
**Zinc sulfide concentrates — Determination
of gold content — Acid dissolution/solvent
extraction/flame atomic absorption
spectrometric method**

*Concentrés sulfurés de zinc — Dosage de l'or — Méthode par dissolution
acide, extraction à l'aide d'un solvant et spectrométrie d'absorption
atomique dans la flamme*

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ISO 15249:1998

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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 15249 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores*.

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

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Zinc sulfide concentrates — Determination of gold content — Acid dissolution/solvent extraction/flame atomic absorption spectrometric method

1 Scope

This International Standard specifies an acid decomposition, solvent extraction, flame atomic absorption spectrometric procedure for the determination of gold in zinc sulfide concentrates.

The method is applicable to the determination of gold in zinc concentrates containing up to 60 % (*m/m*) zinc in the form of zinc blende and related materials.

The method is applicable to gold contents from 0,5 g/t to 12 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 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

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*.

3 Principle

Roasting of the concentrate at 450 °C and 600 °C to remove arsenic and sulfur, followed by decomposition in bromine-aqua regia-hydrofluoric acid. Extraction of the gold into DIBK containing Aliquat 336 from an aqua regia medium and determination by flame atomic absorption spectrometric at 242,8 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and water that complies with grade 2 of ISO 3696.

4.1 Gold metal, minimum 99,99 % purity.

4.2 Nitric acid, (ρ_{20} 1,42 g/ml).

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

4.4 Aqua regia

Mix three parts hydrochloric acid (4.3) and one part nitric acid (4.2) with three parts of water. Prepare freshly as required.

4.5 Bromine liquid, 99,5 % *m/m*.

4.6 Hydrofluoric acid (ρ_{20} 1,19 g/ml).

WARNING — Even when diluted, hydrofluoric acid is extremely dangerous and harmful to the eyes and skin: rubber gloves and goggles should be worn when using this acid. Hydrofluoric acid attacks glassware. Care should be taken to minimize the time of acid contact with glassware. Use only in a mechanically ventilated fume cupboard.

4.7 Di-iso butyl ketone (ρ_{20} 0,81 g/ml).

4.8 Aliquat 336 (methyl-trioctyl ammonium chloride).

4.9 1 % Aliquat 336 in di-iso butyl ketone

Add 1 ml of Aliquat 336 (4.8) to 99 ml of di-iso butyl ketone (4.7) and mix thoroughly.

4.10 Gold standard solutions

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

4.10.1 Gold standard solution (1 000 $\mu\text{g/ml}$).

Weigh 0,100 0 g of gold metal (4.1) into a 50 ml beaker, add 10 ml aqua regia (4.4), cover and heat to dissolve the gold. Wash and remove the cover, and evaporate to near dryness (do not allow to go to dryness otherwise gold will precipitate). Cool, add 5 ml of hydrochloric acid (4.3) and transfer the solution quantitatively to a 100 ml volumetric flask, dilute to volume with water and mix thoroughly.

4.10.2 Gold standard solution (100 $\mu\text{g/ml}$).

Pipette 10 ml of gold standard solution (4.10.1) into a 100 ml volumetric flask, add 5 ml of hydrochloric acid (4.3), dilute to volume with water and mix thoroughly.

4.10.3 Gold standard solution (10 $\mu\text{g/ml}$).

Pipette 10 ml of gold standard solution (4.10.2) into a 100 ml volumetric flask, add 5 ml of hydrochloric acid (4.3), dilute to volume with water and mix thoroughly.

This solution shall be freshly prepared.

4.11 Calibration solutions

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

To each of seven 125 ml separating funnels add 75 ml of water and 20 ml of aqua regia (4.4). Add from a burette (5.2) 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, 5 ml and 6 ml of gold standard solution (4.10.3). Treat these solutions using the extraction procedure given in 7.4.

These standards contain 0 μg , 10 μg , 20 μg , 30 μg , 40 μg , 50 μg and 60 μg of gold.

5 Apparatus

Ordinary laboratory equipment plus the following.

- 5.1 Volumetric glassware**, of class A complying with ISO 385-1, ISO 648 and ISO 1042 and used in accordance with ISO 4787.
- 5.2 Burette**, A grade, 10 ml capacity capable of being read to 0,02 ml.
- 5.3 Muffle furnace**, capable of giving an adequate zone of uniform temperature between 450 °C and 600 °C. A ventilated furnace is preferred.
- 5.4 Crucibles**, porcelain, having a depth of 10 mm to 15 mm and a diameter of 60 mm to 75 mm.
- 5.5 Atomic absorption spectrometer (AAS)**, equipped with background correction and a glass bead, rather than a flow spoiler, in the spray chamber. Scale expansion and a flow adjustable nebulizer are recommended.
- 5.6 Centrifuge**, capable of holding at least 15 ml tubes and revolving at 0,833 s⁻¹ (3 000 rpm).
- 5.7 Centrifuge tubes**, 15 ml volume with caps. Polypropylene is suitable.
- 5.8 Separating funnels**, of 125 ml capacity. Borosilicate glass with PTFE stopcocks are recommended.

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).
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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 0,1 mg, approximately 5 g of the test sample. At the same time as the test portion is weighed, weigh test portions for the level 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, and as far as possible under repeatability conditions, on each test sample.

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

7.3 Decomposition of test portion

Transfer the test portion to a porcelain crucible and place in the muffle furnace (5.3) at 450 °C and heat for 2 h. Raise the muffle furnace temperature to 600 °C and continue heating for 1 h.

Remove the crucible from the furnace, cool, and transfer the roasted test portion to a 250 ml conical beaker.

Add 10 ml of water, swirl to form a slurry and add 2 ml of bromine (4.5) and mix. Cover with a watchglass, swirl to mix and allow to stand at room temperature for 15 min.

Add 15 ml of hydrochloric acid (4.3) and digest on a hotplate at 90 °C until fumes of bromine are expelled. Continue the digestion for 30 min. Remove from the hotplate and cool.

Remove the watchglass and rinse the underside with a minimum of water, collecting the washings in the conical beaker. Carefully add 10 ml of nitric acid (4.2) and 1 ml of hydrofluoric acid (4.6). Return the beaker to the hotplate and gently evaporate to near dryness.

NOTE Do not allow the digestion mixture to go to dryness, as gold will be lost.

Rinse the sides of the beaker with a minimum of water, add 20 ml of aqua regia (4.4), cover with the watchglass, return to the hotplate and heat gently for 10 min. Remove from the hotplate and cool.

7.4 Extraction

Transfer the test solution to a 125 ml separating funnel (5.8), add sufficient water to make the volume to 100 ml and swirl to mix.

Add 8 ml of 1 % Aliquat in di-iso butyl ketone (4.9), stopper and shake for 1 min. Allow the phases to separate and slowly run off the lower aqueous layer until about 4 ml of aqueous material remain. Transfer these, with the organic layer, to a 15 ml centrifuge tube (5.7). Centrifuge until a clear organic layer is obtained.

7.5 Determination of gold

Determine the gold content in the organic layer by flame atomic absorption spectrometry using calibration solutions (4.11). As a guide, the following atomic absorption conditions are recommended; however, the instrument should be optimized to give maximum sensitivity and as near as practical to a linear relationship between absorbance and concentration.

- flame: air-acetylene oxidizing
- wavelength: 242,8 nm
- lamp current: 4 mA
- background corrector: on
- aspiration rate: aspirate di-iso butyl ketone as waste solution; adjust (reduce) flow rate to give an oxidizing flame
- integration time: 3 s
- number of integrations: 5

NOTE The organic used for extraction, [di-iso butyl ketone (4.7)] should be aspirated between each standard solution and between each test solution during the determination.

Perform three measurements on each standard solution. Calculate to three significant figures the mean absorbance for each standard solution, provided that the range of values does not exceed 0,003 absorbance units. If this range is exceeded, repeat the calibrations.

The test solutions should be treated in the same manner. Plot a calibration graph of absorbance versus concentration of gold in the organic layer.

8 Expression of results

The gold content of the test portion w_{Au} , expressed in grams per tonne, is given by the following equation:

$$w_{\text{Au}} = \frac{C}{m} \times \frac{100}{100 - H} \quad \dots (1)$$

where

C is the gold content of the organic layer in micrograms;

m is the mass of test portion in grams;

H is the hygroscopic moisture content as a percentage of the test portion (in the case of a predried test portion being used, $H = 0$).

9 Precision

9.1 Expression of precision

The precision of this analytical method is expressed by the following equations:

$$s_r = 0,037 \sqrt{\bar{X}} + 0,004 \quad \dots (2)$$

$$s_L = 0,082 \sqrt{\bar{X}} + 0,100 \quad \dots (3)$$

where

\bar{X} is the mean content of gold, in grams per tonne, in the sample;

s_r is the within-laboratory standard deviation, in grams of gold per tonne;

s_L is the between-laboratories standard deviation, in grams of gold per tonne.

See annex C.

9.2 Method for obtaining the final result

See annex B.

Calculate the following quantities from the duplicate results X_1 and X_2 and process according to the flow chart in annex B:

$$\text{Mean of duplicates } \bar{X} = (X_1 + X_2)/2 \quad \dots (4)$$

$$\text{Within-laboratory standard deviation } s_r = 0,037 \sqrt{\bar{X}} + 0,004 \quad \dots (5)$$

$$\text{Repeatability limit } r = 2,8 s_r \quad \dots (6)$$

9.3 Precision between laboratories

The precision between laboratories is used to determine the agreement between the results reported by two (or more) laboratories. It is assumed that all laboratories have followed the same procedure.

Calculate the following quantities.

$$\text{Mean of final results } \mu_{1,2} = (\mu_1 + \mu_2)/2 \quad \dots (7)$$

$$\text{Between-laboratories standard deviation } s_L = 0,082 \sqrt{\mu_{1,2} + 0,100} \quad \dots (8)$$

$$\text{Within-laboratory standard deviation } s_r = 0,037 \sqrt{\mu_{1,2} + 0,004} \quad \dots (9)$$

$$\text{Permissible difference } P = 2,8 \sqrt{(s_L^2 + s_r^2)/2} \quad \dots (10)$$

$$\text{Range } E = |\mu_1 - \mu_2| \quad \dots (11)$$

where

μ_1 is the final result, in grams of gold per tonne, reported by laboratory 1;

μ_2 is the final result, in grams of gold per tonne, reported by laboratory 2.

If E is equal to or less than P , the final results are in agreement.

9.4 Check of trueness

The trueness of the analytical method can be checked by applying it to a certified reference material (CRM). The procedure is the same as that described in clause 7. When the precision has been confirmed, the final laboratory result can be compared with the certified value, A_c .

The following two possibilities exist:

$$|\mu_c - A_c| \leq C \quad \text{ISO 15249:1998} \quad \dots (12)$$

If this condition exists, the difference between the reported result and the certified value is statistically insignificant.

$$|\mu_c - A_c| > C \quad \dots (13)$$

If this condition exists, the difference between the reported result and the certified value is statistically significant.

In equations (12) and (13), the symbols are defined as follows:

μ_c is the final result, in grams of gold per tonne, of the certified reference material;

A_c is the certified value, in grams of gold per tonne, of the certified reference material;

C is a quantity, in grams of gold per tonne, depending on the type of the certified reference material used.

NOTE 1 The reference materials used for this purpose should be prepared and certified in accordance with ISO Guide 35.

Where the reference material is certified/characterized by an interlaboratory test programme, the quantity C , in grams of gold per tonne, is given by the following equation:

$$C = 2 \sqrt{s_L^2 + (s_r^2/n) + s^2\{A_c\}} \quad \dots (14)$$

where

$s^2\{A_c\}$ is the variance of the certified value;

n is the number of replicate determinations.

Where the reference material is certified/characterized by one laboratory, the quantity C , in grams of gold per tonne, is given by the following equation:

$$C = 2\sqrt{2s_L^2 + (s_r^2/n)} \quad \dots (15)$$

NOTE 2 It is recommended that this type of certified reference material be avoided, unless the particular CRM is known to have an unbiased certified value.

10 Test report

The test report shall contain the following information:

- a) identification of the test sample;
- b) reference to this International Standard, i.e. ISO 15249;
- c) gold content of the sample, expressed in grams per tonne;
- d) date on which the test was carried out;
- e) any occurrence noticed during the determination that may have had an influence on the results.

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