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

GB/T 3884.1-2025

**Methods for chemical analysis of copper concentrates - Part 1:
Determination of copper content - Iodine titration method and
electrogravimetric method**

铜精矿化学分析方法 第1部分:铜含量的测定 碘量法和电解法

(English Translation)

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1 Scope

This document describes methods for determining the copper content in copper concentrates and copper slag concentrates using iodine titration method and electrogravimetric method.

This document applies to the determination of copper content in copper concentrates and copper slag concentrates. The determination range is 12.00% ~ 50.00%.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 3884.2, Methods for chemical analysis of copper concentrates - Part 2.

Determination of gold and silver contents - Flame atomic absorption spectrometric method and fire assay method

GB/T 6682, Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

No terms and definitions need to be defined in this document.

4.1 Principle

Decompose the sample by hydrochloric acid, nitric acid and sulfuric acid; add sodium thiosulfate solution to the dilute sulfuric acid solution to separate copper ions from the solution in the form of copper sulfide. Dissolve the precipitate in a mixed acid; adjust the pH of the solution to 3.0 ~ 4.0; use ammonium fluoride to mask the iron; add potassium iodide to react with divalent copper. The precipitated iodine is titrated with standard sodium thiosulfate solution using starch as an indicator to determine copper content. Correct the copper content in the filtrate using atomic absorption spectrometry.

4.2 Reagents

Unless otherwise stated, use only reagents that are identified as analytical reagents in the analysis.

4.2.1

Water, GB/T 6682, Grade III.

4.2.2

Pure copper sheet ($w_{\text{Cu}} \geq 99.99\%$). Place the pure copper in a gently boiling acetic acid solution (1+4) for 1 minute; remove it and rinse it with water and anhydrous ethanol at least twice; then bake it in an oven at $100\text{ }^{\circ}\text{C} \sim 105\text{ }^{\circ}\text{C}$ for 4 minutes; cool it;

place it in a ground glass bottle for later use.

4.2.3

Potassium iodide.

4.2.4

Hydrochloric acid ($\rho = 1.19\text{ g/mL}$).

4.2.5

Nitric acid ($\rho = 1.42\text{ g/mL}$).

4.2.6

Sulfuric acid ($\rho = 1.84 \text{ g/mL}$).

4.2.7

Nitric acid (1+1).

4.2.8 Mixed acid of nitric acid and sulfur

Slowly add 3 parts of sulfuric acid (4.2.6) along the wall of the cup to 7 parts of nitric acid (4.2.5) while stirring; mix well and cool before use.

4.2.9 Mixed acid

Mix 15 parts of nitric acid (4.2.5) with 5 parts of perchloric acid ($\rho = 1.67 \text{ g/mL}$); slowly add 3 parts of sulfuric acid (4.2.6) along the wall of the beaker while stirring; mix well and cool before use.

4.2.10

Sodium thiosulfate solution (200 g/L, freshly prepared and used immediately).

4.2.11

Saturated ammonium acetate solution.

4.2.12

Saturated solution of ammonium bifluoride (stored in polyethylene bottles).

4.2.13

Potassium thiocyanate solution (100 g/L).

4.2.14

Sodium thiosulfate standard titration solution ($c_{\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}} \sim 0.04 \text{ mol/L}$), shall be recalibrated once a week, and prepared and calibrated in the following manner.

ρ - mass concentration of copper in the filtrate, obtained from the working curve, in micrograms per milliliter ($\mu\text{g/mL}$);

V_4 - total volume of residual copper solution, in milliliters (mL).

Express the calculation result to two decimal places.

4.7.1 Repeatability

For the measured values of the two independent test results that are obtained under repetitive conditions, within the average range given in Table 1, the absolute difference between the two test results does not exceed the repeatability limit (r); the case of exceeding the repeatability limit (r) is not more than 5%. The repeatability limit (r) is obtained using linear interpolation or extrapolation as shown in Table 1.

4.7.2 Reproducibility

The absolute difference between two independent test results obtained under reproducibility conditions shall not exceed the reproducibility limit (R) within the range of average values given in Table 2, and the number of cases exceeding the reproducibility limit (R) shall not exceed 5%. The reproducibility limit (R) shall be obtained by linear interpolation or extrapolation method according to the data in Table

2.

5.1 Principle

After the sample is decomposed by hydrochloric acid, nitric acid, bromine or perchloric acid, use saturated ammonium acetate solution to adjust the pH of the solution to 3.0 ~

4.0; use ammonium fluoride to mask iron; add potassium iodide to react with divalent copper. The precipitated iodine is titrated with standard sodium thiosulfate solution using starch as an indicator to determine the copper content.

5.2 Reagents

Unless otherwise stated, use only reagents that are identified as analytical reagents in the analysis.

5.2.1

Water, GB/T 6682, Grade III.

5.2.2

Pure copper sheet ($w_{Cu} \geq 99.99\%$). Place the pure copper in a gently boiling glacial acetic acid solution (1+4) for 1 minute; remove it, and use water and anhydrous ethanol to rinse it twice respectively; dry it in an oven at 100 °C ~ 105 °C for 4 minutes; cool it; place it in a ground glass bottle for later use.