ICS 77.120.99
CCS H 14

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 12690.12-2024

Replacing GB/T 12690.12-2003

Chemical analysis methods for non-rare earth impurities in rare earth metals and their oxides - Part 12: Determination of thorium and uranium - Inductively coupled plasma mass spectrometry

稀土金属及其氧化物中非稀土杂质 化学分析方法 第12部分：钍、铀量的测定 电感耦合等离子体质谱法

Issued on: April 25, 2024 **Implemented on: November 1, 2024**

Issued by: State Administration for Market Regulation;
Standardization Administration of China

Table of Contents

Foreword

This document is in accordance with the provisions of GB/T 1:1-2020 "Guidelines for standardization work Part 1: Structure and drafting rules for standardization documents" Drafting: This document is Part 12 of GB/T 12690 "Chemical analysis methods for non-rare earth impurities in rare earth metals and their oxides": The following parts have been published:

--- Part 1: Determination of carbon and sulfur content - High frequency-infrared absorption method;

--- Part 2: Determination of loss on ignition of 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 - Pulse-infrared absorption method and pulse-thermal conductivity method;

--- Part 5: Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium and iron

--- Part 6: Determination of iron content Potassium thiocyanate and 1,10-phenanthroline spectrophotometric method;

--- Part 7: Determination of silicon content;

--- Part 8: Determination of sodium content;

--- Part 9: Determination of chlorine content - Silver nitrate turbidimetric method;

--- Part 10: Determination of phosphorus content - Molybdenum blue spectrophotometric method;

--- Part 11: Determination of magnesium content - Flame atomic absorption spectrometry;

--- Part 12: Determination of thorium content by arsenazo III spectrophotometry and inductively coupled plasma mass spectrometry;

--- Part 13: Determination of molybdenum and tungsten content - Inductively coupled plasma optical emission spectrometry and inductively coupled plasma mass spectrometry;

1 Scope

Part 12 of China's series on the chemical analysis of non-rare-earth impurities in rare earth metals and oxides, covering the determination of thorium and uranium by inductively coupled plasma mass spectrometry. Of all the determinations in the series this is the one with consequences beyond the specification sheet, because thorium and uranium are radioactive. Rare earth ores carry them: monazite in particular is a phosphate of the light rare earths that contains several per cent thorium, and the thorium follows the rare earths through the early stages of processing. What is left in the product determines whether the material can be shipped as an ordinary commodity or falls under the rules for radioactive substances, whether the residues of the process are ordinary tailings or radioactive waste, and whether the people handling it need monitoring. This document describes a method for the determination of thorium and uranium in rare earth metals and their oxides by inductively coupled plasma mass spectrometry, over a range of mass fractions beginning at 0.00005 % for thorium. The method is mass spectrometric because the levels are very low and because the two elements have to be distinguished from each other and from a matrix that is almost entirely rare earth - and a rare earth matrix is a difficult one for ICP-MS, since the elements are numerous, adjacent in mass and form oxide ions that interfere. Most of the procedure is therefore about the matrix: the dissolution, the separation or dilution used to control it, the internal standard, the correction for the oxide and doubly charged interferences, and the calibration against solutions prepared in a matching matrix. Issued on 25 April 2024 and in force since 1 November 2024, it replaces GB/T 12690.12-2003.

This document describes a method for the determination of thorium and uranium in rare earth metals and their oxides: This document is applicable to the determination of thorium and uranium in rare earth metals and their oxides: Determination range (mass fraction): thorium content 0:00005%~ 0:020%; uranium content 0:00005%~0:020%:

2 Normative references

The contents of the following documents constitute the essential clauses of this document through normative references in this document: For referenced documents without a date, only the version corresponding to that date applies to this document; for referenced documents without a date, the latest version (including all amendments) applies to This document:

GB/T 6682 Specifications and test methods for water used in analytical laboratories

GB/T 8170 Rules for rounding off values and expression and determination of limit values

JJF1159 Calibration Specification for Quadrupole Inductively Coupled Plasma Mass

Spectrometer

3 Terms and definitions

There are no terms or definitions that require definition in this document:

4 Method Summary

The sample was decomposed by nitric acid and directly measured by mass spectrometry in a dilute nitric acid medium with argon plasma as the ion source: The instrument was calibrated by the internal standard method: Factors that affect the measurement, such as drift and matrix effect:

5 Reagents and Materials

Unless otherwise specified, only reagents of analytical grade or above and those in compliance with the laboratory grade specified in GB/T 6682 were used in the analysis: Water: Liquid reagents are stored in plastic bottles, and certified standard solutions are preferred: 5:

1 Hydrogen peroxide [w(H2O2)>=30%]: 5:

2 Hydrofluoric acid (19): 5:

3 Nitric acid (1 1): 5:

4 Nitric acid (2 98): 5:

5 Thorium standard solution (rho=1000μg/mL): certified standard solution: