GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

UDC 669.14/.15: 543.06
H 11

GB 223.62-88

Replacing GB 223.3-81 Method III

# Methods for Chemical Analysis of Iron, Steel and Alloy - The Butyl Acetate Extraction Photometric Method for the Determination of Phosphorus Content

**Issued on: February 05, 1988** **Implemented on: February 01, 1989**

**Issued by: National Bureau of Standards;**

**Approved by: Ministry of Metallurgical Industry of PRC, January 18, 1988.**

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# Methods for Chemical Analysis of Iron, Steel and Alloy - The Butyl Acetate Extraction Photometric Method for the Determination of Phosphorus Content

This Standard is applicable to the determination of phosphorus content in pig iron, iron powder, carbon steel, alloy steel, high-temperature alloy, precision alloy. The measuring range is 0.001% ~ 0.05%.

This Standard complies with GB 1467-78 *Method for Chemical Analysis of Metallurgy Product – General Rules and Regulations*.

This Standard complies with GB 7729-87 *Chemical Analysis of Metallurgical Products – General Rule for Spectrophotometric Methods*.

## 1 Method Summary

In 0.65~1.63mol/l nitric acid medium, phosphorus and ammonium molybdate generate phosphorus-molybdenum heteropoly acid, which is extracted by butyl acetate; use stannous chloride to reduce and back-tract the phosphorus-molybdenum heteropoly acid into aqueous phase; then measure the absorbance at the wavelength of 680nm.

The extraction solution contains the following amounts of elements like 2.5μg of zirconium, 20μg of arsenic, 25μg of niobium, tantalum, 50μg of titanium, 500μg of cerium, 1.5mg of tungsten, 2mg of steel, 3mg of cobalt, 5mg of chromium, aluminum, and 50mg of nickel; which shall not interfere with the determination.

When the above limits are exceeded, the arsenic shall be removed by hydrochloric acid, and hydrobromic acid; vanadium shall be reduced by ferrous; zirconium shall be covered by hydrofluoric acid; after oxidizing into high price, the chromium shall be removed by hydrochloric acid; in the EDTA ammoniacal solution, the tungsten shall take beryllium as carrier, and precipitate the phosphorus; niobium, titanium, zirconium, tantalum shall use copper and iron reagents, and chloroform to extract and remove.

## 2 Reagents

**2.1** Oxalic acid: solid.

**2.2** Copper and iron reagents: solid.

**2.3** Boric acid: solid.

**2.4** Butyl acetate.

**2.5** Chloroform.

**2.6** Hydrobromic acid (ρ 1.49g/ml).

**2.7** Perchloric acid (ρ 1.67g/ml)

**2.8** Hydrochloric acid (ρ 1.19g/ml)

**2.9** Hydrochloric acid (1+5).

**2.10** Nitric acid (1+2).

**2.11** Nitric acid (1+2), it is prepared by boiling nitric acid (ρ 1.42g/ml), then remove the nitrogen dioxide, and cooling off.

**2.12** Sulfuric acid (1+2).

**2.13** Hydrofluoric acid (1+10).

**2.14** Ammonium hydroxide (ρ 0.90g/ml).

**2.15** Ammonium hydroxide (1+50).

**2.16** Ferrous sulfate solution (5%): every 100ml of solution contains 1ml of sulfuric acid (1+1).

**2.17** Sodium nitrite solution (10%).

**2.18** Boric acid solution (2%).

**2.19** Ammonium molybdate solution (10%).

**2.20** Stannous chloride solution (1%): take 1g of stannous chloride ($SnCl_2 \cdot 2H_2O$); dissolve into 8ml of hydrochloric acid (2.8); dilute by water to 100ml; it shall be prepared for current use.

**2.21** Beryllium sulfate solution (2%): prepare with sulfuric acid (1+100).

**2.22** Disodium ethylenediaminetetraacetic acid (EDTA for short) solution (10%).

**2.23** Copper and iron reagent solution (6%).

**2.24** Standard phosphorus solution.

oxidized into hexavalent; add 2~3ml of hydrochloric acid (2.8) to volatilize chromium; repeat the operation for 2~3 times; continue to vaporize till the internal conical flask is transparent and back-flows for 3~4min; vaporize again till nearly dry; cool off.

### **3.3.1.3** Specimen with excessive-content of arsenic

Vaporize the solution specimen till smoke according to 3.3.1.1; cool off slightly; add 10ml of hydrochloric acid (2.8), 5ml of hydrobromic acid (2.6) to expel the arsenic; continue to heat till the internal conical flask is transparent and back-flows 3~4min; vaporize again till nearly dry; cool off.

### **3.3.2** Salts dissolvement and interfering elements treatment

### **3.3.2.1** General specimen

Add 30ml of nitric acid (2.10); heat and dissolve salts; titrate sodium nitrite solution (2.17) till chromium is reduced to low-valent and continue to titrate for several excessive drops; boiling it to remove nitrogen oxide; cool off to the room temperature. Transfer the solution into 100ml volumetric flask; dilute with water to the scale; mix evenly.

### **3.3.2.2** Tungsten-containing specimen

Use 20ml of water to dissolve the salts obtained from 3.3.1; add 10ml beryllium sulfate solution (2.21), 10ml of EDTA solution (2.22), 2g of oxalic acid (2.1); use ammonium hydroxide (2.14) to neutralize till pH is 3~4; dilute with water to about 90ml; boiling for 2~3min; then add 10ml of ammonium hydroxide (2.14); boiling for 1min; cool off to the room temperature; filter the solution; wash by ammonium hydroxide (2.15); use water to wash the precipitation into the original conical flask; add 30ml of nitric acid (2.10) to dissolve the precipitation remained on the filter paper; discard the filter paper after cleaning; then follows the procedures from titrating sodium nitrite solution (2.17) in 3.3.2.1.

### **3.3.2.3** Zirconium-containing specimen

Perform as per 3.3.2.1 till after cooling off to the room temperature; add 5ml of hydrofluoric acid (2.13), shake evenly; add 20ml of boric acid solution (2.18); then transfer the solution into 100ml volumetric flask; dilute with water to the scale; mix evenly.

### **3.3.2.4** Specimen containing titanium, niobium, zirconium, tantalum

Add 10ml of water and 15ml of sulfuric acid (2.12) to dissolve the salts obtained from 3.3.1; titrate sodium nitrite solution (2.17) to reduce to hexavalent chromium; then boiling and remove the nitrogen oxide; take off; add 5ml of hydrofluoric acid (2.13) when it is hot; shake evenly; cool off to the room temperature. Transfer the solution into 100ml volumetric flask; dilute with water to the scale; mix evenly.

**------ The End ------**