GB

NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

UDC 669.14/15:543.06

GB 223.53-87

Methods for chemical analysis of iron, steel and alloy - The flame atomic absorption spectrophotometric method for the determination of copper content

Approved on: April 03, 1987 **Implemented on: March 01, 1988**

Approved by: Ministry of Metallurgical Industry

Table of Contents

Methods for chemical analysis of iron, steel and alloy - The flame atomic absorption spectrophotometric method for the determination of copper content

This standard applies to the determination of the amount of copper in cast iron, carbon steel, and low alloy steel. Measuring range is 0. 005% ~ 0. 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 decomposed with hydrochloric acid and nitric acid, perchloric acid is added to evaporate it to produce smoke, AND water is used to dissolve the salt. The sample solution is sprayed into the air-acetylene flame, and the copper hollow cathode lamp is used as the light source. The atomic absorption spectrophotometer is used to measure the absorbance at the wavelength of 324.7 nm.

To eliminate matrix effects, when drawing the calibration curve, it shall add the iron the amount of which is similar to that of the sample solution.

2 Reagent

2.1 Hydrochloric acid (ρ 1.19g/ml).

2.2 Nitric acid (ρ1.42 g/ml).

2.3 Perchloric acid (ρ1.67 g/ml).

2.4 Perchloric acid (1 + 1).

2.5 Iron solution: WEIGH 10.00 g of high purity iron (copper content of less than 0.0005%); PLACE it into an 800 ml beaker; ADD 40 of ml hydrochloric acid (2.1); ADD about 10 ml of nitric acid (2.2) to dissolve it; after it is completely dissolved, TRANSFER it into a 500 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. AND 1 ml of this solution contains 20 mg of iron.

2.6 Copper standard solution

2.6.1 WEIGH 1.0000 g of pure copper (more than 99.9%); PLACE it into a 400 ml beaker; ADD 30 ml of (1 + 1) nitric acid to dissolve it; after it is completely dissolved, TRANSFER it into a 1000 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. AND 1 ml of this solution contains 1.0 mg of copper.

2.6.2 PIPETTE 10.00 ml of copper standard solution (2.6.1); PLACE it into a 100 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. AND 1 ml of this solution contains 100 μg of copper.

3 Instruments

Atomic absorption spectrophotometer, equipped with air-acetylene burner and copper hollow cathode lamp. Air and acetylene shall be pure enough (containing no water, oil, or copper) to provide a stable and clear lean flame.

The atomic absorption spectrophotometer used shall reach the following indicators.

3.1 The minimum precision requirements: The calibration solution of the highest concentration is used for absorbance measurements for 10 times, AND the standard deviation shall not exceed 1.0% of the average absorbance; the calibration solution of the smallest concentration (not zero calibration solution) is used for absorbance measurements for 10 times, AND its standard deviation shall not exceed 0.5% of the average absorbance of the calibration solution of the highest concentration.

3.2 Characteristic concentration: The characteristic concentration of copper in a solution having a similar matrix with that of the final measurement sample shall be less than 0.10 μg of copper/ml.

3.3 Detection limit: The detection limit of copper in a solution having a similar matrix with that of the final measurement sample shall be less than 0.10 μg of copper/ml.

3.4 Linearity of calibration curves: The ratio of the slope of the upper 20% concentration range of the calibration curve (expressed as the change in absorbance) to the slope of the lower 20% concentration range shall be not less than 0.70.

4 Analytical procedures

4.1 Sample amount

WEIGH 0.5000 g of sample.

4.2 Blank test

MAKE blank test together with the sample.

4.3 Determination

4.3.1 PLACE the sample (4.1) into a 100 ml beaker; ADD 5 ml of hydrochloric acid (2.1) and 2 ~ 3 ml of nitric acid (2.2); PLACE it on a hot plate to heat to dissolve it; ADD 5 ml of perchloric acid (2.3); HEAT it until smoke is generated; TAKE it off to cool it down; ADD 20 ml of water; HEAT to dissolve the salts; COOL it down; TRANSFER it into a 100 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. If there is precipitation of carbide, silicate, and so son, they shall be filtered at dry state.

4.3.2 PLACE the sample solution in the atomic absorption spectrophotometer at a wavelength of 324.7 nm; USE the air-acetylene flame; USE water to adjust it to zero; MEASURE its absorbance. Combining with the sample solution absorbance and the absorbance of the blank solution accompanied with the sample, FIND the concentration (μg/ml) of copper from the calibration curve.

Note: When the amount of copper is less than or equal to 0.10%, USE the above solution for direct determination. When the amount of copper is greater than 0.1%, MAKE determination after appropriately diluting it 5 ~ 10 times. During dilution, it shall keep the original acidity, AND the blank is diluted same as that of the sample solution.

4.4 Calibration curve drawing

In six 100 ml volumetric flasks, respectively ADD the iron solution (2.5) of the similar amount with that of the test solution; then respectively ADD 0.00, 1.00, 2.00, 3.00, 4.00, 5.00 OR 0.00, 2.00, 4.00, 6.00, 8.00, 10.00 ml of copper standard solution (2.6.2); respectively ADD 10 ml of perchloric acid (2.4); USE water to dilute it to the mark; MIX it uniformly.

At the atomic absorption spectrophotometer at a wavelength of 324.7 nm, USE the air-acetylene flame; USE water to adjust zero; MEASURE its absorbance.

The absorbance of each solution of the calibration curve series minus the absorbance of the zero concentration solution is the net absorbance of the

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