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Methods for chemical analysis of iron, steel and alloy - The sodium arsenite-sodium nitrite titrimetric method for the determination of manganese content

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Methods for chemical analysis of iron, steel and alloy - The sodium arsenite-sodium nitrite titrimetric method for the determination of manganese content

This standard applies to the determination of the amount of manganese in pig iron, carbon steel, alloy steel, and iron powder. Measuring range is 0. 10% ~ 2. 50%.

This standard follows the requirements GB 1467-78 “Method for chemical analysis of metallurgy product - General rules and regulations”.

1 Method summary

The sample is dissolved in acid; in the sulfuric acid and phosphoric acid medium, the silver nitrate is used as catalyst AND the ammonium persulfate is used to oxidize the manganese to septivalence, AND the sodium arsenite-sodium nitrite standard solution is used for titration. Test solution containing cobalt more than 5 mg will affect the observation of end point, AND it may add nickel to offset the effect of the color of the cobalt ion.

2 Reagent

2.1 Nitric acid (ρ1.42g/ml).

2.2 Nitric acid (2 + 98).

2.3 Hydrochloric acid (ρ1.19g/ml).

2.4 Perchloric acid (ρ1.67g/ml).

2.5 Sulfuric acid (ρ1.84g/ml).

2.6 Sulfuric acid (2 + 3).

2.7 Hydrofluoric acid (ρ1.15 g/ml).

2.8 Ammonium hydroxide (ρ0.90g/ml).

2.9 Sulfuric acid-phosphoric acid mixture: slowly ADD 150ml of sulfuric acid (2.5) and 150ml of phosphoric acid (ρ1.70g/ml) into 700ml of water; STIR it continuously and COOL it down.

2.10 Sulfuric acid-phosphoric acid mixture: MIX the sulfuric acid (2.5), phosphoric acid (ρ1.70g/ml) and water by equal volume; COOL it down.

2.11 Aqua regia: MIX three parts of hydrochloric acid (2.3) and one part of nitric acid (2.1).

2.12 Silver nitrate solution (0.5%): WEIGH 0.5g of silver nitrate; DISSOLVE it into water; ADD a few drops of nitric acid (2.1); USE water to dilute it to 100ml; STORE it into a brown bottle.

2.13 Zinc oxide suspension: WEIGH 50g of zinc oxide; ADD 200ml of water; STIR it during use.

2.14 Ammonium persulfate solution (20%): PREPARE it before use.

2.15 Sodium chloride solution (0.4%): WEIGH 4g of sodium chloride; USE sulfuric acid (2.6) to dissolve it; and DILUTE it to 1l.

2.16 Potassium permanganate solution (0.16%).

2.17 Manganese standard solution.

2.17.1 WEIGH 1.4383g of benchmark potassium permanganate; PLACE it into a 600ml beaker; ADD 30ml of water to dissolve it; ADD 10ml of sulfuric acid (1 +1). ADD hydrogen peroxide (ρ1.10g/ml) to the point when the red is just disappeared; HEAT to boil it for 5 ~ 10 min; COOL it down; TRANSFER it into a 1000ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. AND 1ml of this solution contains 500μg of manganese.

2.17.2 WEIGH 0.5000g of electrolytic manganese [99.99%, electrolytic manganese treatment method: electrolytic manganese is placed into sulfuric acid (5 + 95) for cleaning; after the surface manganese oxide is washed clean, it is taken out and repeated rinsed by distilled water, then placed in the anhydrous ethanol for washing for 4 ~5 times, and taken out and placed in the drier for drying, before use]; PLACE it into a 250ml beaker; ADD 20ml of nitric acid (1 + 3); HEAT to dissolve it; BOIL it to remove all nitrogen oxides; TAKE it off and COOL it to room temperature; TRANSFER it into a 1000ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly; AND 1ml of this solution contains 500μg of manganese.

silicon sample, it may add several drops of hydrofluoric acid (2.7)]; ADD 10ml of sulfuric acid (2.5); HEAT to evaporate it until sulfuric acid smoke is produced; COOL it down; carefully ADD 100ml of water; HEAT to dissolve the salts; ADD ammonium hydroxide (2.8) until a small amount of hydroxide precipitate is produced; then ADD sulfuric acid (2.6) until the precipitate is dissolved; TRANSFER the solution into a 250ml volumetric flask; ADD zinc oxide suspension (2.13) for several times until a large amount of precipitation of hydroxide appears, with an excessive amount of 15ml; after cooling it down; USE water to dilute it to the mark; MIX it uniformly; FILTER it at dry state. PIPETTE 100ml of filtrate; PLACE it into a 300ml conical flask; ADD 15ml of sulfuric acid-phosphoric acid mixture (2.10).

3.2.1.3 Pig iron sample

PLACE the sample (3.1) into a 300ml conical flask; ADD 30ml of sulfuric acid-phosphoric acid mixture (2.9); HEAT to dissolve it at low temperature [if the sample is hard to be dissolved, it may add 1ml of nitric acid (2.1) to assist dissolution; AND if the solution evaporation is too excessive, it may add a small amount of water]; ADD nitric acid (2.1) for oxidization; BOIL it to remove the nitrogen oxides; USE a rapid filter paper to filter it into another 300ml conical flask; USE nitric acid (2.2) to rinse the conical flask and filter paper for several times; ADD water to dilute it to make its volume reach to about 80ml.

3.2.2 Titration

In the solution (3.2.1.1 or 3.2.1.2 or 3.2.1.3); ADD 10ml of silver nitrate solution (2.12) and 10ml of ammonium persulfate solution (2.14); HEAT to boil it at low temperature for 45s; TAKE it off; PLACE it for 2min; USE flow water to cool it to room temperature; ADD 10ml of sodium chloride solution (2.15); SHAKE it uniformly; immediately USE sodium arsenite-sodium nitrite standard solution (2.18) for titration at constant speed (not more than 6ml per minute); when the solution is in slight red, MAKE titration at slower speed until the pink is disappeared, which is the end point [to check whether the titration is excessive, it may add 1 drop of potassium permanganate solution (2.16) in the test solution after titration, AND it indicates that the titration is normal if the solution is in slightly red].

4 Calculation of analysis results

CALCULATE the percentage of manganese in accordance with the formula (2):

$$\mathrm{Mn}(\%)=\frac{TV}{m_0}\times 100 \quad \cdots\cdots (2)$$

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