的测定 硫氰酸钾、1,10-二氮杂菲分光光度法

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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 12690.6-2017 is the Chinese national standard on chemical analysis methods for

non-rare earth impurity of rare earth metals and their oxides - part 6: determination of iron content - potassium thiocyanate, 1,10-phenanthroline spectrophotometric method, 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 iron content in rare earth metals and their oxides. This section applies to the determination of iron in rare earth metals and their oxides. Measuring range: 0.0001% to 0.0050%.

2 Method summary

The sample is dissolved in hydrochloric acid or nitric acid. In a slightly acidic medium, iron forms a purple-red ternary complex with potassium thiocyanate and 1,10-phenanthroline. The complex was extracted with methyl isobutyl ketone and its absorbance was measured at a spectrophotometer wavelength of 520 nm.

3 Reagent

3.1 Hydrogen peroxide (30%).

3.2 methyl isobutyl ketone.

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

3.4 nitric acid (1:1), excellent grade pure.

3.5 Potassium thiocyanate solution (500 g/L). 3.6 1,10-phenanthroline ethanol solution (2.5g/L). Weigh 0.25g 1,10-phenanthroline dissolved in 100mL of ethanol.

3.7 iron standard storage solution Accurately weigh 0.1000g iron (99.9%) in 150mL beaker, add 30mL hydrochloric acid (3.3), low temperature plus Heat until dissolved, cool to room temperature. Into a 1000mL volumetric flask, diluted with water to the mark, shake. This solution contains 1mL 100 µg iron.

3.8 iron standard solution Pipette 10.00mL iron standard stock solution (3.7) in 1000mL volumetric flask, diluted with water to the mark, shake. 1 mL of this solution contains 1 µg of iron.

4 Instruments

Spectrophotometer.

5 Sample

5.1 Sample in the oven at 105 °C bake 1h, and placed in a desiccator cooled to room temperature for use.

5.2 metal samples need to remove the surface oxide layer, immediately after the sample weighed.