



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

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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**

(English Translation)

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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 (rho 1.19 g/mL).

3.4

Nitric acid (rho 1.42 g/mL).

3.5

Nitric acid (1+3).

3.6

Perchloric acid (rho 1.67 g/mL).

3.7

Hydrofluoric acid (rho 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.

3.9

Sodium citrate solution (30%).

3.10

Hydroxylamine hydrochloride solution (10%).

3.11 2,9-dimethyl-1,10-phenanthroline (neocuproine) ethanol solution (0.1%). weigh 1 g of neocuproine and dissolve in 1000 mL of absolute ethanol (3.1); store in a brown bottle.

3.12 Copper standard solution

3.12.1

Weigh 0.1000 g of copper metal (99.9% above) in a 250 mL beaker.

Add 10 mL of nitric acid (3.4). Cover the watch glass. Slightly heat and dissolve. Add 5 mL of perchloric acid (3.6). Continue heating and evaporation till high perchloric acid smokes. Cool for a little while. Add a small amount of water to dissolve the salt. Move the solution into a 1000 mL flask. Use water to dilute to scale. Well mix. This solution shall contain 100 µg of copper per 1 mL.

3.12.2 Pipette 50.00 mL of copper standard solution (3.12.1) into a 500 mL

flask. Use water to dilute to scale. Well mix. This solution shall contain 10 µg of copper per 1 mL.

4.1 Sample quantity

Weigh the sample according to Table 1.

Table 1

Copper content, % Sample amount, g 0.010 ~ 0.100 0.5000 >0.100 ~ 1.00 0.2500

4.2 Blank test

Perform the blank test with sample.

4.3.1 General sample. place the sample (4.1) in a 150 mL conical flask; add

20 mL of hydrochloric acid-nitric acid mixed acid (3.8); heat till the sample is dissolved [high silicon sample is added with an appropriate amount of hydrofluoric acid (3.7) to assist dissolving]; add 5 mL of high-fluoride acid (3.6), continue heating and evaporation to till high-fluoride acid smokes; high fluoride residue is less than 1 mL; cool for a little while.

4.3.2 High chromium sample. place the sample (4.1) in a 150 mL conical

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.

where,

m₁ - copper content checked from working curve, g;

V₁ - volume of sub-test solution, mL;

V - total volume of test solution, mL;

m - sample amount, g.

6 Precisions

The precisions of this Standard were determined in 1988 by a joint test of ten laboratories at six levels. The precisions are shown in the following table.

Level rang, % (m/m) Repeatability r Reproducibility R 0.010 ~ 1.00 $\lg r = -1.5993 + 0.6856 \lg m$
 $\lg R = -1.1792 + 0.7367 \lg m$ If the difference between two independent test results exceeds the repeatability or reproducibility value calculated by the precision function listed in the table, the two results shall be considered suspicious.