

ICS 77.120.99
CCS H 65

XB

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

XB/T 601.3-2008

**Chemical analysis methods of lanthanum boride -
Determination of tungsten content - Inductively coupled plasma
atomic emission spectrographic method
(English Translation)**

Issued on: February 1, 2008

Implemented on: July 1, 2008

**Issued by: National Development and Reform Commission of the
People's Republic of China**

Table of Contents

Foreword
1 Scope
2 Principles of the method
3 Reagents
4 Instruments
5 Sample
6 Analysis steps
6.1 Sample
6.2 Determine the quantity
6.3 Preparation of analysis test solution

Foreword

This standard is divided into the following 5 parts.

XB/T 601.1-2008 Methods for chemical analysis of lanthanum hexaboride. Determination of boron content. Acid-base titration XB/T 601.2-2008 Methods for chemical analysis of lanthanum hexaboride Determination of iron, calcium, magnesium, chromium, manganese, and copper content inductively coupled plasma Emission spectroscopy XB/T 601.3-2008 Methods for chemical analysis of lanthanum hexaboride-Determination of tungsten content-Inductively coupled plasma emission spectrometry XB/T 601.4-2008 Methods for chemical analysis of lanthanum hexaboride. Determination of carbon content. High frequency induction combustion and infrared absorption method.

XB/T 601.5-2008 Methods for chemical analysis of lanthanum hexaboride-Determination of acid-soluble silicon content-Silicon molybdenum blue spectrophotometric method this part is part 3.

This part was proposed and managed by the National Rare Earth Standardization Technical Committee.

1 Scope

This section specifies the determination method of tungsten content in lanthanum hexaboride.

This section applies to the determination of tungsten content in lanthanum hexaboride. Measurement range. 0.0010% to 0.10%.

2 Principles of the method

The sample is dissolved in nitric acid, tungsten is complexed with hydrofluoric acid, and the lanthanum matrix is separated at the same time. Boric acid remains in the sample solution. The matrix matching method is used to match the background.

The subtraction method eliminates the influence of the matrix, and is determined by argon plasma spectrometry.

3 Reagents

3.1

Nitric acid ($\rho=1.42\text{g/mL}$).

3.2

Hydrofluoric acid ($\rho=1.14\text{g/mL}$).

3.3

Tungsten standard storage solution. Weigh 0.1261g tungsten trioxide (WO_3) $>99.9\%$, dried at 110°C for 1h, and place it in 200mL in the beaker, add 1g of sodium hydroxide and a little water, heat to dissolve it until it is clear, then transfer it to a 100mL volumetric flask after cooling, dilute to the mark with water, and mix uniform. 1mL of this solution contains 1,000 mg of tungsten. Store in a plastic bottle.

3.4

Tungsten standard solution. Pipette 10.00mL tungsten standard stock solution (3.3) into a 100mL volumetric flask, add 2mL hydrofluoric acid (3.2) Dilute to the mark with water and mix well. This solution 1mL contains 100 μg of tungsten. Store in a plastic bottle.

3.5

Tungsten standard solution. Pipette 10.00mL tungsten standard solution (3.4) into a 100mL volumetric flask, add 2mL hydrofluoric acid (3.2);

Dilute to the mark with water and mix well. This solution 1mL contains tungsten 10.0 μg . Store in a plastic bottle.

3.6

Boric acid solution (50g/L).

3.7

Argon gas. purity $> 99.99\%$.

4 Instruments

4.1

Inductively coupled plasma emission spectrometer. the reciprocal line dispersion rate is not more than 0.25nm/mm.

4.2

Light source. Argon plasma light source. The use power is not more than 2kW.

5 Sample

The sample is mixed and dried at 105°C to 110°C for 1 h, then cooled to room temperature in a desiccator and weighed immediately.

6.1 Sample

Weigh 0.5g sample (5) to the nearest 0.0001g.

6.2 Determine the quantity

Weigh two samples, measure in parallel, and take the average value.

6.3 Preparation of analysis test solution

Place the sample (6.1) in a.200mL polytetrafluoroethylene beaker, add 5mL of water to wet the sample. Slowly add nitric acid dropwise while shaking XB/T 601.3-2008