

ICS 77.120.99  
CCS H 65

**XB**

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

**XB/T 601.5-2008**

Replacing XB/T 601.6-1993

---

**Chemical analysis methods of lanthanum boride -  
Determination of acid dissolving silicon content - Silicon  
molybdenum blue spectral luminosity 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 and utensils |
| 5 Sample                   |
| 6 Analysis steps           |
| 6.1 Sample                 |

### 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 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 5. This part is a reference to XB/T 601.6-1993 "Methods for chemical analysis of lanthanum hexaboride, silicon molybdenum blue spectrophotometric method Revision of "Determination of Acid Soluble Silicon Amount". Compared with XB/T 601.6-1993, the main changes are as follows.

---Edited the standard text;

--- Added precision (repeatability) clause.

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

## 1 Scope

This section specifies the method for determining the acid-soluble silicon content in lanthanum hexaboride.

This section applies to the determination of acid-soluble silicon content in lanthanum hexaboride. Measurement range. 0.0020%~0.10%.

## 2 Principles of the method

The sample is decomposed by dilute nitric acid. In a 0.1mol/L sulfuric acid medium, orthosilicic acid and ammonium molybdate generate yellow silicomolybdenum heteropoly acid, which improves sulfuric acid.

Concentration to 1.2mol/L to eliminate the interference of phosphorus and arsenic, use ascorbic acid to reduce silicomolybdenum yellow to silicomolybdenum blue, with a spectrophotometer wavelength of 660nm Measure the absorbance at the place. Use the working curve to find the silicon content.

## 3 Reagents

### 3.1

Nitric acid (1+1), pure superior grade.

### 3.2

Hydrochloric acid (1+1), excellent grade pure.

### 3.3

Sulfuric acid (1+1), pure superior grade.

### 3.4

Sulfuric acid (1 + 17), pure superior grade, stored in plastic bottles.

### 3.5

Ammonia (1+1), ultra-pure, stored in a plastic bottle.

### 3.6

Ammonium molybdate  $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$  solution (50g/L), stored in a plastic bottle.

### 3.7

Ascorbic acid solution (30g/L), prepared when used.

### 3.8

Silicon standard stock solution. Weigh 0.2139g of spectrally pure dioxygen that has been pre-fired at 950°C for 2h and cooled to room temperature in a desiccator Add 5g of

anhydrous sodium carbonate (superior grade) to the silicide in a platinum crucible, and melt it at 1000°C until it becomes transparent. After cooling, it is leached with hot water and heated to dissolve it.

Cool to room temperature, transfer to a 1000mL volumetric flask, dilute to the mark with water, and mix. Store in a plastic bottle, this solution is 1mL Contains 100µg silicon.

### **3.9**

Silicon standard solution. Pipette 50.0mL silicon standard stock solution (3.8) into a 1000mL volumetric flask, dilute to the mark with water, and mix Evenly, store in a plastic bottle. This solution 1mL contains 5µg silicon.

### **3.10**

p-nitrophenol solution (1g/L).

## **4 Instruments and utensils**

### **4.1**

Spectrophotometer.

### **4.2**

Teflon beaker with a capacity of 100 mL.

## **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 the sample (5) according to Table 1, with an accuracy of 0.0001g.

XB/T 601.5-2008