
**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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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.

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 $0,833\text{ s}^{-1}$ (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).

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.