GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 77.120.60

H 13

GB/T 3884.2-2012

Replacing GB/T 3884.2-2000

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：金和银量的测定 火焰原子吸收光谱法和火试金法

Issued on: December 31, 2012 **Implemented on: October 1, 2013**

Issued by: General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China;

Standardization Administration of the People's Republic of China.

Table of Contents

Foreword 3

1 Scope 5

2 Method 1 - Flame atomic absorption spectrometric method 5

3 Method 2 - Fire assay method 9

Foreword

GB/T 3884 *Methods for chemical analysis of copper concentrates* is divided into 14 parts:

- *Part 1: Determination of copper content - Iodine titration method*
- *Part 2: Determination of gold and silver contents - Flame atomic absorption spectrometric method and fire assay method*
- *Part 3: Determination of sulfur content - Gravimetric method and combustion-titration method*
- *Part 4: Determination of magnesium oxide content - Flame atomic absorption spectrophotometry method*
- *Part 5: Determination of fluoride content - Ion selective electrode method*
- *Part 6: Determination of lead, zinc, cadmium and nickel content - Flame atomic absorption spectrometry method*
- *Part 7: Determination of lead content - Na_2EDTA titration method*
- *Part 8: Determination of zinc content - Na_2EDTA titration method*
- *Part 9: Determination of arsenic and bismuth contents - Hydride generation-atomic fluorescence spectrometry method - The potassium bromate titration method and the silver diethyldithiocarbamate photometric method*
- *Part 10: Determination of antimony content - Hydride generation atomic fluorescence spectrometry method*
- *Part 11: Determination of mercury content - Cold atomic absorption spectrometric method*
- *Part 12: Determination of fluoride content and chloride content - Ion chromatography*
- *Part 13: Determination of copper - Electrogravimetric method*
- *Part 14: Determination of gold and silver - Fire assay gravimetric and flame atomic absorption spectrometric method*

This Part is Part 2.

This Part was drafted in accordance with the rules given in GB/T 1.1-2009.

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

1 Scope

This method specifies the method for determining gold and silver contents in copper concentrates.

This method applies to the determination of gold and silver contents in copper concentrates. Determination range of method 1: silver: 10.0g/t to 300.0g/t; determination range of method 2: gold: 0.50g/t to 40.00g/t; silver: 50.0g/t to 2,500.0g/t.

2 Method 1 - Flame atomic absorption spectrometric method

2.1 Method summary

DISSOLVE the specimen by nitric acid and perchloric acid. In a dilute hydrochloric acid medium, MEASURE the absorbance of silver by an air-acetylene flame at an atomic absorption spectrometer at a wavelength of 328.1nm. DEDUCT the background absorption. CALCULATE the silver content according to the standard curve.

2.2 Reagents

Unless otherwise stated, only analytically pure reagents and distilled or deionized water or water of comparable purity are used in the analysis.

2.2.1 Ammonium hydrogen fluoride.

2.2.2 Hydrochloric acid (ρ = 1.19g/mL).

2.2.3 Hydrochloric acid (1+9).

2.2.4 Nitric acid (ρ = 1.42g/mL).

2.2.5 Nitric acid (1+1).

2.2.6 Perchloric acid (ρ = 1.67g/mL).

2.2.7 Silver standard storage solution: WEIGH 0.1000g of pure silver (w_{Ag}≥99.99%) in a 200mL beaker, ADD 20mL of nitric acid (2.2.5), HEAT to completely dissolve, COOL to room temperature, and TRANSFER to a 200mL brown volumetric flask. DILUTE with chlorine-free ion exchange water to the scale, and MIX well. 1mL of this solution contains 500μg of silver.

2.2.8 Silver standard solution: PIPETTE 10.00mL of silver standard storage solution (2.2.7) into a 100mL volumetric flask, DILUTE to the scale with hydrochloric acid (1+9), and MIX well. 1mL of this solution contains 20μg of silver.

2.3 Instruments

Atomic absorption spectrometer, with silver hollow cathode lamp.

Under the best operating conditions of the instrument, those that can reach the following indexes may be used.

— Characteristic concentration: The characteristic concentration of silver shall not be greater than 0.018μg/mL in a solution consistent with the matrix of the measurement specimen solution.

— Precision: The absorbance is measured 10 times with the highest concentration standard solution. The standard deviation shall not exceed 1.0% of the average absorbance. The absorbance is measured 10 times with the lowest concentration of standard solubility (not the “zero” standard solubility). The standard deviation shall not exceed 0.5% of the average absorbance of the highest concentration standard solution.

— Linearity of operating curve: The operating curve is divided into five segments according to the concentration. The ratio of the absorbance difference of the highest segment to the absorbance difference of the lowest segment is not less than 0.85.

2.4 Specimen

2.4.1 The sample size shall be no more than 100μm.

2.4.2 The sample shall be dried at 100°C to 105°C for 1h and then placed in a desiccator to cool to room temperature.

2.5 Analytical procedure

3.2.1 Sodium carbonate: industrial pure, powdery.

3.2.2 Lead oxide: industrial pure, powdery (gold content < 0.01g/t, and silver content < 0.5g/t).

3.2.3 Silicon dioxide: industrial pure, powdery.

3.2.4 Borax: powdery.

3.2.5 Starch: powdery.

3.2.6 Potassium nitrate: powdery.

3.2.7 Sodium chloride: industrial pure, powdery.

3.2.8 Pure silver: 99.99%.

3.2.9 Lead foil: 0.1mm to 0.15mm thick, 3g to 5g in mass, square, with a gold content of less than 0.1g/t.

3.2.10 Nitric acid (ρ=1.42g/mL), guaranteed reagent.

3.2.11 Nitric acid (1+7), free of chloride.

3.2.12 Nitric acid (1+1), free of chloride.

3.2.13 Ammonium ferric sulfate indicator: TAKE a saturated solution of ammonium ferric sulfate, ADD three portions of nitric acid (1+3), and MIX well.

3.2.14 Standard titration solution of potassium thiocyanate

3.2.14.1 Preparation: WEIGH 0.5g of potassium thiocyanate. PLACE it in a 100mL beaker. DISSOLVE it in water. PIPETTE into a 1,000mL volumetric flask. DILUTE to the scale, and MIX well. After standing for one week, filter and set aside.

3.2.14.2 Calibration: WEIGH three portions of 10.00mg to 15.00mg of pure silver (3.2.8). PLACE them in a 50mL porcelain crucible (3.3.9), respectively. ADD 10mL to 15mL nitric acid (3.2.12), slightly HEAT to dissolve, and STEAM to about 1mL to 2mL. ADD a small amount of water and 0.5mL of ammonium ferric sulfate indicator (3.2.13). USE the standard titration solution of potassium thiocyanate (3.2.14) to titrate to light red as the endpoint.

CALCULATE the actual concentration of the standard titration solution of potassium thiocyanate according to Formula (2).

$$c = \frac{m_1}{M \cdot V_2} \qquad \cdots\cdots (2)$$

lead buckle content (if the contents of iron, arsenic, antimony, bismuth, nickel, etc. are high, INCREASE the amount appropriately).

Silicon dioxide: The amount added is calculated as the amount required to be equal to the slag of the degree of silicic acid of 0.5.

Potassium nitrate and starch: ADD as appropriate according to the contents of sulfur and carbon in the specimen.

3.5.4.2 Melting

PLACE the prepared clay crucible (3.3.3) in an assay furnace (3.3.2) at 900°C. RISE temperature to 1,100°C within 30min, and INSULATE for 15min before discharging. POUR the melt into a preheated cast iron mold (3.3.10). RETAIN the crucible for remelting. After cooling, SEPARATE the lead buckle from the slag. RETAIN the slag for reprocessing. HAMMER the lead buckle into a cube. The suitable lead buckle shall be bright on the surface and weigh 30g to 45g. Otherwise, the ingredients shall be re-adjusted and melted. REMOVE the covering agent from the slag, and RECOVER it in the original crucible.

3.5.4.3 Cupellation

PLACE the lead buckle in a cupel (3.3.7) preheated in an assay furnace (3.3.2) at 900°C for 30min, and CLOSE the furnace door for 1min to 2min. After the black film on the surface of the lead liquid is removed, slightly OPEN the furnace door to lower the furnace temperature to 840°C as soon as possible for cupellation. The cupellation ends when the particles appear to flash. MOVE the cupel to the furnace door, and PLACE it in a cupel tray after slight cooling. RETAIN the cupel residue for disposal.

3.5.4.4 Parting

USE the hemostatic forceps (3.3.11) to remove the gold and silver particles. BRUSH the adhered impurities. HAMMER into thin slices. PLACE in a 30mL porcelain crucible (3.3.8). ADD 10mL of hot nitric acid (3.2.11). REMAIN near boiling on a low-temperature heating plate. STEAM to about 2mL. REMOVE for slight cooling. ADD another 10mL of hot nitric acid (3.2.12). STEAM to about 2mL. REMOVE for cooling. USE hot water to wash the crucible wall. PIPETTE the solution into a 50mL porcelain crucible (3.3.9) by pouring. USE hot water to wash the crucible wall twice. After cooling, ADD about 0.5mL of ammonium ferric sulfate indicator (3.2.13). USE the standard titration solution of potassium thiocyanate (3.2.14) to titrate to light red as the endpoint. PLACE the porcelain crucible containing gold particles on a high-temperature electric furnace to burn for 5min. WEIGH after removal and cooling.

NOTE: When the ratio of silver to gold in the particles is less than 3:1, pure silver shall be added to the

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 3 websites:

1. https://www.ChineseStandard.us

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. https://www.ChineseStandard.net

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - https://www.ChineseStandard.us).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

3. https://www.google.com/search?tbm=bks&q=ChineseStandard.net

- SEARCH the standard ID, such as GB 4943.1-2022.
- Google Books -- Select your currency.
- Processed by Google (delivery, tax invoice etc.). Delivered in 9 seconds by Google.
- Tips: Download an unprotected True-PDF (text-editable) from Google-Books:
  1. https://play.google.com/books → 2. Sign in → Google account
  3. Find the BOOK you bought → 4. Click "3-dots" → Export
  5. Save as "*.pdf" (Save True-PDF to your local computer for offline reading/printing)

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

Accountable person and shareholder: Wayne Zheng

About Us (Goodwill, Policies, Fair Trading…): https://www.chinesestandard.net/AboutUs.aspx

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: https://www.linkedin.com/in/waynezhengwenrui/

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