



SLOVENSKI STANDARD
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Copper sulfide concentrates -- Determination of gold and silver contents -- Fire assay gravimetric and flame atomic absorption spectrometric method

iTeh STANDARD PREVIEW

Concentrés de sulfure de cuivre -- Dosage de l'or et de l'argent -- Méthode gravimétrique par essai au feu et spectrométrie d'absorption atomique dans la flamme

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INTERNATIONAL
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**Copper sulfide concentrates —
Determination of gold and silver
contents — Fire assay gravimetric and
atomic absorption spectrometric method**
(standards.iteh.ai)

*Concentrés de sulfure de cuivre — Dosage de l'or et de l'argent —
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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 10378 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes A, B, C, D and E form an integral part of this International Standard. Annexes F, G, H, J and K are for information only.

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Copper sulfide concentrates — Determination of gold and silver contents — Fire assay gravimetric and atomic absorption spectrometric method

1 Scope

This International Standard specifies a fire assay gravimetric and atomic absorption spectrometric method for the determination of gold and silver contents in copper sulfide concentrates containing 15 % (m/m) to 60 % (m/m) copper, mainly in the form of chalcopyrite and related sulfide minerals.

The method is applicable to gold contents from 0,5 g/t to 300 g/t and silver contents from 25 g/t to 1 500 g/t.

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

Fire assaying for the determination of gold and silver comprises a series of steps to separate firstly the precious metals from most of the associated metals, followed by separation of the gold from silver and other metals preconcentrated into a precious metal alloy.

The stages which comprise the determinations are described in 3.1 to 3.5 inclusive.

3.1 Fusion

Crucible fusion of the samples mixed with a litharge-based flux which, under reducing conditions, collects the precious metals in a metallic lead button.

3.2 Cupellation

Oxidizing fusion in which the base metals present in the lead button are substantially separated from the precious metals. Cupellation produces a bead largely comprising a silver-gold alloy with small quantities of other metals.

3.3 Parting

Separation of gold from silver-gold alloy or doré bead by treatment with nitric acid. The gold prills are weighed. Gold prills with a mass less than 0,05 mg are dissolved in aqua regia and the gold determined

by flame atomic absorption spectrometry (FAAS). Silver is determined in the parting solution by FAAS.

3.4 Retreatment

Retreatment of all residues to maximize the recovery of gold and silver. The addition of collectors for either gold or silver is not required, as both metals are present in sufficient amounts to be readily visible after the cupellation stage. The residue beads are dissolved in acids followed by analysis of both metals by FAAS.

3.5 Correction for blank contamination

Contamination by gold and silver impurities in the reagents is corrected for by fusing the reagents without the test portion.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled or deionized chloride-free water.

4.1 Sodium carbonate, anhydrous.

4.2 Litharge (PbO), assay grade with a gold content of less than 0,01 g/t and a silver content of less than 0,2 g/t.

4.3 Silica, precipitated.

4.4 Potassium nitrate or sodium nitrate.

NOTE 1 If sodium nitrate is used, the masses specified for potassium nitrate will have to be modified:

$$\text{g of KNO}_3 \times \frac{85,0}{101,1} = \text{g of NaNO}_3$$

4.5 Flour.

4.6 Borax, fused anhydrous sodium tetraborate (borax glass powder).

4.7 Nitric acid, concentrated (ρ_{20} 1,42 g/ml), free from chlorides.

4.8 Nitric acid, diluted 17 + 83.

Slowly add 170 ml of concentrated nitric acid (4.7) to 830 ml of water, while stirring.

4.9 Nitric acid, diluted 1 + 1.

Slowly add 500 ml of concentrated nitric acid (4.7) to 500 ml of water, while stirring.

4.10 Lead, foil, with a gold content of less than 0,01 g/t and a silver content of less than 0,2 g/t.

4.11 Silver, minimum purity 99,99 %.

4.12 Platinum, minimum purity 99,99 %.

4.13 Palladium, minimum purity 99,99 %.

4.14 Gold, minimum purity 99,99 %.

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

4.16 Mercury(II) nitrate, solution containing 0,2 % (*m/m*) of Hg²⁺.

Add 3,415 g of mercury(II) nitrate monohydrate [Hg(NO₃)₂·H₂O] to 1 000 ml of water.

WARNING — Care must be taken when using mercury(II) nitrate solution because of the toxicity of this reagent.

4.17 Thiourea, 10 g/l solution.

Add 1 g of thiourea to 100 ml of water.

4.18 Aqua regia.

Carefully add 150 ml of hydrochloric acid (4.15) to 50 ml of concentrated nitric acid (4.7). Prepare freshly when required.

4.19 Blank solution.

Carefully add 500 ml of hydrochloric acid (4.15) and 100 ml of concentrated nitric acid (4.7) to 300 ml of water. Dilute to 1 000 ml and mix.

4.20 Standard solutions.

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

4.20.1 Silver, standard stock solution A
(1 ml $\hat{=}$ 500 μ g of Ag).

Weigh 0,500 0 g of silver metal to the nearest 0,1 mg. Transfer to a 100 ml beaker, add 10 ml of concentrated nitric acid (4.7) and warm to dissolve. Cool and add 20 ml of concentrated nitric acid (4.7). Transfer to a 1 000 ml volumetric flask, fill up with water nearly to the mark, mix and cool to room temperature; then fill up exactly to the mark and mix again.

4.20.2 Silver, standard solution B (1 ml $\hat{=}$ 50 μ g of Ag).

Transfer 10,00 ml of silver standard stock solution A (4.20.1) to a 100 ml volumetric flask, fill up with water nearly to the mark, mix and cool to room temperature; then fill up exactly to the mark and mix again.

Prepare a fresh solution per batch.

4.20.3 Gold, standard solution (1 ml $\hat{=}$ 1 000 μ g of Au).

Weigh 1 000 g of gold metal to the nearest 0,1 mg. Transfer to a 150 ml beaker, add 25 ml of aqua regia solution (4.18) and cover with a watch glass. Warm to dissolve and heat almost to dryness. If necessary, add 25 ml of aqua regia solution (4.18) and repeat the digestion almost to dryness.

NOTE 3 The solution should not be allowed to evaporate to dryness; otherwise, metallic gold will precipitate.

Add 25 ml of hydrochloric acid (4.15) and 25 ml of water, and warm to dissolve the salts. Cool and transfer to a 1 000 ml volumetric flask. Add 75 ml of hydrochloric acid (4.15), fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

4.20.4 Gold and silver, standard solution (1 ml $\hat{=}$ 100 μ g of Au and 50 μ g of Ag).

Pipette 10,00 ml of silver standard stock solution A (4.20.1) into a 100 ml volumetric flask. Add 25 ml of hydrochloric acid (4.15). Pipette 10,00 ml of gold standard solution (4.20.3) into the volumetric flask. Fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

4.21 Calibration solutions.

NOTE 4 Calibration solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.21.1 Calibration solutions A (to be freshly prepared) **for the determination of gold and silver in primary and residual beads and of silver in parted prills.**

Pipette 0,0 ml, 1,00 ml, 2,00 ml, 5,00 ml and 10,00 ml of gold and silver standard solution (4.20.4) into a series of 100 ml one-mark volumetric flasks.

Add 25 ml of hydrochloric acid (4.15) to each flask, fill up nearly to the mark with water, mix and cool to

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

These solutions contain 0,0 μ g of Au/ml, 1,00 μ g of Au/ml, 2,00 μ g of Au/ml, 5,00 μ g of Au/ml and 10,00 μ g of Au/ml and 0,0 μ g of Ag/ml, 0,50 μ g of Ag/ml, 1,00 μ g of Ag/ml, 2,50 μ g of Ag/ml and 5,00 μ g of Ag/ml.

4.21.2 Calibration solutions B (to be freshly prepared) **for the determination of silver in parting solutions.**

Pipette 0,0 ml, 1,00 ml, 2,00 ml, 4,00 ml, 6,00 ml, 8,00 ml and 10,00 ml of silver standard solution B (4.20.2) into a series of 100 ml volumetric flasks. Add 10 ml of nitric acid (4.8), fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

These solutions contain 0,0 μ g of Ag/ml, 0,50 μ g of Ag/ml, 1,00 μ g of Ag/ml, 2,00 μ g of Ag/ml, 3,00 μ g of Ag/ml, 4,00 μ g of Ag/ml and 5,00 μ g of Ag/ml.

5 Apparatus

5.1 Assay crucible furnace, with a maximum required operating temperature of 1 200 °C.

5.2 Muffle furnace, with a maximum required operating temperature of 1 100 °C. Temperature indication, automatic temperature control and controlled air flow are preferable.

5.3 Crucibles, made of fireclay, of nominal capacity 500 ml to 600 ml, capable of withstanding corrosion by the samples and fluxes at 1 100 °C. The crucible shall be of such size that the charge for the retreatment of residues (7.7) does not fill more than 3/4 of the depth of the crucible.

5.4 Cupels, made of magnesium oxide, of nominal capacity 50 g of molten lead. The inside bottom of the cupels shall be concave, as recommended in the fire assay texts referred to in annex K.

NOTE 5 Bone ash cupels give losses which are greater than those obtained with magnesium oxide cupels by a factor of 5 to 10. Bone ash cupels are not recommended, but may be used as long as the losses are accounted for.

5.5 Conical mould, made of cast iron, of sufficient capacity to contain all of the molten lead plus slag from the crucible fusion.

5.6 Analytical balance, sensitive to 1 mg.

5.7 Microbalance, sensitive to 1 µg.

5.8 Ordinary laboratory glassware, washed free of chlorides.

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

5.10 Atomic absorption spectrometer (AAS).

Instrumental conditions:

Flame: air/acetylene

Wavelengths:

242,8 nm for gold, or

328,1 nm for silver

5.11 Inductively coupled plasma (ICP) atomic emission spectrometer (optional)

5.12 Vibratory disc mill or **swing grinding mill**.

5.13 Hotplate.

6 Sample

6.1 Test sample

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

NOTE 6 A test sample is not required if predried test portions are to be used (see annex A).

6.2 Test portion

The test portion shall be 20 g, weighed to the nearest 0,1 mg. Taking multiple increments, extract a test portion from the test sample in such a manner that it is representative of the contents of the dish or tray. 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.

NOTES

7 If arsenic is present in the sample at a concentration above 2 % (*m/m*), this element should be removed by following the procedure in annex F; otherwise, interference with the cupellation stage may occur.

8 A small test programme has been conducted using test portions of 10 g instead of 20 g (see annex J).

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 9 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 Trial fusion

Carry out a trial fusion as described in annex B, to ensure that the mass of the lead button is between 30 g and 45 g.

7.3 Blank tests

Carry out a reagent blank test as described in annex C in parallel with the analysis, using the same quantities of all reagents with the addition of sufficient flour (4.5) to the flux to give a lead button of between 30 g and 45 g. Omit the test portion and the potassium nitrate. The total blank should not exceed 5 µg of gold or 100 µg of silver.

7.4 Charge preparation

Determine the mass of potassium nitrate (4.4) required in the charge as indicated by the trial fusion (see annex B) and include this reagent in the flux mixture (see table 1).

Thoroughly mix the test portion with a flux of the composition specified in table 1.

NOTE 10 The intimate mixing of flux components and the test portion is very important. All flux components should be in a finely divided state with a preferred particle size of less than 0,5 mm.

Table 1 — Recommended masses of flux components for the preparation of charges

Flux components	Mass g
Sodium carbonate (4.1)	30
Litharge (4.2)	210
Silica (4.3)	25
Potassium nitrate (4.4)	—

NOTES

11 If the copper content is greater than 30 % (*m/m*), the mass of litharge should be 30 times that of copper plus 35 g for the lead button. Alternatively, a 10 g or 15 g test portion can be used while retaining the flux composition given in table 1. If there are difficulties experienced in achieving a fluid melt, the amount of silica recommended in table 1 can be reduced to 19 g, while including 6 g of borax.

12 The quantities of oxidizing reagent in the mixture depend upon the reducing power of the test sample. The trial fusion (see annex B) will determine the mass of potassium nitrate necessary to yield a lead button of mass 30 g to 45 g.

Place the mixture in an assay crucible (5.3).

7.5 Primary fusion

Place the crucibles in the furnace (5.1) preheated to approximately 900 °C.

NOTE 13 If oil-fired or gas-fired furnaces are used, the fuel should be turned off immediately before opening the furnace.

Slowly raise the furnace temperature to 1 000 °C. Maintain this temperature until the fusion has been tranquil for at least 10 min.

NOTES

14 The total fusion time should not exceed 40 min. Some electric furnaces of an older type may require a longer time to reach the higher temperature. In such cases, the fusion time should be kept to a minimum.

15 Crucible lids or a borax or salt cover can be used to prevent loss of material by dusting or ebullition.

Pour the mixture into a dry conical mould (5.5), taking care to ensure that no loss of lead or slag occurs. Reserve the crucible for retreatment fusion.

Allow the mixture to cool and carefully separate the lead button from the slag. Hammer the lead button

as necessary to remove any small particles of adhering slag. Reserve the slag for retreatment.

Weigh the lead button. If the button weighs less than 30 g or more than 45 g, discard the button and slag and repeat the assay after appropriate adjustment of the oxidizing agent (see annex B).

NOTE 16 Buttons weighing less than 30 g may show poor collection efficiencies, whereas those exceeding 45 g may contain higher amounts of copper and other base metals.

7.6 Cupellation

Preheat a cupel (5.4) in the muffle furnace (5.2) at 790 °C ± 10 °C for approximately 20 min. Place the lead button obtained in 7.5 in the cupel. Allow the cupellation to proceed at the lowest possible muffle temperature.

NOTES

17 This temperature will generally be 790 °C ± 10 °C. Variations depend on the cupel type and furnace conditions.

18 High cupellation temperatures will cause higher silver losses. Low temperatures can cause "freezing" of the bead and incomplete cupellation. If only gold is determined, the furnace temperature can be raised to 890 °C ± 10 °C after approximately 80 % (*m/m*) of the molten lead has been absorbed.

Remove the cupel from the furnace and cool.

Carefully extract the precious metal bead and remove any adhering cupel material. Flatten the bead slightly and place in a 30 ml porcelain crucible.

Reserve the cupel for retreatment of residues.

7.7 Retreatment of residues

Crush both the cupel and the slag in a small jaw crusher (or manually) and place in the vibratory disc mill or swing grinding mill (5.12). Grind for 30 s to reduce the material to minus 150 µm.

NOTES

19 Longer grinding may cause caking of the material and heating of the grinding barrel.

20 The mill may be cleaned between samples by grinding small portions of broken glass or quartz.

21 To avoid contamination, it is recommended that grinding equipment be set aside specifically for residue retreatment.

22 Crucible lids or a borax or salt cover can be used. Improved silver recovery is indicated in the retreatment of residues, possibly through a better maintenance of reducing conditions throughout the fusion.

Thoroughly mix the ground residues with a flux of the composition specified in table 2.

Table 2 — Recommended masses of flux components for the retreatment of residues

Flux components	Mass
	g
Sodium carbonate (4.1)	60
Litharge (4.2)	60
Silica (4.3)	60
Flour (4.4)	4
Borax (4.6)	30

NOTES

23 The mass of flour required may vary depending on furnace characteristics and the amount of residual oxidant present. Therefore, the actual amount required should be determined by trial.

24 The combined mass of slag and cupel of the primary fusion, in addition to the flux components given in table 2, may exceed the capacity of the fire assay crucibles. In this case, it is permissible to split the residues into equal halves and fuse separately in two crucibles. The lead buttons obtained should then be scorified together and the resultant lead button cupelled.

Place the mixture in the original assay crucible.

NOTE 25 It is recommended that a fireclay lid or a layer of borax approximately 12 mm thick be used with this fusion. If borax is used as a cover, the amount used in the flux may be reduced accordingly.

Carry out the fusion as detailed in 7.5, slowly raising the furnace temperature to a final temperature of 1 100 °C. Pour the mixture into a dry conical mould (5.5), taking care that no loss of lead occurs. Discard the crucible.

Allow the mixture to cool and carefully separate the lead button from the slag. Hammer the lead button as necessary, to remove any pieces of slag.

NOTE 26 The addition of a collector onto the lead button is not normally necessary. However, if losses are very low, 2 mg of palladium wire may be hammered onto the lead button or added to the charge prior to the residue fusion, to collect both gold and silver into one bead. Silver can be used as a collector if it is not being determined.

Cupel the lead button by the procedure specified in 7.5 to obtain a second silver/gold bead.

7.8 Determination of gold in the primary bead

Add 10 ml of dilute nitric acid (4.8) to the bead in the porcelain crucible prepared in 7.6 and heat gently on a hotplate (5.13) for 20 min or until the reaction ceases.

NOTES

27 It is essential that chloride be absent during parting; otherwise, some of the gold may dissolve.

28 When the bead is treated with hot dilute nitric acid, silver will start to dissolve provided that the ratio of silver to gold in the bead exceeds 2,5 to 1. The rate of dissolution increases with increasing silver content of the bead. Rapid attack of the bead should be avoided by further dilution and slow heating to prevent disintegration of the gold. Should the ratio of silver to gold be less than 2,5 to 1 as shown by failure to part in hot dilute nitric acid, the bead should be inquarted (see annex D).

29 Carefully pour the solution into a 150 ml beaker, using a glass rod to avoid losses.

Add 15 ml of warm dilute nitric acid (4.9) to the porcelain crucible and continue heating gently until parting is complete. This should take approximately 25 min.

Carrefullly pour the solution into the 150 ml beaker. Wash the crucible and gold with four 15 ml volumes of hot water. Collect all the washings in the same 150 ml beaker. Reserve the solution for the determination of silver as specified in 7.10.

NOTE 29 The possibility of gold particles occurring in the collected parting and washing solutions can be determined by evaporating the solutions slowly down to 2 ml to 3 ml, then continuing with the determination as specified in 7.9.

Dry the gold sponge in the porcelain crucible on the hotplate.

Place the crucible in the muffle furnace (5.2) to anneal the gold at dull red heat for approximately 5 min.

Cool and weigh the gold to the nearest 1 µg (m_1). If the mass of the gold is less than 0,05 mg, it is recommended that the gold be dissolved in 2 ml of aqua regia solution (4.18) and the gold content be determined by atomic absorption spectrometry by the procedure specified in 7.9. Alternatively, repeat the fusion and cupellation, dissolve the prepared primary bead in aqua regia solution (4.8) and determine the gold content spectrometrically as specified in 7.9.