

ICS 77.120.30



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

GB/T 3884.2-2025

**Methods for chemical analysis of copper concentrates - Part 2:
Determination of gold and silver contents - Flame atomic
absorption spectrometric method and fire assay method**

铜精矿化学分析方法 第2部分：金和银含量的测定 火焰原子吸收光谱法和火试金
法

(English Translation)

Issued on: August 29, 2025

Implemented on: March 1, 2026

Issued by: State Administration for Market Regulation

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1 Scope

This document describes the methods for determining the gold and silver content in copper concentrate and copper slag concentrate, using flame atomic absorption spectrometry and fire assay.

Methods 1 and 2 of this document are applicable to the determination of gold and silver content in copper concentrate and copper slag concentrate. Method 1 has a determination range of silver. 10.0 g/t ~ 500.0 g/t; Method 2 has a determination range of gold. 0.50 g/t ~ 56.00 g/t, silver. 50.0 g/t ~ 2500.0 g/t. Method 3 is applicable to the determination of gold and silver content in copper sulfide concentrate with a copper content of 15% ~ 60%, with a determination range of gold. 0.50 g/t ~ 300.00 g/t, silver.

25.0 g/t ~ 1500.0 g/t.

2 Normative references

The following documents, through normative references in this document, constitute essential provisions of this document. For dated references, only the edition cited applies; for undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385 Laboratory glassware - Burettes

ISO 648 Laboratory glassware - Single-volume pipettes

ISO 1042 Laboratory glassware - One-mark volumetric flasks

ISO 3696 Water for analytical laboratory use - Specification and test methods

ISO 4787 Laboratory glass and plastic ware - Volumetric instruments - Methods for testing of capacity and for use

ISO 9599 Copper, lead, zinc and nickel sulfide concentrates - Determination of hygroscopic moisture content of the analysis sample - Gravimetric method

3 Terms and definitions

This document does not contain any terms or definitions that require clarification.

4.1 Principle

The sample is dissolved in nitric acid, hydrofluoric acid, perchloric acid, hydrochloric acid. In a dilute hydrochloric acid medium, the absorbance of silver is measured using a flame atomic absorption spectrometer, at a wavelength of 328.1 nm with an air- acetylene flame. The silver content is calculated using the standard curve method.

4.2 Reagents

Unless otherwise specified, only reagents confirmed to be of analytical grade and distilled or deionized water or water of equivalent purity shall be used in the analysis.

4.2.1

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

4.2.2

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

4.2.3

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

4.2.4

Perchloric acid ($\rho = 1.67 \text{ g/mL}$).

4.2.5

Silver standard stock solution. Weigh 0.1000 g of silver ($w_{\text{Ag}} \geq 99.99\%$) into a 200 mL beaker; add 20 mL of nitric acid (1 + 1); heat until completely dissolved; cool to room temperature; transfer to a 200 mL amber volumetric flask; dilute to the mark with chloride-free water; mix well. 1 mL of this solution contains 500 μg of silver.

4.2.6 Silver standard solution. Transfer 10.00 mL of the silver standard stock solution

(4.2.5) to a 100 mL volumetric flask; dilute to the mark with hydrochloric acid (1 + 3); mix well. 1 mL of this solution contains 50 μg of silver.

4.3 Instruments

Flame atomic absorption spectrometer with a silver hollow cathode lamp.

Under optimal instrument operating conditions, any instrument meeting the following specifications may be used.

- Characteristic concentration. In a solution consistent with the matrix of the sample solution, the characteristic concentration of silver shall not exceed 0.018 µg/mL;
- Precision. When measuring absorbance 10 times with the highest concentration standard solution, the standard deviation shall not exceed 1.0% of the average absorbance. When measuring absorbance 10 times with the lowest concentration standard solution (not the "zero" standard solution), the standard deviation shall not exceed 0.5% of the average absorbance of the highest concentration standard solution;
- Linearity of the working curve. Dividing the working curve into 5 equal segments according to concentration, the ratio -- of the absorbance difference in the highest segment to the absorbance difference in the lowest segment -- shall not be less than 0.85.

4.4 Samples

4.4.1

The sample particle size shall not exceed 100 µm.

4.4.2

The sample shall be dried at 100 °C ~ 105 °C for 1 hour, then placed in a desiccator and cooled to room temperature.

4.5.1 Sample

Weigh 0.5 g of sample (4.4), accurate to 0.0001 g.

4.5.2 Parallel test

Perform two parallel tests and take the average value.

4.5.3 Blank test

Perform a blank test along with the sample.

4.5.4 Determination

4.5.4.1

Place the sample (4.5.1) in a 250 mL PTFE beaker; add a small amount of water to moisten; add 15 mL of nitric acid (4.2.1); heat on a hot plate at low temperature to dissolve; evaporate to a volume of about 10 mL; remove and let cool slightly. Add 5 mL of hydrofluoric acid (4.2.3); continue heating to dissolve while shaking occasionally;

dissolve to a small volume; remove and let cool slightly. Add 5 mL of perchloric acid (4.2.4); cover with a PTFE watch glass; heat to dissolve until there is no black residue at the bottom of the beaker; remove and let cool slightly. Rinse the watch glass and beaker walls with water; add 5 mL of hydrochloric acid (4.2.2); continue heating until the perchloric acid emits white fumes. Evaporate to near dryness; then remove and cool.

4.5.4.2

Add hydrochloric acid (4.2.2) according to Table 1; rinse the watch glass and beaker walls with water; heat to dissolve the salts; remove and cool to room temperature.

Transfer the solution to a volumetric flask of the appropriate volume (the volume of the volumetric flask is the measurement volume in Table 1); dilute with water to the mark;

mix well; let it stand (or filter dry).

Table 1 -- Constant volume and volume for adding hydrochloric acid

4.5.4.3

Using an air-acetylene flame, at the analytical wavelength of silver (328.1 nm), zero the instrument with water and measure the absorbance of the blank solution and the sample solution. Determine the corresponding silver mass concentration from the working curve.

4.5.5 Plotting the working curve**4.5.5.1**

Pipette 0 mL, 0.20 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of silver standard solution (4.2.6) into a set of 100 mL volumetric flasks (when the mass fraction of silver in the sample is 10.0 g/t ~ 20.0 g/t, add an equal amount of copper and iron matrix solution to the sample solution). Add 10.0 mL of hydrochloric acid (4.2.2); dilute to the mark with water; mix well.

4.5.5.2 Under the same conditions as the sample solution, at the analytical wavelength

of silver (328.1 nm), zero the instrument with water and measure the absorbance of the silver standard solutions. Subtract the absorbance of the "zero" concentration solution from the series of standard solutions. Plot the working curve with the mass concentration of silver as the abscissa and the corresponding absorbance as the ordinate.