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

XB/T 617.4-2014

**Chemical analysis methods of neodymium iron boron alloy -
Part 4: Determination of iron content - Potassium dichromate
titrimetry**

钕铁硼合金化学分析方法 第4部分：铁量的测定 重铬酸钾滴定法

(English Translation)

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Foreword

This document was issued on 14 October 2014 by the Ministry of Industry and Information Technology of the People's Republic of China, and has applied since 1 April 2015.

It is an XB/T standard: a recommended industry standard of China's rare earth industry.

XB/T 617, Chemical analysis methods for neodymium iron boron alloy, consists of seven parts: Part 1, determination of total rare earth content; Part 2, determination of fifteen rare earth elements; Part 3, determination of boron, aluminium, copper, cobalt, magnesium, silicon, calcium, vanadium, chromium, manganese, nickel, zinc and gallium contents by inductively coupled plasma atomic emission spectrometry; Part 4, determination of iron content by potassium dichromate titration; Part 5, determination of zirconium, niobium, molybdenum, tungsten and titanium contents by inductively coupled plasma atomic emission spectrometry; Part 6, determination of carbon content by high-frequency combustion infrared absorption; Part 7, determination of oxygen and nitrogen contents by pulse heating infrared absorption and thermal conductivity.

This part is Part 4 of XB/T 617.

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

This part is under the jurisdiction of the National Technical Committee on Rare Earth of Standardization Administration of China (SAC/TC 229).

1 Scope

This part of XB/T 617 specifies the method for the determination of iron content in neodymium iron boron alloy.

This part is applicable to the determination of iron content in neodymium iron boron alloy. The determination range is 50.00 % to 80.00 %.

2 Principle

The sample is decomposed with hydrochloric acid. In sulfuric-phosphoric acid medium, iron(III) is reduced to iron(II) with tin(II) chloride and titanium(III) chloride as combined reductants, and the iron(II) is titrated with potassium dichromate standard solution to a purple end point, with sodium diphenylamine sulfonate as indicator.

3 Reagents and materials

3.1

Hydrochloric acid ($\rho = 1.19 \text{ g/mL}$).

3.2

Sulfuric acid ($\rho = 1.84 \text{ g/mL}$).

3.3

Phosphoric acid ($\rho = 1.70 \text{ g/mL}$).

3.4

Hydrochloric acid (1+2).

3.5

Hydrochloric acid (1+9).

3.6

Tin(II) chloride solution (50 g/L)

Dissolve 5 g of tin(II) chloride in 10 mL of hydrochloric acid (3.1), dilute to 100 mL with water and mix.

3.7

Titanium(III) chloride solution

Dilute titanium(III) chloride solution (15 %) ten times with hydrochloric acid (3.5); prepare when required.

3.8

Sodium tungstate solution (250 g/L)

Dissolve 25 g of sodium tungstate in water (filter if turbid), add 5 mL of phosphoric acid (3.3), dilute to 100 mL with water and mix.

3.9

Sulfuric-phosphoric acid mixture, prepared from sulfuric acid (3.2) and phosphoric acid (3.3).

3.10

Potassium dichromate standard titration solution, $c(K_2Cr_2O_7) = 0.01667 \text{ mol/L}$: weigh 4.9030 g of reference potassium dichromate, previously dried at 150°C for 1 h and cooled in a desiccator, into a 500 mL beaker, dissolve in water, transfer to a 1,000 mL volumetric flask, dilute to the mark with water and mix.

3.11

Potassium dichromate solution, $c(K_2Cr_2O_7)$ approx. 0.0030 mol/L : dissolve 1.7651 g of reference potassium dichromate in water in a 500 mL beaker, transfer to a 2,000 mL volumetric flask, dilute to the mark with water and mix.

3.12

Sodium diphenylamine sulfonate indicator (2 g/L), stored in a brown glass bottle.

4 Sample

The sample shall be weighed immediately after sampling.