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**Copper sulfide concentrates —
Determination of copper content —
Electrogravimetric method**

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*Concentrés de sulfure de cuivre — Dosage du cuivre — Méthode
électrogravimétrique*

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10469 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes A and B form an integral part of this International Standard. Annex C is for information only.

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Copper sulfide concentrates — Determination of copper content — Electrogravimetric method

1 Scope

This International Standard specifies an electrogravimetric method for the determination of the copper content of copper sulfide concentrates in the range 15 % (m/m) to 50 % (m/m).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*.

ISO 9599:1991, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*.

ISO Guide 35:1985, *Certification of reference materials — General and statistical principles*.

3 Principle

The test portion is decomposed in nitric and sulfuric acids, and copper is separated from interfering elements:

— from silver by precipitation of silver chloride;

— from arsenic, antimony, selenium and tin by fuming with hydrobromic acid;

— from iron by precipitation of copper sulfide with sodium thiosulfate or by precipitation of iron(III) oxide hydrate (bismuth and tellurium are also separated in this way).

Electrogravimetric deposition of copper occurs in the presence of nitric acid, sulfuric acid and traces of chloride. Under these conditions, coprecipitation of molybdenum does not occur.

Traces of copper in the electrolyte, the filtrate of the copper sulfide precipitation, all precipitates and residues are determined by flame atomic absorption spectrometry (FAAS).

Copper deposits are dissolved and analysed for coprecipitated elements, mainly silver, by flame atomic absorption spectrometry or Plasma-OES.

NOTE 1 The normal mercury content of copper concentrates does not usually affect the copper result. At a level of 0,005 % (m/m), or greater, the mercury content of the copper deposit should be checked. This procedure is not described in this International Standard.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

4.1 Nitric acid, concentrated (ρ_{20} 1,42 g/ml).

4.2 Nitric acid, diluted 1 + 1.

Slowly add 500 ml of concentrated nitric acid (4.1) to 500 ml of water, while stirring.

4.3 Sulfuric acid, concentrated (ρ_{20} 1,84g/ml).

4.4 Sulfuric acid, diluted 1 + 1.

Slowly add 500 ml of concentrated sulfuric acid (4.3) to 500 ml of water, while stirring and cooling.

4.5 Sulfuric acid, diluted 1 + 4.

Slowly add 200 ml of concentrated sulfuric acid (4.3) to 800 ml of water, while stirring and cooling.

4.6 Sodium thiosulfate pentahydrate, 450 g/l solution.

4.7 Nitration mixture.

Slowly add 250 ml of concentrated sulfuric acid (4.3) to 250 ml of concentrated nitric acid (4.1).

4.8 Sodium chloride, 10 g/l solution.

4.9 Sodium chloride, 0,5 g/l solution.

4.10 2-Propanol.

4.11 Ethanol, minimum purity 95 % (V/V).

4.12 Methanol, minimum purity 95 % (V/V).

4.13 Ammonium iron(III) sulfate, solution.

Add 50 ml of dilute sulfuric acid (4.4) and 43 g of ammonium iron(III) sulfate dodecahydrate $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ to 950 ml of water.

4.14 Iron(III) nitrate, solution.

Add 30 g of iron(III) nitrate nonahydrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ to 1 000 ml of water.

4.15 Ammonia, solution (ρ_{20} 0,91 g/ml).

4.16 Ammonia, solution diluted 1 + 99.

4.17 Hydrobromic acid, (ρ_{20} 1,50 g/ml).

4.18 Perchloric acid, (ρ_{20} 1,53 g/ml).

4.19 Hydrofluoric acid, (ρ_{20} 1,14 g/ml).

4.20 Copper metal, minimum purity 99,999 %.

4.21 Silver metal, minimum purity 99,99 %.

4.22 Sodium sulfate (Na_2SO_4), anhydrous.

4.23 Hydrochloric acid, concentrated (ρ_{20} 1,16 g/ml to 1,18 g/ml).

4.24 Hydrochloric acid, diluted 1 + 1.

Slowly add 500 ml of concentrated hydrochloric acid (4.23) to 500 ml of water, while stirring.

4.25 Arsenic oxide (As_2O_3), minimum purity 99,5 %.

4.26 Antimony oxide (Sb_2O_3), minimum purity 99,5 %.

4.27 Bismuth metal, minimum purity 99,99 %.

4.28 Selenium, minimum purity 99,99 % or **selenium dioxide** (SeO_2), minimum purity 98 %.

4.29 Tellurium, minimum purity 99,99 %.

4.30 Ammonium molybdate tetrahydrate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$.

4.31 Lead metal, minimum purity 99,99 % or **lead oxide** (PbO), minimum purity 99 %.

4.32 Mercury metal, minimum purity 99,99 % or **mercury(II) oxide** (HgO), minimum purity 99 %.

4.33 Standard solutions.

NOTE 2 Standard solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.33.1 Copper, standard solution (1 ml \triangleq 0,1 mg of Cu).

Dissolve 0,100 0 g of copper metal (4.20) in 10 ml of warm dilute nitric acid (4.2) and heat to evaporate to approximately 5 ml in order to remove nitrogen oxides.

Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.2 Copper, standard solution
(1 ml $\hat{=}$ 60 mg of Cu).

Dissolve 60 g of copper metal (4.20) in 500 ml of warm dilute nitric acid (4.2).

Transfer to a 2 litre beaker and heat to evaporate to approximately 250 ml in order to remove nitrogen oxides. Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.3 Silver, standard stock solution
(1 ml $\hat{=}$ 1 mg of Ag).

Use only chloride-free reagents and water.

Dissolve 1,000 g of silver metal (4.21) in 20 ml of dilute nitric acid (4.2) and heat to evaporate nitrogen oxides. Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again. Store in a brown bottle in a darkened room.

4.33.4 Silver, standard solution
(1 ml $\hat{=}$ 40 μ g of Ag).

Transfer 10,00 ml of silver standard stock solution (4.33.3) to a 250 ml volumetric flask. Fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again. Prepare this solution on the day of use.

4.33.5 Arsenic, standard solution
(1 ml $\hat{=}$ 1 mg of As).

Dissolve 1,320 g of arsenic oxide (4.25) in 100 ml of water and 20 ml of dilute nitric acid (4.2). Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.6 Antimony, standard solution
(1 ml $\hat{=}$ 1 mg of Sb).

Dissolve 1,197 g of antimony oxide (4.26) in 500 ml of dilute hydrochloric acid (4.24). Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.7 Bismuth, standard solution
(1 ml $\hat{=}$ 1 mg of Bi).

Dissolve 1,000 g of bismuth metal (4.27) in 100 ml of dilute nitric acid (4.2). Heat to evaporate nitrogen oxides. Transfer to a 1 000 ml volumetric flask, fill up

nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.8 Selenium, standard solution
(1 ml $\hat{=}$ 1 mg of Se).

Dissolve 1,000 g of selenium or 1,405 g of selenium dioxide (4.28) in 100 ml of dilute nitric acid (4.2). Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.9 Tellurium, standard solution
(1 ml $\hat{=}$ 1 mg of Te).

Dissolve 1,000 g of tellurium (4.29) in 20 ml of dilute nitric acid (4.2). Dilute with water to 50 ml, dissolve the precipitate with concentrated hydrochloric acid (4.23) and heat to evaporate nitrogen oxides. Transfer to a 1 000 ml volumetric flask, add 20 ml of concentrated hydrochloric acid (4.23) and fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.10 Molybdenum, standard solution
(1 ml $\hat{=}$ 1 mg of Mo).

Dissolve 1,840 g of ammonium molybdate tetrahydrate (4.30) in water. Transfer to a 1 000 ml volumetric flask, add 20 ml of dilute nitric acid (4.2), fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.11 Lead, standard solution
(1 ml $\hat{=}$ 1 mg of Pb).

Dissolve 1,000 g of lead metal or 1,077 g of lead oxide (4.31) in 20 ml of dilute nitric acid (4.2). Heat to evaporate nitrogen oxides (when using lead metal). Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.33.12 Mercury, standard solution
(1 ml $\hat{=}$ 1 mg of Hg).

Dissolve 1,000 g of mercury metal or 1,080 g of mercury(II) oxide (4.32) in 20 ml of dilute nitric acid (4.2). Heat to evaporate nitrogen oxides (when using mercury metal). Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

4.34 Calibration solutions.

NOTE 3 Calibration solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.34.1 Calibration solutions A.

Pipette 0,0 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml of copper standard solution (4.33.1) into a series of 500 ml one-mark volumetric flasks.

Add 40 ml of dilute sulfuric acid (4.4) and 13 g of sodium sulfate (4.22) to each flask. Dilute with water, stir to dissolve the salts, fill up nearly to the mark, mix and equilibrate at room temperature, then fill up exactly to the mark and mix again.

These solutions contain 0 mg of Cu, 1 mg of Cu, 2 mg of Cu, 3 mg of Cu and 4 mg of Cu in a 500 ml volume.

In situations where the test solution contains more than 4 mg of Cu, dilute with the calibration solution containing 0,0 µg of Cu/ml until the copper concentration in the test solution is below 4 mg/500 ml.

4.34.2 Calibration solutions B.

Pipette 0,0 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml of copper standard solution (4.33.1) into a series of 500 ml one-mark volumetric flasks.

Add 40 ml of dilute sulfuric acid (4.4) and 20 ml of dilute nitric acid (4.2) to each flask. Fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

These solutions contain 0 mg of Cu, 1 mg of Cu, 2 mg of Cu, 3 mg of Cu and 4 mg of Cu in a 500 ml volume.

In situations where the test solution contains more than 4 mg of Cu, dilute with the calibration solution containing 0,0 µg of Cu/ml until the copper concentration in the test solution is below 4 mg/500 ml.

4.34.3 Calibration solutions C.

Pipette 0,0 ml, 10,00 ml, 20,00 ml, 30,00 ml and 40,00 ml of copper standard solution (4.33.1) into a series of 500 ml one-mark volumetric flasks.

Add 20 ml of dilute sulfuric acid (4.4), 10 ml of dilute nitric acid (4.2) and 50 ml (see note 4) of ammonium iron(III) sulfate solution (4.13) (corresponding to approximately 250 mg of Fe to each flask). Fill up nearly to the mark with water, mix and equilibrate at room

temperature; then fill up exactly to the mark and mix again.

These solutions contain 0 mg of Cu, 1 mg of Cu, 2 mg of Cu, 3 mg of Cu and 4 mg of Cu in a 500 ml volume.

NOTE 4 If the test portion contains less than 50 mg of Fe [i.e. < 2,5 % (m/m) of Fe], only 10 ml of ammonium iron(III) sulfate solution (4.13) should be added each time.

In situations where the test solution contains more than 4 mg of Cu, dilute with the calibration solution containing 0,0 µg of Cu/ml until the copper concentration in the test solution is below 4 mg/500 ml.

4.34.4 Calibration solutions D.

Pipette 0,0 ml, 5,00 ml, 10,00 ml, and 25,00 ml of silver standard solution (4.33.4) into four of 50 ml one-mark volumetric flasks.

Add 10 ml of dilute nitric acid (4.2) and a volume of copper standard solution (4.33.2) depending on the presumed copper content of the concentrate [1 ml \triangleq 3 % (m/m) of Cu] to each flask. Fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

These solutions correspond to silver contents of 0,00 % (m/m), 0,01 % (m/m), 0,02 % (m/m) and 0,05 % (m/m) in the copper concentrate.

4.34.5 Calibration solutions for impurities.

Pipette 10 ml of dilute nitric acid (4.2) and 0,0 ml, 0,2 ml, 0,4 ml, 1,0 ml, and 2,0 ml of each of the following standard solutions:

- arsenic standard solution (4.33.5);
- antimony standard solution (4.33.6);
- bismuth standard solution (4.33.7);
- selenium standard solution (4.33.8);
- tellurium standard solution (4.33.9);
- molybdenum standard solution (4.33.10);
- lead standard solution (4.33.11);
- mercury standard solution (4.33.12).

into a series of 50 ml one-mark volumetric flasks.

Add a volume of copper standard solution (4.33.2) depending on the presumed copper content of the

concentrate [1 ml \triangleq 3 % (m/m) of Cu]. Fill up nearly to the mark with water, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

These solutions correspond to trace element contents of 0,00 % (m/m), 0,01 % (m/m), 0,02 % (m/m), 0,05 % (m/m) and 0,10 % (m/m) in the copper concentrate.

NOTE 5 These calibration solutions should be prepared freshly just before use.

5 Apparatus

5.1 Usual laboratory equipment, including fume hoods, hot plates, drying oven, analytical balance and water bath with thermostat for the temperature control of solutions.

5.2 Ordinary laboratory glassware.

5.3 Volumetric glassware, class A, complying with ISO 385-1, ISO 648 and ISO 1042, and used in accordance with ISO 4787.

5.4 Desiccator.

5.5 Filter papers, of different porosity (dense and medium).

5.6 Equipment for static electrolysis.

5.7 Platinum electrodes, net electrodes as cathodes, spiral electrodes as anodes.

NOTE 6 Winkler electrodes have been found suitable.

5.8 Atomic absorption spectrometer (AAS).

Instrument conditions:

Flame: air/acetylene; for molybdenum, use a dinitrogen monoxide/acetylene flame

Wavelengths:

324,7 nm for copper

328,1 nm for silver

see 7.9.2 for other elements

5.9 Inductively coupled plasma (ICP) atomic emission spectrometer (optional).

5.10 Platinum dish.

5.11 Polytetrafluoroethylene (PTFE) dish.

6 Sample

6.1 Test sample

Prepare an air-equilibrated test sample in accordance with ISO 9599.

NOTE 7 A test sample is not required if predried test portions are to be used (see annex A).

6.2 Test portion

Taking multiple increments, extract approximately 2 g from the test sample and weigh to the nearest 0,1 mg (*m*). At the same time as test portions are being weighed for analysis, weigh test portions for the determination of hygroscopic moisture in accordance with ISO 9599.

Alternatively, the method specified in annex A may be used to prepare predried test portions directly from the laboratory sample.

7 Procedure

7.1 Number of determinations

Carry out the determinations at least in duplicate, as far as possible under repeatability conditions, on each test sample.

NOTE 8 Repeatability conditions exist where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment, within short intervals of time.

7.2 Blank test

Carry out a blank test parallel with the analysis, using the same quantities of all reagents but omitting the test portion. The purpose of the blank test in this method is to check the quality of reagents. If a significant value is obtained as a result of the blank test, check all reagents and rectify the problem.

7.3 Dissolution of test portion

Transfer the test portion into a 400 ml or 500 ml conical or a tall-form beaker or a 500 ml Erlenmeyer flask. Moisten with 10 ml of water. Add 20 ml of dilute nitric acid (4.2), cover the beaker with a watch glass or, if using an Erlenmeyer flask, with a drip catcher and heat for about 10 min at 60 °C to 70 °C.

Add 40 ml of dilute sulfuric acid (4.4) and heat until strong white fumes are evolved.

7.4 Separation of arsenic, antimony, tin, selenium and silver

Add 20 ml of water and 20 ml of hydrobromic acid (4.17) and heat until strong white fumes are evolved.

WARNING — Extreme caution must be taken during this step.

NOTE 9 This step may be omitted if the test sample contains less than 0,01 % (*m/m*) each of As, Sb, Sn and Se.

If the residue appears dark (presence of carbon), slowly add a small amount of the nitration mixture (4.7) to the hot solution until the solution becomes colourless or bluish. Add 20 ml of water and heat again until strong white fumes are evolved. Allow to cool. Add 100 ml of water, warm to dissolve soluble salts and precipitate silver completely as silver chloride by adding approximately 1 ml of sodium chloride solution (4.8).

NOTE 10 This step may be omitted if the test sample contains less than 0,01 % (*m/m*) of Ag.

Heat until boiling, then allow to cool.

Filter through a dense filter paper (5.5), wash with cold water and collect the filtrate in a 400 ml or 500 ml conical or tall-form beaker, or a 500 ml Erlenmeyer flask (only for sulfide separation). Reserve the filter paper and residue for the determination of copper by FAAS (as described in 7.7.2), unless it has been proven by previous testing that the copper in the sample is completely soluble using the initial solution (7.3).

7.5 Copper separation

Separate copper from interfering ions in accordance with 7.5.1 or 7.5.2.

7.5.1 Sulfide separation

Dilute to 200 ml with water and heat until boiling. Add 50 ml of sodium thiosulfate solution (4.6) (copper sulfide is thus precipitated).

Continue boiling for 10 min, then filter off the precipitate using a filter paper of medium porosity (5.5). Collect the filtrate in a 500 ml volumetric flask, and immediately wash the filter and precipitate with cold

water. Reserve the filtrate for the determination of copper by FAAS (as described in 7.7.1).

Put the filter and copper sulfide precipitate back into the vessel used for precipitation, cover with a watch glass or drip catcher, add 30 ml of nitric acid (4.1) and 10 ml of sulfuric acid (4.3) and heat until strong white fumes are evolved.

If the residue appears dark (presence of carbon), slowly add a small amount of the nitration mixture (4.7) until the residue becomes colourless or bluish. Allow to cool. Add 20 ml of water and heat again until strong white fumes are evolved.

Dilute with water to approximately 100 ml, filter into a 400 ml tall-form beaker and wash the filter and vessel with water. Reserve the filter for the determination of copper by FAAS (as described in 7.7.2).

NOTE 11 The following step may be omitted if the test sample contains less than 0,01 % (*m/m*) of Bi and/or Te.

Add 3 ml of iron(III) nitrate solution (4.14) and sufficient ammonia solution (4.15) to make the solution alkaline. Boil for a short time, filter the precipitate using a filter paper of medium porosity (5.5) and wash out with dilute ammonia solution (4.16). Catch the filtrate in a 400 ml tall-form beaker. Dissolve the precipitate in a paper filter using a little hot dilute sulfuric acid (4.5), wash with hot water and repeat the precipitation with ammonia solution (4.15). Filter the precipitate again, using the same filter and collect the filtrate in the 400 ml tall-form beaker already used.

Add dilute sulfuric acid (4.4) to make the solution slightly acidic. Reserve the filter and precipitate for the determination of copper by FAAS (as described in 7.7.2).

Add 20 ml of dilute nitric acid (4.2), 10 ml of sodium chloride solution (4.9) and dilute with water to approximately 300 ml. Use this solution for the electrolytic deposition of copper (as described in 7.6).

7.5.2 Hydroxide separation

Evaporate the filtrate retained in 7.4 until fuming of sulfuric acid begins to occur.

Add approximately 150 ml of water and dissolve the residue completely. Slowly add ammonia solution (4.15), while stirring, until the basic copper salt precipitated at first is dissolved again. Then add a further 30 ml of ammonia solution (4.15) in excess. Heat to the onset of boiling, filter using a filter paper of medium porosity (5.5), wash with warm diluted ammonia solution (4.16) and collect the filtrate and washing solutions in a 600 ml beaker.

If the test portion contains less than 50 mg of Fe [i.e. < 2,5 % (*m/m*) of Fe], add 10 ml of ammonium iron(III) sulfate solution (4.13) (equivalent to approximately 50 mg of iron) before the precipitation of iron(III) hydroxide.

Rinse the precipitate back into the original beaker with water and dissolve any precipitate adhering to the filter paper using 15 ml of warm dilute sulfuric acid (4.5) in small amounts. Wash the filter paper with warm water, collecting the washings in the original beaker. Dissolve the precipitate completely by adding 10 ml of dilute sulfuric acid (4.4), warm gently and dilute with water to obtain a volume of approximately 100 ml.

Add ammonia solution (4.15), while stirring, until a slight precipitate of iron(III) hydroxide appears, then add a further 30 ml of ammonia solution (4.15) in excess.

Heat to the onset of boiling, filter using the same filter paper again, wash with warm dilute ammonia solution (4.16) and collect the filtrate and washings in the 600 ml beaker already used. Reserve the precipitate and beaker for the determination of copper by FAAS (as described in 7.8).

Heat to evaporate the filtrate to a volume of approximately 250 ml.

NOTE 12 The dark blue colour of the complex ion $[(\text{Cu}(\text{NH}_3)_4)]^{2+}$ should disappear.

Add 20 ml of dilute sulfuric acid (4.4), 20 ml of dilute nitric acid (4.2) and 10 ml of sodium chloride solution (4.9). Use this solution for the electrolytic deposition of copper (as described in 7.6).

7.6 Electrolytic deposition

Before starting, prepare the cathode in the same way as for finishing the copper deposition (see below). Weigh the cathode to the nearest 0,1 mg (m_1).

Carry out the electrolytic deposition of copper without stirring and without heating, preferably overnight, using platinum electrodes (5.7).

NOTES

13 A "Winkler" net as the cathode and a spiral as the anode, touching the bottom of the electrolysis beaker, cover glasses (two halves of a watch glass), a current of approximately 0,3 A to 0,5 A (current density is approximately 5 mA/cm² to 10 mA/cm²) and a voltage of between 2,5 V and 4 V are considered suitable.

14 The addition of sulfamic acid ($\text{NH}_2 - \text{SO}_3\text{H}$) is not necessary. The use of considerably greater currents, heating and stirring is not recommended.

When working during the day continue with the electrolytic deposition until the electrolyte is colourless, then raise the current to approximately 1 A for at least one more hour.

After finishing the copper deposition (if working overnight, next morning), wash the glass covers and wall of the beaker.

Remove the cathode, washing first with water then with 2-propanol (4.10) or ethanol (4.11) or methanol (4.12) and dry in a drying oven at approximately 80 °C for 5 min to 10 min. Allow to cool in the desiccator (5.4) and weigh the cathode to the nearest 0,1 mg (m_2).

Reserve the electrolyte for the determination of copper by FAAS (as described in 7.7.2 or 7.8, as appropriate).

NOTES

15 The surface of the copper deposit should appear bright. If there is a dark colour, the determination should be repeated, including the separation steps for the coprecipitated elements. Considerable amounts of selenium and tellurium can be detected as a dark layer between the copper and platinum surfaces, after dissolving the copper with cold dilute nitric acid (4.2).

16 For the determination of coprecipitated elements, such as silver and mercury, refer to 7.9.

7.7 Atomic absorption determination — Sulfide separation method

7.7.1 FAAS determination of copper in the filtrate of the sulfide precipitation

Dilute the filtrate (reserved in 7.5.1) in the 500 ml volumetric flask with water nearly to the mark, mix and equilibrate at room temperature; then fill up exactly to the mark and mix again.

Aspirate the test solution and the calibration solutions A (4.34.1) into the atomic absorption spectrometer (5.8) using an air/acetylene flame and a wavelength of 324,7 nm. Note the absorbances. For exact determination, use the test solution and the two nearest calibration solutions (one less concentrated than and the other more concentrated than the test solution). Repeat these measurements twice.

Calculate the mass of copper (m_3) in the test solution.