



SLOVENSKI STANDARD
SIST ISO 10469:1998

01-november-1998

6 U_fcj]'gi `Z[Xb]`_cbWbhfUj]!`8c`c Yj Ub^YVU_fU!`9`Y`frc[fUj]a Yff]^g_Ua YrcXU

Copper sulfide concentrates -- Determination of copper content -- Electrogravimetric method

Concentrés de sulfure de cuivre -- Dosage du cuivre -- Méthode électrogravimétrique

STANDARD PREVIEW
(standards.iteh.ai)

Ta slovenski standard je istoveten z: ISO 10469:1994

<https://standards.iteh.ai/catalog/standards/sist/e3cd3295-f8b5-4df5-9a62-a0b28d8c77f6/sist-iso-10469-1998>

ICS:

73.060.99 Druge rude Other metalliferous minerals

SIST ISO 10469:1998

en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 10469:1998

<https://standards.iteh.ai/catalog/standards/sist/e3cd3295-f8b5-4df5-9a62-a0b28d8c77f6/sist-iso-10469-1998>

INTERNATIONAL
STANDARD

ISO
10469

First edition
1994-06-15

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

iTeh STANDARD PREVIEW

(standards.iTeh.ai)
*Concentrés de sulfure de cuivre — Dosage du cuivre — Méthode
électrogravimétrique*

SIST ISO 10469:1998

<https://standards.iTeh.ai/catalog/standards/sist/e3cd3295-f8b5-4df5-9a62-a0b28d8c77f6/sist-iso-10469-1998>



Reference number
ISO 10469:1994(E)

Contents

	Page
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	1
5 Apparatus	5
6 Sample	5
7 Procedure	5
8 Expression of results	9
9 Precision	10
10 Test report	11

Annexes

A Procedure for the preparation and determination of the mass of a predried test portion	12
B Flowsheet of the procedure for the acceptance of analytical values for test samples	14
C Derivation of precision equations	15

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.

<https://standards.iteh.ai/catalog/standards/sist/c5cd5299-f8b5-4df5-9a62-a0b28d8c77f6/sist-iso-10469-1998>

iTeh STANDARD PREVIEW

This page intentionally left blank
(standards.iteh.ai)

SIST ISO 10469:1998

<https://standards.iteh.ai/catalog/standards/sist/e3cd3295-f8b5-4df5-9a62-a0b28d8c77f6/sist-iso-10469-1998>

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 $\hat{=}$ 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.