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Method for chemical analysis of iron, steel and alloy

The diantipyrylmethane photometric method for the

determination of titanium content

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Methods for chemical analysis of iron, steel and alloy

The diantipyrylmethane photometric method for the determination of titanium content

1 Subject content and application scope

This standard specifies the determination of titanium content by using the diantipyrylmethane photometric method.

This standard applies to the determination of titanium content in nickel base and iron-nickel based alloy. Determination range: 0.010% ~ 2.400%.

2 Method summary

Use acid to dissolve the sample; in 1.2~3.6 mol/L hydrochloric acid medium, use ascorbic acid to reduce iron; the titanium and diantipyrylmethane will form a yellow complex; determine its absorbance.

In color liquid, the vanadium content is less than 2 mg; the molybdenum content is less than 1.5 mg; the tungsten content is more than 1 mg [Translator note: should be "less than 1 mg". However, there is no Corrigendum from Chinese Authority yet]; the contents of niobium, tantalum, zirconium, and rare earth are less than 0.5 mg; the boron content is less than 0.2mg; the contents of tin, antimony, lead, and bismuth are less than 0.1 mg; there will be no interference. When the tungsten content is more than 1 mg, use citric acid to complex; when the molybdenum content is more than 1.5 mg, add molybdenum with the same content in the working curve to offset interference.

3 Reagents

3.1 Potassium pyrosulphate.

3.2 Hydrochloric acid (pl. 19 g / mL).

3.3 Hydrochloric acid (2+1).

3.4 Nitric acid (pl. 42 g/mL).

3.5 Ammonium hydroxide (p 0.90 g/mL).

4.2 Determination

4.2.1 Place the sample (4.1) in a 150 mL conical flask; add 10~20 mL of mixed-acid of hydrochloric acid (3.2) and nitric acid (3.4) in appropriate proportion heat to dissolve; add 15 mL of sulfuric acid (3.6) (For sample that contains tungsten, directly add 30 mL of sulfuric acid (3.7); heat to dissolve; drop-add nitric acid (3.4) to destroy carbide). Evaporate it until oleum emits; take down to cool slightly.

4.2.2 Add 50 mL of water; heat to dissolve the salts. Take down to cool to room temperature. If the tungsten content in the transferred sample solution is more than 1 mg, after oleum emits, cool slightly; add 10 mL of ammonium citrate solution (3.11) and 10 mL of water; cool slightly; add 35 mL of ammonium hydroxide (3.5); heat until the tungstic acid is dissolved; and boil until there is no ammonia odor. Cool slightly; add 15 mL of sulfuric acid (3.6); cool to room temperature.

Note: the insoluble residues usually contain little titanium, so it can be ignored. If the titanium in residues needs to be recycled, use slow filter paper with a small amount of pulp to filter the solution (4.2.1~4.2.2) in a 100 mL volumetric flask. Use sulfuric acid (3.8) to wash the filter paper; retain the filtrate. Transfer the residues and filter paper to a platinum crucible to ash and burn; add 2 drops of sulfuric acid (3.6) and 2~3 mL of hydrofluoric acid (3.9); evaporate them to dry. Add 0.5 g of potassium pyrophosphate (3.1) to melt; use a small amount of sulfuric acid (3.7) to leach; combine them in filtrate.

4.2.3 Transfer the solution (4.2.2) in a 100 mL volumetric flask; dilute with water to the scale; mix uniformly.

4.2.4 Transfer two portions of 10.00 mL of solution (4.2.3) (transfer 5.00 mL, when the titanium content is more than 0.5%); place into two 50 mL volumetric flasks respectively; Conduct according to 4.2.5 and 4.2.6.

4.2.5 Color liquid: add 5 mL of ascorbic acid solution (3.10); mix uniformly; add 15 mL of hydrochloric acid (3.3); place for 5 minutes (the solution temperature shall be above 20 °C, so as to ensure the reduction of iron); add 15 mL of diantipyrylmethane solution (3.12); use water to dilute to the scale; mix uniformly. Place it under room temperature for more than 40 minutes.

4.2.6 Reference solution: Conduct according to 4.2.5, except that diantipyrylmethane solution is not added.

4.2.7 Transfer part of color liquid in absorption vessel (see table 2); use reference solution as the reference; measure the absorbance at wavelength of 390 nm of spectrophotometer.

4.2.8 Find the corresponding titanium content in color liquid from the working curve, according to the measured absorbance of solution.

4.3 Working curve drawing

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