INTERNATIONAL STANDARD

ISO

15247

Zinc sulfide concentrates — Determination of silver content — Acid dissolution and flame atomic absorption spectrometric method

Concentrés sulfurés de zinc — Dosage de l'argent — Méthode par dissolution acide 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 second edition cancels and replaces the first edition (ISO 15247:1999), of which the warning in A.3.1 in Annex A has been revised.

This corrected version of ISO 15247:2015 incorporates the following corrections:

— in 4.4, "hydrofluoric acid" has been changed to "hydrochloric acid";

— in 9.1, in the definition of variables used in Formula (3), "s_R" has been changed to "s_L".

Zinc sulfide concentrates — Determination of silver content — Acid dissolution and flame atomic absorption spectrometric method

1   Scope

This International Standard specifies an acid dissolution and flame atomic absorption spectrometric method for the determination of silver content of zinc sulfide concentrates.

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

The method is applicable to silver contents from 10 g/t to 500 g/t.

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, zinc and nickel sulfide concentrates — Determination of hygroscopic moisture content of the analysis sample — Gravimetric method*

3   Principle

Decomposition of the concentrate in hydrochloric and nitric acids. Dissolution of the digestion residue in hydrochloric acid and measurement by flame atomic absorption at 328,1 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   Silver metal, minimum 99,99 % purity.

4.2   Nitric acid, (ρ_{20} 1,42 g/ml), chloride content < 0,5 mg/ml.

4.3   Nitric acid, (500 ml/l).

To 250 ml of water carefully add, with stirring, 250 ml of nitric acid (4.2).

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

4.5 Hydrochloric acid, (200 ml/l).

To 800 ml of water carefully add, with stirring, 200 ml of hydrochloric acid (4.4).

4.6 Ammonia solution, (ρ_{20} 0,89 g/ml).

4.7 Ammonia solution, (250 ml/l).

To 750 ml of water add, with stirring, 250 ml of ammonia solution (4.6).

4.8 Silver standard solutions

4.8.1 Silver standard solution, (1 000 µg/ml).

Weigh 0,500 0 g of silver metal (4.1) into a 250 ml conical beaker, add 50 ml of nitric acid (4.3), cover, and heat gently until the metal dissolves. Remove the cover and evaporate gently to near dryness. Add 250 ml of hydrochloric acid (4.4) and warm until the solution clears. Cool, and transfer to a 500 ml volumetric flask. Dilute to volume with water and mix thoroughly.

This solution should be freshly prepared, unless it is being used on a regular basis.

4.8.2 Silver standard solution, (100 µg/ml).

Pipette 10 ml of silver standard solution (4.8.1) into a 100 ml volumetric flask containing 45 ml of hydrochloric acid (4.4). Dilute to volume with water and mix thoroughly.

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

Silver standard solutions should be stored in brown glass bottles.

4.9 Calibration solutions

To six 100 ml volumetric flasks, each containing 20 ml of hydrochloric acid (4.4), add from a burette (5.2) 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, and 5 ml of silver standard solution (4.8.2). Dilute to volume with water and mix thoroughly.

These standards contain 0 µg, 1 µg, 2 µg, 3 µg, 4 µg, and 5 µg of silver per ml and shall be freshly prepared.

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

5 Apparatus

Ordinary laboratory apparatus and the following.

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

5.2 Burette, grade A 10 ml capacity, capable of being read to 0,02 ml.

5.3 Atomic absorption spectrometer (AAS), equipped with a glass bead in the spray chamber rather than a flow spoiler.

5.4 Balance, precision analytical, capable of being read to 0,1 mg.

6 Samples

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 1 g of test sample. At the same time as the test portion is weighed, weigh test portions for the determination of hygroscopic moisture in accordance with ISO 9599.

Alternatively, the method specified in Annex A can 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.

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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 250 ml conical beaker and moisten with 1 ml of water.

All glassware should be washed in ammonia (4.7) and rinsed with water prior to use to remove any silver adhering to the glass surface.

Add 35 ml of nitric acid (4.3), cover with a watch glass, and heat at a low temperature until the reaction ceases.

Add 10 ml of hydrochloric acid (4.4), raise the cover slightly, and evaporate to dryness. Remove from the hotplate and cool.

Add a further 10 ml of hydrochloric acid (4.4) and again evaporate to dryness.

Rapid heating can cause samples to splatter or spit. Care should be taken to ensure that this does not occur.

Re-dissolve in 25 ml of hydrochloric acid (4.5). Heat to boiling, remove from the hotplate and cool.

Transfer the solution to the volumetric flask indicated in Table 1 by washing and diluting to volume with hydrochloric acid (4.5).

Table 1 — Volumetric flask size

Ag content g/t	Volumetric flask ml
10 to 250	50
250 to 500	100

7.4 Determination of silver

Determine the silver content of the test portion by flame atomic absorption spectrometry using calibration solutions (4.9). As a guide, the following atomic absorption settings 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:	328,1 nm
Lamp current:	5 mA
Background correction:	none
Aspiration rate:	optimize for maximum signal
Integration time:	3 s
Number of integrations:	5

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 calibration.

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

During all FAAS determinations, the test solutions and calibration solutions should have the same temperature as well as the same acid concentrations.

8 Expression of results

The silver content of the test portion, w_{Ag}, expressed in grams per tonne, is given by Formula (1):

$$w_{Ag} = \frac{C \times V}{m} \times \frac{100}{100 - H} \tag{1}$$

where

C is the silver content of the analysis solution, in micrograms per millilitre;

V is the volume of the analysis solution, in millilitres (see 7.3);

m is the mass of the test portion, in grams;

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

9 Precision

9.1 Expression of precision

The precision of this analytical method is expressed by Formulae (2) and (3):

$$s_r = 0,009\,5\,\overline{X} + 0,182\,6 \tag{2}$$

$$s_L = 0,031\,1\,\overline{X} + 0,881\,3 \tag{3}$$

where

$\overline{X}$ is the mean content of silver, in grams per tonne, in the sample;

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

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

NOTE   Additional information is given in 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 flowchart in Annex B:

a) Mean of duplicates:

$$\overline{x} = \frac{x_1 + x_2}{2} \tag{4}$$

b) Within-laboratory standard deviation:

$$s_r = 0,009\,5\,\overline{X} + 0,182\,6$$

c) Repeatability limit:

$$r = 2,8\,s_r \tag{5}$$

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 followed the same procedure.

Calculate the following quantities:

a) Mean of final results:

$$\mu_{1,2} = \frac{\mu_1 + \mu_2}{2} \tag{6}$$

b) Within-laboratory standard deviation:

$$s_L = 0,031\,1\,\mu_{1,2} + 0,881\,3 \tag{7}$$

c) Between-laboratories standard deviation:

$$s_r = 0{,}009\,5\,\mu_{1,2} + 0{,}182\,6 \tag{8}$$

d) Permissible difference:

$$P = 2{,}8 \sqrt{s_L^2 + \frac{s_r^2}{2}} \tag{9}$$

e) Range:

$$E = |\mu_1 - \mu_2| \tag{10}$$

where

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

> μ_2 is the final result, in grams of silver 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:

a) $|\mu_C - A_C| \le C$ (11)

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

b) $|\mu_C - A_C| > C$ (12)

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

where

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

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

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

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 silver per tonne, is given by Formula (13):

$$C = 2\sqrt{s_L^2 + \frac{s_r^2}{n} + s^2\{A_c\}} \tag{13}$$

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 silver per tonne, is given by the following equation:

$$C = 2\sqrt{2s_L^2 + \frac{s_r^2}{n}} \tag{14}$$

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

10 Test reports

The test report shall include the following information:

a) identification of the sample;

b) a reference to this International Standard, i.e. ISO 15247;

c) mass fraction of cadmium in the sample, expressed as a percentage;

d) date on which the test was carried out;

e) any occurrences noticed during the determination which could have had an influence on the results.