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

GB/T 12690.20-2024

Chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides - Part 20: Determination of minor amounts of fluorine and chlorine in rare earth oxides - Ion chromatography method

稀土金属及其氧化物中非稀土杂质化学分析方法 第20部分：稀土氧化物中微量氟、氯的测定 离子色谱法

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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 for standardization documents" Drafting is required. This document is part 20 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. Gravimetric method for determination of loss on ignition of rare earth oxides; Part

1 Scope

GB/T 12690.20-2024 is the Chinese national standard on chemical analysis methods for non-rare earth impurities of rare earth metals and their oxides - part 20: determination of minor amounts of fluorine and chlorine in rare earth oxides - ion chromatography 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 28 November 2024 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 June 2025. 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 describes a method for the determination of trace amounts of fluorine and chlorine in rare earth oxides. This document is applicable to the determination of fluorine and chlorine content in rare earth oxides. Determination range (mass fraction). fluorine 0.001 0% to 0.010%; Chlorine 0.001 0% to 0.010%.

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. in this document.

GB/T 6379.2 Accuracy (trueness and precision) of measurement methods and results Part

2. Determination of repeatability of standard measurement methods Basic methods of sex and reproducibility

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

JJG 823 Ion Chromatograph

3 Terms and definitions

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

4 Principle

The sample is decomposed by sulfuric acid, and the fluorine and chlorine in it escape with water vapor and are separated from the sample, absorbed by water, and determined by ion chromatography. Fluorine and chlorine were identified qualitatively by peak time and quantified by standard curve method.

5 Reagents or materials

Unless otherwise specified, only reagents of confirmed high purity or above and first-grade water that complies with GB/T 6682 were used in the analysis. Use certified standard solutions.

5.1 Sodium fluoride, reference substance.

5.2 Sodium chloride, reference substance.

5.3 Hydrogen peroxide [$w(\text{H}_2\text{O}_2) \geq 30\%$].

5.4 Sulfuric acid (2 1).

5.5 Eluent. Select a suitable eluent according to the instructions for use of the separation column.

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 - Arsenazo III spectrophotometric method 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; Part

19 Determination of arsenic and mercury content; - Part

20.Determination of trace amounts of fluorine and chlorine in rare earth oxides - Ion chromatography; Part

21.Determination of sulfate content in rare earth oxides - Barium sulfate turbidimetric method. Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents. This document was proposed and coordinated by the National Rare Earth Standardization Technical Committee (SAC/TC 229). This document was drafted by: Hunan Rare Earth Metal Materials Research Institute Co., Ltd., Qiandong Rare Earth Group Co., Ltd., Jiangxi Tungsten The company has cooperated with the Rare Earth Product Quality Supervision and Inspection Center, Baotou Rare Earth Research Institute, Guohe General Testing Evaluation and Certification Co., Ltd., Fujian Changting Jinlong Rare Earth Co., Ltd., Qingdao Shenghan Chromatography Technology Co., Ltd. The main drafters of this document are. Wang Guichao, Zhang Manning, Liu Rongli, Shi Xuefeng, Wen Bin, Xu Na, Liu Pengyu, Liu Chun, Wang Jinfeng, Chen Yanqun, Luo Zhiya, Zou Shihui, Wang Ling, Tian Haifeng, Zhao Yan, Zhang Xiuyan, Deng Yu, Zhang Jinmei.

In the field of chemical composition analysis of rare earth products, my country has established detection methods for total rare earth content, non-rare earth impurities, and rare earth impurities. GB/T 12690 Chemical analysis methods for non-rare earth impurities in rare earth metals and their oxides GB/T 12690.1226-1990 "Chemical analysis methods for rare earth metals and their oxides" is the basis, and GB/T 8762.3- 1988 "Emission spectrometry for determination of acid-soluble silicon dioxide in fluorescent grade yttrium oxide" GB/T 8762.4-1988 "Emission spectrometry for determination of acid-soluble silicon dioxide in fluorescent grade yttrium oxide" Determination of the amount of iron oxide, lead oxide, nickel oxide and copper oxide by emission spectrometry" GB/T 8762.6-1988 "Determination of the amount of iron oxide, lead oxide, nickel oxide and copper oxide by emission spectrometry" GB/T 11074.3-7-1989 Chemical Analysis Method for Samarium Oxide The integrated series of method standards It uses advanced detection methods and basically covers all rare earth metals and their rare earth oxide matrices, providing The method provides a quick and accurate method for the determination of rare earth impurity content with good operability. According to the differences in the test objects, test methods, and the differences between the rare earth metals and rare earth oxide matrices, GB/T 12690 is proposed to be 21 parts. Part

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

2.Gravimetric method for determination of loss on ignition of rare earth oxides; Part