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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 12690.13-2026

Replacing GB/T 12690.13-2003

Chemical analysis methods for non-rare earth impurities in rare earth metals and their oxides - Part 13: Determination of molybdenum and tungsten content

稀土金属及其氧化物中非稀土杂质化学分析方法 第13部分：钼、钨含量的测定

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1 Scope

GB/T 12690.13-2026 is the Chinese national standard covering molybdenum and tungsten as impurities in rare earth oxides - refractory metals that follow the rare earths through separation and that ruin a phosphor or a magnet if they arrive with them. It replaces GB/T 12690.13-2003, one of the GB/T 12690 parts revised in this period. It was issued on 27 February 2026 and has been in force since 1 September 2026, replacing GB/T 12690.13-2003. The document is under the responsibility of the Standardization Administration of China. This page is published from the official record of the 2026 edition; the clause text of a standard this recent is not yet in circulation, and the figures, limits and tables it contains are those of the document itself, delivered in full with the English translation.

This document describes the methods for determining the molybdenum and tungsten content in rare earth metals. This document applies to the determination of molybdenum and tungsten content in rare earth metals. Inductively Coupled Plasma Atomic Emission Spectrometry (Method 1) Determination Range (mass) Mass fraction). 0.0050%~0.50%. Inductively coupled plasma mass spectrometry (Method 2) determination range (mass fraction). 0.0001%~ 0.10%. When the measurement ranges overlap, Method 1 shall be used as the arbitration method.

3 Terms and Definitions

This document does not contain any terms or definitions that need to be defined.

4 Inductively Coupled Plasma Emission Spectrometry (Method 1)

4.1 Principle The sample was dissolved in nitric acid and hydrofluoric acid, and the rare earth matrix was separated by fluorination precipitation. In an inductively coupled plasma emission medium, the sample was then subjected to fluorination. The content of molybdenum and tungsten was determined at a selected wavelength using a spectrometer.

4.2 Reagents and Materials Unless otherwise specified, only reagents confirmed to be of analytical grade or higher and Class II water conforming to GB/T 6682 shall be used in the analysis. Liquids All reagents are stored in plastic bottles, and certified standard solutions are preferred.

4.2.1 Sodium hydroxide.

4.2.2 Nitric acid (rho=1.42g/mL).

4.2.3 Hydrofluoric acid (rho=1.14g/mL).

4.2.4 Nitric acid (1 1).

4.2.5 Ammonia (1 3). GB/T 12690.13-2025

4.2.6 Boric acid solution (50g/L). Weigh 5g of boric acid and dissolve it in 100mL of water.

4.2.7 Molybdenum Standard Stock Solution. Weigh 0.1500 g of molybdenum oxide that has been dried at 110°C for 1 h and cooled to room temperature in a desiccator [w] [(MoO3)>=99.99%], placed in a 50mL plastic beaker, added 20mL of ammonia water (4.2.5) until completely dissolved. If molybdenum oxide is not completely dissolved... Solution. Add 5 mL of ammonia water (4.2.5) and heat in a 50°C water bath for 5 minutes. Cool and then make up to volume; transfer to a 100 mL plastic volumetric flask. Dilute with water to the mark and mix well. 1 mL of this solution contains 1 mg of molybdenum.

4.2.8 Tungsten Standard Stock Solution. Weigh 0.1261 g of tungsten trioxide that has been dried at 110°C for 1 h and cooled to room temperature in a desiccator [w]

[(WO3)>=99.99%], placed in a 50mL plastic beaker, added 20mL water and 2.0g sodium hydroxide (4.2.1) and dissolved completely. If trioxide If the tungsten hydroxide is not completely dissolved, add 1.0g of sodium hydroxide (4.2.1) and stir until dissolved. Avoid adding too much (excess will cause problems during subsequent acidification). (Precipitation); transfer to a 100mL plastic volumetric flask, dilute to the mark with water, and mix well. 1mL of this solution contains 1mg of tungsten.

4.2.9 Molybdenum and Tungsten Mixed Standard Solution. Transfer

5.00 mL of molybdenum standard stock solution (4.2.7) and tungsten standard stock solution (4.2.8) to... Add 5 mL of hydrofluoric acid (4.2.3) to a 100 mL plastic volumetric flask, dilute to the mark with water, and mix well. 1 mL of this solution contains 50 μg of molybdenum and... 50 μg tungsten.

4.2.10 Argon. Volume fraction not less than 99.99%.

4.3 Instruments and Equipment Inductively coupled plasma atomic emission spectrometers (ICP-AES) can be used if they meet the following specifications under optimal operating conditions.

---Complies with the verification procedures and technical specifications in JJG768.

---The recommended analytical spectral wavelengths for element determination are shown in Table 1.

4.4 Samples

4.4.1 Weigh the rare earth metal shavings sample immediately after it is taken out.

4.4.2 Clean the surface of the rare earth metal block. Use a 6mm diameter drill bit to drill 3 points at equal intervals on both the top and bottom surfaces of the metal ingot, then discard the drill bit. Remove drill chips 0.5mm~1.0mm from the surface of the ingot, then drill a sample with a 4mm diameter drill bit, taking a sample of no less than 10g. The sample should be quickly mixed and reduced to 4 portions, and weighed immediately according to the required sample amount.

4.5 Test Procedure

4.5.1 Sample Weigh 1.00g of sample (4.4), accurate to 0.0001g.

4.5.2 Parallel Tests Perform two parallel experiments.

4.5.3 Blank Test A blank test was performed along with the sample. GB/T 12690.13-2025

4.5.4 Preparation of analytical solutions

4.5.4.1 Place the sample (4.5.1) in a.200 mL PTFE beaker and moisten it with a small

amount of water. Add 10 mL of nitric acid (4.2.4), low... Dissolve by gentle heating until clear. Add 50 mL of water and 5 mL of hydrofluoric acid (4.2.3), heat to near boiling, and maintain the temperature on an 80°C hot plate. 10 min. Allow to cool to room temperature, transfer to a 100 mL plastic volumetric flask, dilute to the mark with water, and mix well. After the precipitate settles, use a double-layer chromatography system. Slow-speed dry filtration with filter paper.

4.5.4.2 Transfer

10.00 mL of the test solution (4.5.4.1) into a 50 mL plastic volumetric flask, add 5 mL of boric acid solution (4.2.6) and 1 mL of nitric acid. (4.2.2) Dilute with water to the mark, mix well, and then test.

Note. If the instrument is equipped with a hydrofluoric acid resistant injection system, boric acid solution can be omitted (4.2.6).

4.5.5 Preparation of Standard Solutions Transfer 0 mL,

0.10 mL,

0.50 mL,

10.00 mL of a mixed molybdenum and tungsten standard solution, respectively. (4.2.9) Add 4 drops (approximately

0.2 mL) of hydrofluoric acid (4.2.3) and 5 mL of boric acid solution (4.2.6) to seven 50 mL plastic volumetric flasks. 1 mL of nitric acid (4.2.2) was diluted to the mark with water and mixed well. The molybdenum and tungsten concentrations of this series of standard solutions were 0 μg/mL and 0 μg/mL, respectively. 0.10μg/mL, 0.50μg/mL, 1.00μg/mL, 2.50μg/mL, 5.00μg/mL, 10.00μg/mL.

Note. If the instrument is equipped with a hydrofluoric acid resistant injection system, boric acid solution can be omitted (4.2.6).

4.5.6 Measurement

4.5.6.1 Drawing the working curve Under the selected instrument operating conditions, the series of standard solutions (4.5.5) were subjected to argon plasma spectroscopy using the selected analytical lines. Plot a working curve with the light signal intensity of the analyte as the ordinate and the mass concentration of a series of standard solutions as the abscissa. The linear correlation coefficient should not exceed [value missing]. Less than 0.9995.

4.5.6.2 Determination of blank test solution After the working curve (4.5.6.1) meets the requirements for determination, the blank test (4.5.3) solution is subjected to argon plasma analysis using the selected analytical lines. Bulk spectroscopy determination. The instrument automatically processes data based on the working curve, calculates and outputs the mass concentration of the analyte in the blank test solution.