

Rare Earth Standard of the People's Republic of China

XB/T 617.6—2014

Chemical analysis methods for neodymium  
iron boron alloy Part 6: Determination  
of carbon content —— High  
frequency-infrared absorption method  
钕铁硼合金化学分析方法 第6部分：碳量  
的测定 高频-红外吸收法  
(English Translation)

Issue Date: 2014-10-14

Implementation Date: 2015-04-01

## Foreword

SAC/TC 229 China Rare Earth Standardization Technical Committee is in charge of this English translation. In case of any doubt about the contents of the English translation, the Chinese original shall be considered authoritative.

The XB/T 617 *Chemical analysis methods of neodymium iron boron alloy* consists of the following seven parts:

- Part 1: *Determination of total rare earth content - Oxalate gravimetry*
- Part 2: *Determination of fifteen REO relative contents*
- Part 3: *Determination of boron, aluminum, copper, cobalt, magnesium, silicon, calcium, vanadium, chromium, manganese, nickel, zinc and gallium contents-Inductively coupled plasma atomic emission spectrometry*
- Part 4: *Determination of iron content - The potassium dichromate titrimetry*
- Part 5: *Determination of zirconium, niobium, molybdenum, tungsten and titanium contents - Inductively coupled plasma atomic emission spectrometry*
- Part 6: *Determination of carbon content - High frequency-infrared absorption method*
- Part 7: *Determination of oxygen and nitrogen contents - Impulse-Infrared and impulse-thermal conductance absorption method*

This is the sixth part of XB/T 617.

This standard is drafted in accordance with the rules given in the GB/T 1.1-2009.

This standard was prepared by the SAC/TC 229.

# Chemical Analysis Methods for Neodymium Iron Boron Alloy

## Part 6: Determination of Carbon Content —— High Frequency-infrared Absorption Method

### 1 Scope

This part specifies a method for the determination of carbon content in neodymium iron boron alloy.

This part is applicable to the determination of carbon content in neodymium iron boron alloy. The determination range is 0.005 0% -0.50%.

### 2 Principle

In the presence of fluxing agent, pure oxygen gas-flow is imported into the high frequency induction furnace to sufficiently burn the sample at high temperature. Carbon is oxidized to carbon dioxide. Then carbon dioxide is loaded into carbon dioxide infrared cell by the carrier gas for the determination of carbon content.

### 3 Reagent and material

3.1 Tungsten granule  $\omega(\text{C}) < 0.000\ 3\%$ ,  $\omega(\text{S}) < 0.000\ 3\%$ .

3.2 Tin granule  $\omega(\text{C}) < 0.000\ 5\%$ ,  $\omega(\text{S}) < 0.000\ 8\%$ .

3.3 Reference material: select three suitable state-level steel reference materials.

3.4 Oxygen [ $\omega(\text{O}) \geq 99.5\%$ ].

3.5 Carbon crucible: it shall be pre-ignited in an over-1200°C high-temperature furnace for 6 to 8 hour, then place it in a desiccator, and cool for later use (valid within 2 days)

### 4 Apparatus

High frequency-infrared carbon and sulfur analyzer.

### 5 Sample

The sample for the determination of carbon content is in the form of chips or blocks.

Weigh the sample immediately after sampling.

### 6 Procedure

#### 6.1 Test portion

Weigh 0.4-0.5g sample (Chapter 5) to the nearest 0.0001g.

#### 6.2 Parallel test

Carry out the parallel determination for test portions (6.1) and calculate the mean value.

#### 6.3 Apparatus preparation

Inspect the reagent for the apparatus to ensure that it is available. Turn on the analyzer, preheat it and perform system inspection according to the analyzer's operating instructions.

#### 6.4 Blank calibration

6.4.1 Put 1.2 g of tungsten granule (3.1) and 0.3 g of tin granule (3.2) in the carbon crucible (3.5). Turn on the high-frequency furnace and place the carbon crucible on the crucible holder. The crucible is raised into the combustion tube during the measurement.

6.4.2 After burning in high frequency furnace, repeat 6.4.1 3–5 times. The average blank value of carbon shall be less than 0.000 5% before the next step of determination.

#### 6.5 Curve calibration

Weigh two reference materials (3.3), carry out the parallel determination according to the operation procedures (6.6) and calibrate, then repeat once. The fluctuation of the results shall be within the tolerant fluctuation for the standard value.

#### 6.6 Determination

Turn on the high-frequency furnace, put the test portion (6.1) into the carbon crucible (3.5) and add 1.2 g of tungsten granule (3.1) and 0.3 g of tin granule (3.2). Place the crucible on the crucible holder, raise the crucible into the combustion tube to burn. Record the percentage of carbon content (if the analyzer cannot display the results automatically, calculate the results according to Chapter 7).

### 7 Expression of results

The carbon content, expressed as mass fraction, is calculated by formula (1):

$$\omega(C) = \frac{m_1}{m_0} \times 100\% \quad \dots\dots\dots (1)$$

Where

$m_1$ : is the mass, in grams (g), of carbon in the test portion,

$m_0$ : is the mass, in grams (g), of test portion,

### 8 Precision

#### 8.1 Repeatability

The absolute difference, of the determined values obtained from two independent determinations under the repeatability conditions, is not greater than the repeatability limit ( $r$ ), which is in the range of the following average values. The cases that the absolute difference is greater than the repeatability limits ( $r$ ) are less than 5%. The repeatability limit ( $r$ ) is calculated by linear interpolation method on the basis of the data listed in Table 1.

Table 1

Carbon mass fraction / %	Repeatability limit ( $r$ ) / %
0.009 1	0.000 8
0.014	0.002
0.019	0.005
Note: Repeatability limit ( $r$ ) is $2.8 \times S_r$ , $S_r$ is Repeatability standard deviation.	

## 8.2 Tolerance

The difference of the analytical results among laboratories shall be no more than the tolerances listed in Table 2.

Table 2

Carbon mass fraction / %	Tolerance / %
0.005 0~0.010 0	0.001 5
>0.010 0~0.050	0.004 5
>0.050~0.10	0.010
>0.10~0.50	0.03

## 9 Quality guarantee and control

Verify the validity of the method by reference materials each week. National or industrial grade reference materials shall take the priority to the self-made controlling sample. Try to find out the cause in case of abnormalities, correct the mistakes then verify again.