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

GB/T 12690.4-2021

Replacing GB/T 12690.4-2003

**Chemical analysis methods for non-rare earth impurities of rare earth metals and
their oxides - Part 4: Determination of oxygen and nitrogen contents -
Impulse-infrared absorption and impulse-thermal conductivity method**

稀土金属及其氧化物中非稀土杂质化学分析方法 第4部分：氧、氮量的测定 脉冲-
红外吸收法和脉冲-热导法

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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 of Standardization Documents" Drafting. This document is the fourth part of GB/T 12690 "Chemical Analysis Methods of Non-rare Earth Impurities in Rare Earth Metals and Their Oxides". GB/T 12690 The following parts have been released.

- 1. Determination of carbon and sulfur content by high-frequency-infrared absorption method;
- 2. Determination of Ignition Loss in Rare Earth Oxides by Gravimetric Method;
- 3. Gravimetric method for the determination of water content in rare earth oxides;
- 4. Determination of oxygen and nitrogen content by pulse-infrared absorption method and pulse-thermal conductivity method;
- 5. Determination of cobalt, manganese, lead, nickel, copper, zinc, aluminum, chromium, magnesium, cadmium, vanadium and iron content;
- 6. Determination of iron content potassium thiocyanate, 1,10-phenanthroline spectrophotometry;
- 7. Determination of silicon content;
- 8. Determination of sodium content;
- 9. Determination of chlorine content, silver nitrate turbidimetric method;
- 10. Determination of Phosphorus Molybdenum Blue Spectrophotometry;
- 11. Determination of Magnesium Content Flame Atomic Absorption Spectrometry;
- 12. Determination of thorium content Arsenazo III spectrophotometry and inductively coupled plasma mass spectrometry;

1 Scope

GB/T 12690.4-2021 is the Chinese national standard on chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides - part 4: determination of oxygen and nitrogen contents - impulse-infrared absorption and impulse-thermal conductivity 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 11 October 2021 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 May 2022. 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 document specifies the determination method of oxygen and nitrogen content in rare earth metals (except metal ytterbium). This document is applicable to the determination of oxygen and nitrogen content in rare earth metals (except metal ytterbium). Measuring range (mass fraction). oxygen 0.0050%~ 0.30%; Nitrogen 0.0020%~0.20%.

2 Normative references

The contents of the following documents constitute the indispensable clauses of this document through normative references in the text. Among them, dated quotations Only the version corresponding to that date is applicable to this document; for undated reference documents, the latest version (including all amendments) is applicable to This document.

GB/T 8170 Numerical rounding rules and the expression and determination of limit values

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle

In an inert atmosphere, the sample in the graphite crucible is heated and melted, and the oxygen in the sample is precipitated as carbon monoxide or (and) carbon dioxide. After the furnace, enter the infrared detector for measurement. Nitrogen precipitates out as nitrogen and enters the thermal conductivity detector for measurement.

5 Reagents and materials

Unless otherwise specified, only reagents and distilled water or deionized water or

equivalent purity water are used in the analysis.

5.1 Nickel bag or nickel basket with cover, the blank value (mass fraction) of oxygen and nitrogen is not more than 0.00050%, and the nickel content (mass fraction) is not less than 99%, and It has been corroded by mixed acid (HNO₃ HPO₄ HAc) in advance.

5.2 Carbon tetrachloride or acetone, oxygen, nitrogen blank value (mass fraction) is not more than 0.00050%.

5.3 Graphite crucible.

5.4 Oxygen standard samples. Choose three suitable certified standard samples within the range of oxygen content (mass fraction) 0.0050%~0.30%.

5.5 Nitrogen standard samples. Choose three suitable certified standard samples within the range of nitrogen content (mass fraction) 0.0020%~0.20%.

5.6 Helium [$\phi(\text{He}) \geq 99.99\%$].