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Methods for chemical analysis of iron, steel and alloy The neocuproine-chloroform extraction photometric method for the determination of copper content

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Methods for chemical analysis of iron, steel and alloy The neocuproine-chloroform extraction photometric method for the determination of copper content

1 Subject and scope

This Standard specifies the neocuproine-chloroform extraction photometric method for the determination of copper content.

This Standard is applicable to the determination of copper content in pig iron, carbon steel, alloy steel, super alloy and precision alloys. The determination range: 0.010% ~ 1.00%.

2 Method summary

After the sample is acid-solubilized, the copper is reduced to monovalent with hydroxylamine hydrochloride in the presence of citric acid. In the range of PH 5~6, neocuproine and copper generate 2:1 insoluble yellow complex. Extract with chloroform and measure its absorbance. Chromium has negative interference when it is more than 4 mg in the extraction solution; other common coexisting elements have no interference.

3 Reagents

3.1 Absolute ethanol.

3.2 Trichloromethane.

3.3 Hydrochloric acid (ρ 1.19 g/mL).

3.4 Nitric acid (ρ 1.42 g/mL).

3.5 Nitric acid (1+3).

3.6 Perchloric acid (ρ 1.67 g/mL).

3.7 Hydrofluoric acid (ρ 1.15 g/mL).

3.8 Hydrochloric acid-nitric acid mixed acid: mix hydrochloric acid (3.3), nitric acid (3.4) and water according to a proportion of 3+1+3.

flask; add 15 mL of hydrochloric acid (3.3); heat and dissolve; add nitric acid (3.4) in drops for oxidation; add 10 mL of high-fluoride acid (3.6); continue heating till high-fluoride acid smokes and chromium is oxidized to high price; add hydrochloric acid (3.4) in drops to remove chromium as chromium fluoride; high fluoride residue is less than 1 mL; cool for a little while.

4.3.3 In 4.3.1 or 4.3.2 solution, add into 20 mL of water. Heat to dissolve the salt. Cool to room temperature. Move the test solution into a 100 mL flask. Use water to dilute to scale. Well mix (If graphite carbon, tungstic acid and other insoluble matter exit, use medium-speed filter paper for dry filtration and removal).

4.3.4 Pipette 10.00 mL of test solution [pipette 5.00 mL when copper content is 0.50% ~ 1.00%] into a 125 mL separating funnel. Use water to dilute to about 25 mL.

4.3.5 Add 10 mL of sodium citrate solution (3.9), 5 mL of hydroxylamine hydrochloride solution (3.10) and mix well. Add 5 mL of neocuproine ethanol solution (3.11) and mix well. Add 10 mL of chloroform (3.2). After shaking for 30 s, laying for stratification, use cotton wool to filter the organic phase into a 25 mL flask that has been dried and is filled with 5 mL of absolute ethanol (3.1). In the separating funnel, add into 5 mL of chloroform (3.2). After shaking for 15 s, laying for stratification, combine the organic phase into a 25 mL flask. Use absolute ethanol (3.1) to dilute to scale; well mix.

4.3.6 Pipette the color liquid into the absorption dish (use 3 cm absorption dish when copper content is 0.01% ~ 1.00%, 1 cm absorption dish for copper content greater than 0.01% ~ 1.00%). Take the blank solution that is with sample as reference. Measure its absorbance at a wavelength of 456 nm on spectrophotometer. Check the corresponding copper content in the color liquid from the working curve.

4.4 Drawing of working curve

Pipette 0, 1.00, 2.00, 3.00, 4.00, 5.00 mL (pipette 0, 2.50, 5.00, 7.50, 10.00, 12.50 mL when copper content is greater than 0.10% ~ 1.00%) of copper standard solution (3.12.2) into 125 mL separating funnels, respectively. Add water to dilute to about 25 mL. The followings shall be carried out according to 4.3.5, 4.3.6. Use reagent blank solution as reference. Measure its absorbance. Take copper as abscissa and absorbance as ordinate to draw working curve.

5 Calculation of analysis results

Calculate the percentage of copper by the following formula:

------ The End ------