ICS 77.080
CCS H11

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 223.61-1988

Methods for chemical analysis of iron, steel and alloy--The ammonium phosphomolybdate volumetric method for the determination of phosphorus content

钢铁及合金化学分析方法 磷钼酸铵容量法测定磷量

Issued on: February 5, 1988 **Implemented on: February 1, 1989**

Issued by: State Bureau of Standards

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1 Scope

GB/T 223.61-1988 is the Chinese national standard on methods for chemical analysis of iron, steel and alloy--the ammonium phosphomolybdate volumetric method for the determination of phosphorus content, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 5 February 1988 by the State Bureau of Standards, and has been in force since 1 February 1989. Classification: ICS 77.080, CCS H11. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

Dissolve the sample with oxidizing acid; at nitric acid acidity of

2.2 mol/L, add ammonium molybdate to generate ammonium phosphomolybdate deposition; after filtering, dissolve with excessive standard sodium hydrate solution; taking phenolphthalein solution as indicator, back-titrate the excessive sodium hydrate with nitric acid standard solution till the pink just disappears (the end-point) (about pH8). The general reaction formula of dissolving ammonium molybdate precipitation with sodium hydrate is as. Arsenic of less than 100μg, tantalum < 500 μg, zirconium, vanadium or niobium < 1 mg, wolfram < 8 mg, titanium < 10 mg and silicon < 20 mg contained in the test solution, may not interfere the determination; exceeding the limits above, remove arsenic by hydrochloric acid and hydrobromic acid; mask zirconium, niobium, tantalum, titanium and silicon by hydrofluoric acid; reduce vanadium by hydroxylamine hydrochloride; remove wolfram (in ammonia solution at presence of EDTA) by beryllium carrier.

2 Reagents

2.1 Ammonium nitrate. solid.

2.2 Ethylene diamine tetraacetic acid disodium (hereinafter referred to as EDTA disodium). solid.

2.5 Nitric acid (rho=

2.6 Nitric acid (1+3).

2.7 Nitric acid (2+100).

2.10 Ammonium hydroxide (rho=

0.90 g/ml).

2.11 Ammonium hydroxide (5+95).

2.12 Sodium nitrite solution (10%).

2.13 Beryllium sulfate solution (2%). prepare with sulfuric acid (1+100).

2.14 Hydroxylamine hydrochloride solution (10%). prepare just before use.

2.15 Ammonium molybdate solution. weigh 135 g of ammonium molybdate [(NH4)6·Mo7O24·4H2O], dissolve it in warm water and cool it; dilute to 1000 ml with water, stir, and slowly pour into 1000 ml of nitric acid (2+3, mix; add 5 mg of diammonium hydrogen phosphate, standing 24h; and filter it with slow filter paper before use.

2.16 Neutral water. boil the distilled water to remove carbon dioxide, and then cool it by following water. Prepare just before use.

2.17 Phenolphthalein solution. weigh

0.25 g of phenolphthalein, dissolve it in 30ml of alcohol, and dilute to 50 ml with water.

2.18 Standard sodium hydrate solution, C(NaOH) =

0.1 mol/L or C(NaOH) =

0.05 mol/L.

2.18.1 Preparation Weigh 4 g or 2 g of sodium hydrate, dissolve it in 1000 ml of neutral water (2.16), and store the solution in a sealed plastic bottle.

2.18.2 Calibration Weigh 0.3000 g of reference potassium hydrogen phthalate (stove at 105°C in advance, place it in a dryer and cool it to the ambient temperature) (three portion), dissolve them with 80 ml of neutral water respectively, and add 2~3 drops of phenolphthalein solution (2.17) respectively; titrate them with standard sodium hydrate solution (2.18) to light red; the maximum difference of the amount consumption (mL) of standard sodium hydrate solution for the three solutions shall not exceed 0.05ml, the mean shall be taken, and the concentration of standard sodium hydrate solution is taken according to the formula below. Where, C - The mass concentration of the standard sodium hydrate solution, mol/l; m - The mass of potassium hydrogen phthalate weighed, g; V - The mean volume of the standard sodium hydrate solution consumed for calibration, ml; 204.22 - The mole mass of potassium hydrogen phthalate, g/mol.

2.19 Standard nitric acid solution, C(HNO3)=

0.1 mol/l or C(HNO3)=

0.05 mol/l.

2.19.1 Preparation Pipette

3.3 ml of nitric acid (2.5) that has been remove nitric oxide and cooled up, and dilute it to 1000 ml with water, mix.

2.19.2 Calibration Pipette

25.00 ml of standard sodium hydrate solution (2.18) (three portions), add 50 ml of neutral water (2.16) and 3 drops of phenolphthalein (2.17) respectively, titrate them with standard nitric acid solution (2.19.1) respectively till the red disappears; the maximum difference of the consumptions of standard nitric acid solutions (2.19.1) for three portions of the solution shall not exceed

0.05 mL, and the mean (V1) shall be taken according to the formula below. Where, K - The coefficient for reduction of standard nitric acid solution to standard sodium hydrate solution;

V - The mean volume of standard nitric acid solution used for titrating, ml; 25.00 - The volume of the standard sodium hydrate solution (2.18) pipetted, ml.

3 Procedure

3.1 Sample Amount The sample shall be selected as required by Table 1. Table 1

3.2 Blank Test Carry out the blank test in parallel with the test with the sample.

3.3.1 Sample dissolution

3.3.1.1 Common sample Place the sample (3.1) into a 300 ml conical beaker, and add nitric acid as required by Table 1, and heat for dissolving [for indissoluble sample, add appropriate amount of hydrochloric acid (2.8) dropwise to promote dissolution]. Add perchloric acid as required by Table 1, heat it till the fume appears, slightly cool, add

0.5 ml of hydrofluoric acid (2.4) dropwise, and heat again and make it fume till the conical beaker becomes transparent inside and refluxes 3~ 4 min (it the sample contains over 10 mg of manganese, add more 7~8 ml of perchloric acid (2.3), heat and make it fume till the conical beaker becomes transparent inside and refluxes 20~25 min, so as to oxidize phosphorus completely); and continue to evaporate it to nearly dry, and cool it up.

3.3.1.2 Sample containing over 50 mg of chromium Proceed the procedure as

3.3.1.1 till adding

0.5 ml of oxygen-fluorine acid (2.4); after this, re-evaporate and make it fume till chromium is oxidized to hexavalent one; add hydrochloric acid (2.8) dropwise by several times, and repeat 2~3 times; and re-evaporate till the conical beaker becomes transparent inside and refluxes 3~4 min; and re-evaporate again to nearly dry, and cool it up.

3.3.1.3 Sample containing over limit of arsenic Proceed the procedure as

3.3.1.1 till adding

0.5 ml of hydrofluoric acid (2.4); after this, re-evaporate till fume appears; take it down and cool it slightly; add 10 ml of hydrochloric acid (2.8) and 5 ml of hydrobromic acid (2.9); heat and evaporate till white fume appears and remove arsenic. Continue to evaporate till the conical beaker becomes transparent inside and refluxes 3~4 min; and re-evaporate it to nearly dry, and cool it up.

3.3.2 Salt dissolution Add 5ml of nitric acid (2.5) and 40 ml of water to dissolve the salt, add sodium nitrite solution (2.12) dropwise to reduce chromium to low-valency one, and add 2 excessive drops, and boil for 1~2 min; remove nitric oxide.