
**Copper, lead and zinc sulfide
concentrates — Determination of gold
and silver — Fire assay gravimetric
and flame atomic absorption
spectrometric method**

*Concentrés de sulfure de cuivre, de plomb et de zinc — 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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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

This third edition cancels and replaces the second edition (ISO 10378:2005), in which [6.2](#) has been technically revised and the warning notice in [A.3.1](#) has been updated.

Introduction

This International Standard describes a method for the determination of the mass fraction of gold and silver in copper, lead, and zinc sulfide concentrates. This International Standard was prepared to enable laboratories to determine the mass fraction of gold and silver in suitable samples using instrumental methods.

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Copper, lead and zinc sulfide concentrates — Determination of gold and silver — Fire assay gravimetric and flame atomic absorption spectrometric method

WARNING — This International Standard may involve hazardous materials, operations, and equipment. It is the responsibility of the user of this International Standard to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a fire assay gravimetric and flame atomic absorption spectrometric method for the determination of the mass fraction of gold and silver in copper, lead, and zinc sulfide concentrates as follows.

— Copper concentrates

The method is applicable to the determination of mass fractions of gold from 0,5 g/t to 300 g/t and of mass fractions of silver from 25 g/t to 1 500 g/t in copper sulfide concentrates containing mass fractions of copper from 15 % to 60 %.

— Lead concentrates

The method is applicable to the determination of mass fractions of gold from 0,1 g/t to 25 g/t and of mass fractions of silver from 200 g/t to 3 500 g/t in lead sulfide concentrates containing mass fractions of lead from 10 % to 80 %.

— Zinc concentrates

The method is applicable to the determination of mass fractions of gold from 0,1 g/t to 12 g/t and of mass fractions of silver from 10 g/t to 800 g/t in zinc sulfide concentrates containing mass fractions of zinc up to 60 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 glassware — Volumetric instruments — Methods for testing of capacity and for use*

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

3 Principle

3.1 General

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 pre-concentrated into a precious-metal alloy.

The stages that comprise the determinations are described in 3.2 to 3.6 inclusive.

3.2 Fusion

The samples are fused in a crucible after mixing with a litharge-based flux which, under reducing conditions, collects the precious metals in a metallic lead button.

3.3 Cupellation

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

3.4 Parting

Gold is separated from the primary bead by treatment with nitric acid. The gold prill is weighed. Gold prills having a mass less than 50 µg are dissolved in aqua regia and the gold is determined by atomic absorption spectrometry (AAS). Silver is determined in the parting solution by AAS.

3.5 Retreatment

All residues are retreated 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 second bead is dissolved in acids followed by analysis of both metals by AAS.

3.6 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 water that complies with grade 2 of ISO 3696.

4.1 Sodium carbonate, anhydrous.

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

4.3 Silica, precipitated.

4.4 Potassium nitrate or sodium nitrate

NOTE 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), chloride concentration <0,5 µg/ml.

4.8 Nitric acid, diluted 1+1.

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

4.9 Lead, foil, having a mass fraction of gold of less than 0,01 g/t and a mass fraction of silver of less than 0,2 g/t.

4.10 Silver, of minimum purity 99,99 %.

4.11 Hydrochloric acid (ρ_{20} 1,16 g/ml to 1,19 g/ml).

4.12 Thiourea, 10 g/l solution.

Add 1 g of thiourea to 100 ml of water.

4.13 Aqua regia

Mix three parts of hydrochloric acid (4.11) with one part of nitric acid (4.7). Prepare freshly as required.

4.14 Standard solutions

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

4.14.1 Silver, standard stock solution A (500 µg of Ag/ml).

Weigh 0,500 0 g of silver metal to the nearest 0,1 mg. Transfer to a 100 ml beaker, add 20 ml of diluted nitric acid (4.8), 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.

4.14.2 Silver, standard solution B (50 µg of Ag/ml).

Pipette 10,00 ml of silver standard stock solution A (4.14.1) into 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.

4.14.3 Gold, standard solution (1 000 µg of Au/ml).

Weigh 1,000 g of gold metal to the nearest 0,1 mg. Transfer to a 200 ml beaker, add 25 ml of aqua regia solution (4.13), and warm to dissolve. Cool and transfer to a 1 000 ml volumetric flask. Add 75 ml of hydrochloric acid (4.11), fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

4.14.4 Gold and silver, standard solution (100 µg of Au/ml + 50 µg of Ag/ml).

Pipette 10,00 ml of silver standard stock solution A (4.14.1) into a 100 ml volumetric flask. Add 40 ml of hydrochloric acid (4.11). Pipette 10,00 ml of gold standard solution (4.14.3) 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.

4.15 Calibration solutions

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

4.15.1 Gold/silver calibration solutions

Pipette 0,0 ml, 1,00 ml, 2,00 ml, 5,00 ml, and 10,00 ml of gold and silver standard solution (4.14.4) into a series of 100 ml one-mark volumetric flasks.

Add 40 ml of hydrochloric acid (4.11) 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 µg of Au/ml, 1,00 µg of Au/ml, 2,00 µg of Au/ml, 5,00 µg of Au/ml, and 10,00 µg of Au/ml; and 0,0 µg of Ag/ml, 0,50 µg of Ag/ml, 1,00 µg of Ag/ml, 2,50 µg of Ag/ml, and 5,00 µg of Ag/ml, and shall be freshly prepared.

4.15.2 Silver calibration 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 (4.14.2) into a series of 100 ml volumetric flasks. Add 10 ml of nitric acid (4.7), 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 µg of Ag/ml, 0,50 µg of Ag/ml, 1,00 µg of Ag/ml, 2,00 µg of Ag/ml, 3,00 µg of Ag/ml, 4,00 µg of Ag/ml, and 5,00 µg of Ag/ml, and shall be freshly prepared.

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

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 Assay crucibles, made of fire clay, of nominal capacity 200 ml to 600 ml, capable of withstanding corrosion by the samples and fluxes at 1 100 °C.

The crucible shall be of such a size that the charge does not fill the crucible to a depth greater than 3/4 the depth of the crucible.

5.4 Cupels, made of magnesium oxide, or bone-ash cupels having a nominal capacity of 50 g of molten lead.

The inside bottom of the cupel shall be concave, as recommended in the fire assay texts referred to in the Bibliography.

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 or less.

5.8 Ordinary laboratory glassware, washed free of chlorides.

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

5.10 Atomic absorption spectrometer (AAS), equipped with background correction and a glass bead in the spray chamber.

5.11 Inductively coupled plasma (ICP) atomic emission spectrometer.

5.12 Pulverizer.

5.13 Hotplate.

6 Sample

6.1 Test sample

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

NOTE 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 a test portion from the test sample in such a manner that it is representative of the whole contents of the dish or tray. Weigh to the nearest 1 mg approximately 10 g to 20 g of the test sample. 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.

NOTE 1 The precision of determination of the mass of contained gold in a lot is the sum of sampling and analytical precision. The presence of particulate gold in a concentrate contributes to imprecision of the analytical component of total precision. When it has been established that the presence of particulate gold in a concentrate causes unacceptable imprecision, the following mixing and sampling methods are recommended.

a) Mix the sample by either of the following preferred mixing methods as described in ISO 12743:2006:

- 15.3.2 c), strip mixing;
- 15.3.2 e), riffle, or rotary divider.

Mixing methods in items a), b), and d) in ISO 12743:2006, 15.3.2 are not recommended.

b) Extract a test portion from the test sample using either of the following methods described in ISO 12743:2006:

- 15.4.1 a), rotary sample division;
- 15.4.1 f), ribbon division.

Methods in items b) to e) and g) in ISO 12743:2006, 15.4.1 are not recommended.

Alternatively, the method specified in [Annex A](#) may be used to prepare predried test portions directly from the laboratory sample.

If a mass fraction of arsenic above 2 % is present in the sample, this element should be removed by following the procedure in Annex J; otherwise, interference with the cupellation stage may occur.

NOTE 2 If the mass fraction of copper is greater than 30 %, a 10 g or 15 g test portion is preferable (see the fourth paragraph of [7.4](#)).

For lead concentrates, the test portion should be 10 g to ensure an adequate supply of lead.

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 1 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.

NOTE 2 In the case where the ratio of silver to gold does not exceed 2,5 to 1 and the procedure specified in [7.10](#) is carried out for the silver determination, separate determinations for gold and silver will be necessary (see [Annex D](#)). Four test portions, therefore, are required, i.e. two for determinations of gold and two for silver.

NOTE 3 [Annex I](#) contains a flowsheet of the method.

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](#)) and flour ([4.5](#)) required in the charge, as indicated by the trial fusion (see [Annex B](#)), and include this reagent in the flux mixture. Typical masses of the flux components for copper, lead, and zinc concentrates are shown in [Tables 1](#) to [3](#) respectively.

Thoroughly mix the test portion with a flux.

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 — Typical masses of flux components for copper concentrates

Flux components	Mass g
Sodium carbonate (4.1)	30
Litharge (4.2)	210
Silica (4.3)	25
Potassium nitrate (4.4)	—
Flour (4.5)	—
Test portion	20

If the mass fraction of copper is greater than 30 %, 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 borax.

Table 2 — Typical masses of flux components for lead concentrates

Flux components	Mass, g
Sodium carbonate (4.1)	30
Litharge (4.2)	100
Silica (4.3)	10
Borax (4.6)	10
Potassium nitrate (4.4)	—
Flour (4.5)	—
Test portion	10

Table 3 — Typical masses of flux components for zinc concentrates

Flux components	Mass, g
Sodium carbonate (4.1)	30
Litharge (4.2)	120
Silica (4.3)	10
Borax (4.6)	10
Potassium nitrate (4.4)	—
Flour (4.5)	—
Test portion	20

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.

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 to 1 050 °C. Maintain this temperature until the fusion has been tranquil for at least 10 min.

The optimum furnace setting temperature depends on the furnace structure and the position of the thermometer. If unfused material remains in the bottom of assay crucibles, the setting temperature of the furnace should be raised and the assay repeated.

To minimize crucible corrosion and build-up of impurities in the lead button, the overall fusion time should not exceed 40 min. However, should the fusion take an extended time to settle, the fusion time may need to exceed 40 min.

It is recommended that a fire-clay lid or a layer of salt or borax approximately 12 mm thick be used with this fusion to prevent loss of material by dusting or ebullition. If borax is used as a cover, the amount used in the flux may be reduced accordingly. If some “shotting” of the lead is noted in the slag (this is common for zinc concentrates), it is recommended that the extra borax be retained.

Pour the mixture into a dry conical mould (5.5), taking care 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 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

Place the lead button obtained in 7.5 into a preheated cupel (5.4) in a muffle furnace (5.2) at 900 °C. Allow the cupellation to proceed at the lower muffle temperature of approximately 860 °C with a steady air flow.

Variations depend on the cupel type and furnace conditions. In the case of cupels made of bone ash, a cupellation temperature of 820 °C is recommended.

NOTE 1 In the case where only gold is being determined, it could be effective to raise the furnace temperature to 900 °C to finish the cupellation after the visible lead melt on the cupel is approximately 10 mm in diameter [approximately 80 % (mass fraction) of lead absorbed].

High cupellation temperatures will cause higher silver losses and low temperatures can cause “freezing” of the bead and incomplete cupellation. It is recommended that loss of silver during the cupellation process be determined, to decide upon the furnace conditions (see Annex E).

Remove the cupel from the furnace and cool.

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

NOTE 2 A test tube can be used instead of a porcelain crucible.

NOTE 3 If the ratio of silver to gold in the primary bead is greater than 2,5 to 1, the silver can be determined by the gravimetric method instead of the procedure specified in 7.10. For the gravimetric method, weigh the primary bead, in micrograms, to the nearest 1 µg (m_0), carry out the parting by the procedure specified in 7.8, and determine the impurities in the parting solutions and washings by the procedure specified in Annex G.

NOTE 4 If it is difficult to recover the bead because of its small size, 1 mg of palladium can be added before fusion. In this case, the palladium bead is dissolved and determined by the procedure specified in 7.9.

Reserve the cupel for retreatment of residues.

7.7 Retreatment of residues

Place both the cupel and the slag in a pulverizer (5.12) and pulverize for about 20 s to reduce the material to minus 150 µm.

If magnesium cupels are used, it is recommended that the slag and the cupel be retreated separately.

NOTE 1 Longer grinding can cause caking of the material and heating of the grinding barrel.

NOTE 2 The pulverizer may be cleaned between samples by grinding small portions of broken glass or quartz.

Thoroughly mix the ground residues with a flux. Typical composition of the flux is shown in [Table 4](#).

Table 4 — Typical masses of flux components for the retreatment of residues

Flux components	Mass, g	
	Magnesium oxide cupel	Bone ash cupel
Sodium carbonate (4.1)	50 to 60	40
Litharge (4.2)	50 to 60	45
Silica (4.3)	50 to 60	20
Flour (4.4)	4	2 to 3
Borax (4.6)	30 to 50	15

The mass of flour shown in [Table 4](#) is typical. The mass should be sufficient to produce a 30 g to 45 g lead button.

The combined mass of slag and cupel of the primary fusion, in addition to the flux components given in [Table 4](#), may exceed the capacity of the assay crucibles, or the re-fusion may be so reactive that the fusions may froth over. In these cases, it is permissible to split the residues into equal halves and fuse separately in two crucibles. The lead buttons obtained should be cupelled separately, or be scorified together and the resultant lead button cupelled.

Place the mixture in the original assay crucible.

Carry out the fusion as detailed in [7.5](#) and discard the crucible and slag.

Cupel the lead button as detailed in [7.6](#) to obtain a second bead and discard the cupel.

7.8 Determination of gold in the primary bead

Add 10 ml of dilute nitric acid ([4.8](#)) to the primary 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.

NOTE 1 It is essential that chloride be absent during parting; otherwise, some of the gold may dissolve.

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 mass fraction of silver 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](#)).

If there is danger of the gold sponge crumbling during the parting operation, it is recommended that the operation be carried out with sulfuric acid (see Annex F).

Carefully pour the solution into a 200 ml beaker by decantation to avoid losses.

Add 15 ml of warm dilute nitric acid ([4.8](#)) to the porcelain crucible and continue heating gently until parting is complete. This should take approximately 25 min.