



BSI Standards Publication

**Copper and zinc sulfide concentrates —
Determination of silver content — Acid
digestion and flame atomic absorption
spectrometric or inductively coupled plasma
optical emission spectrometric method**

National foreword

This British Standard is the UK implementation of ISO 11456:2025.

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**Copper and zinc sulfide
concentrates — Determination of
silver content — Acid digestion
and flame atomic absorption
spectrometric or inductively
coupled plasma optical emission
spectrometric method**

*Concentrés sulfurés de cuivre et de zinc — Dosage de l'argent
— Méthode par digestion acide et spectrométrie d'absorption
atomique dans la flamme ou spectroscopie d'émission optique
par plasma à couplage inductif*

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Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

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Copper and zinc sulfide concentrates — Determination of silver content — Acid digestion and flame atomic absorption spectrometric or inductively coupled plasma optical emission spectrometric method

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

1 Scope

This document specifies an acid digestion and flame atomic absorption spectrometric (AAS) method, or inductively coupled plasma optical emission spectrometric (ICP-OES) method for the determination of the mass fraction of silver in copper and zinc sulfide concentrates.

The method is applicable to concentrates having silver contents in the following ranges.

- copper concentrates: 10 g/t to 800 g/t ;
- zinc concentrates: 10 g/t to 800 g/t

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 glass and plastic ware — Volumetric instruments — Methods for testing of capacity and for use*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

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

ISO 12743, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

The test portion is decomposed by acid digestion using nitric and hydrochloric acids. Silver is determined by comparison against matrix-matched standards using AAS or ICP-OES.

5 Reagents

During the analysis only reagents of analytical grade and grade 2 water of ISO 3696 shall be used.

5.1 Solids

5.1.1 Silver metal ($\geq 99,99\%$).

5.1.2 Copper metal ($\geq 99,99\%$), ($< 0,000\,05\%$ silver).

Care should be taken when choosing the copper metal, because normal copper cathode contains about 0,001 % of silver.

5.1.3 Zinc metal ($\geq 99,99\%$), ($< 0,000\,05\%$ silver).

5.1.4 Iron metal ($\geq 99,99\%$), ($< 0,000\,05\%$ silver).

5.1.5 Yttrium oxide (Y_2O_3 , $\geq 99,99\%$).

5.2 Acids

5.2.1 Nitric acid ($\rho_{20} = 1,38\text{ g/ml}$).

5.2.2 Nitric acid, dilute (1 + 1).

Slowly add 500 ml of nitric acid ([5.2.1](#)) to 500 ml of water while stirring.

5.2.3 Hydrochloric acid ($\rho_{20} = 1,16\text{ g/ml}$).

5.2.4 Hydrochloric acid, dilute (1 + 1).

Slowly add 250 ml of hydrochloric acid ([5.2.3](#)) to 250 ml of water while stirring.

5.2.5 Hydrochloric acid, dilute (1 + 3).

Slowly add 250 ml of hydrochloric acid ([5.2.3](#)) to 750 ml of water while stirring.

5.2.6 Sulfuric acid ($\rho_{20} = 1,84\text{ g/ml}$).

5.2.7 Sulfuric acid, dilute (1 + 1).

Slowly add 100 ml of sulfuric acid ([5.2.6](#)) to 100 ml of water while stirring. The addition of sulfuric acid to water generates heat and shall be performed with adequate precautions.

5.2.8 Perchloric acid ($\rho_{20} = 1,77\text{ g/ml}$).

5.2.9 Hydrofluoric acid ($\rho_{20} = 1,13\text{ g/ml}$).

5.3 Standard solutions

5.3.1 Primary silver standard (1 000 mg/l).

Weigh 1,000 g of silver metal (5.1.1) into a 300 ml beaker. Add 50 ml of dilute nitric acid (5.2.2) and 50 ml of water and swirl to mix. Heat gently until nitrogen oxides are generated. Cool to room temperature. Quantitatively transfer this solution into a 1 000 ml volumetric flask, dilute to the mark with water and mix. Store in an amber-coloured glass container.

Alternatively, purchase a suitable high-quality prepared standard solution.

NOTE There are the reference solutions with the metrological traceability secured.

5.3.2 Working silver standard A (100 mg/l).

Pipette 20 ml of primary silver standard (5.3.1) into a 200 ml volumetric flask containing 100 ml of hydrochloric acid (5.2.3) and 50 ml of water and mix well. Allow to the solution to cool and then dilute to the mark with water and mix. Store in an amber-coloured glass container.

This solution should be freshly prepared. If any turbidity is present, then remake this standard.

5.3.3 Working silver standard B (10 mg/l).

Pipette 5 ml of primary silver standard (5.3.1) into a 500 ml volumetric flask containing 250 ml of dilute hydrochloric acid (5.2.4) and mix well. Allow to the solution to cool and then dilute to the mark with water and mix. Store in an amber-coloured glass container.

This solution should be freshly prepared. If any turbidity is present, then remake this standard.

5.3.4 Copper matrix/interference standard solution (25,0 mg/ml).

Dissolve 5,00 g of copper metal (5.1.2) with 50 ml of dilute nitric acid (5.2.2). Heat and evaporate to approximately 25 ml to remove nitrogen oxides. Transfer to a 200 ml volumetric flask and add 100 ml of dilute hydrochloric acid (5.2.4). Then fill up with water nearly to the mark, mix and cool to room temperature. Then fill up exactly to the mark and mix again.

5.3.5 Zinc matrix/interference standard solution (25,0 mg/ml).

Dissolve 5,00 g of zinc metal (5.1.3) with 50 ml of dilute hydrochloric acid (5.2.4). Transfer to a 200 ml volumetric flask and add 50 ml of dilute hydrochloric acid (5.2.4). Then fill up with water nearly to the mark, mix and cool to room temperature. Then fill up to the mark and mix again.

5.3.6 Iron matrix/interference standard solution (25,0 mg/ml).

Dissolve 5,00 g of iron metal (5.1.4) with 50 ml of dilute hydrochloric acid (5.2.4). Heat gently until the reaction is complete. Add 5 ml of nitric acid (5.2.1) and heat. Transfer to a 200 ml volumetric flask and add 50 ml of dilute hydrochloric acid (5.2.4). Then fill up with water nearly to the mark, mix and cool to room temperature. Then fill up to the mark and mix again.

5.3.7 Primary yttrium standard (1 000 mg/l).

Weigh 1,270 g of yttrium trioxide (5.1.5) into a 300 ml beaker. Add 50 ml of dilute nitric acid (5.2.2). Heat gently. Cool to room temperature. Quantitatively transfer this solution into a 1 000 ml volumetric flask, dilute to the mark with water and mix well.

5.3.8 Yttrium internal standard solution (100 mg/l).

Pipette 20 ml of primary yttrium standard (5.3.7) into a 200 ml volumetric flask containing 100 ml of hydrochloric acid (5.2.3) and 50 ml of water. Allow the solution to cool. Dilute to volume with water and mix well.

6 Apparatus

Use laboratory glassware and equipment that is free of silver and chloride contamination.

6.1 Analytical balance, sensitivity to 0,1 mg.

6.2 Laboratory hotplate with temperature controller.

6.3 Normal laboratory glassware, which shall conform to ISO 648 and ISO 1042 and shall be used in accordance with ISO 4787.

6.4 Polytetrafluoroethylene beaker, 200 ml capacity.

6.5 Insoluble filter paper, an ash content of a mass fraction of 0,01 % or less and a nominal particle retention of 25 µm or less.

6.6 Atomic absorption spectrometer (AAS).

6.7 Inductively coupled plasma optical emission spectrometer (ICP-OES).

7 Instrument operating parameters

7.1 Analytical parameters

A variety of AAS and ICP-OES are available and the manufacturer's instructions supplied with the instrument provide adequate information for their operation. Refer to the manufacturer's instructions for optimizing AAS and ICP-OES. The instrument should be optimized to give maximum sensitivity and as near as practical to a linear relationship between absorbance or emission intensity and concentration.

7.2 Flame of AAS

When using the AAS, use an air-acetylene (oxidizing) flame.

7.3 Measurement wavelength

The recommended measurement wavelengths are as follows:

- AAS: 328,1 nm.
- ICP-OES: Silver 328,068 nm.
- Yttrium: 371,030 nm or 324,228 nm.

The wavelength resolution of the ICP-OES differs depending on the type of device. Therefore, interference should be checked for in advance and an appropriate measurement wavelength should be selected. Higher order spectral lines may be selected in a device that can measure higher order spectral lines. When a suitable emission line without interference cannot be selected, the interference amount shall be suitably corrected.

The ICP-OES measuring wavelength with an emission intensity suitable for the intended range of determination should be selected from the measuring wavelengths of the respective element. In this case, the detection limit, measuring accuracy, etc. should be sufficiently investigated.

There are two types of observation methods in ICP-OES from the emission part, which are the radial view method and the axial view method, and both may be used. However, the axial observation method has a narrower linear range of the calibration curve and more interferences than the radial view method.

7.4 Background correction mechanism

When using the AAS, a background correction mechanism shall be used. On the other hand, when using the ICP-OES, a background correction mechanism may be used.

8 Sample and sample preparation

8.1 Laboratory sample

Laboratory samples shall be taken and prepared in accordance with the procedures described in ISO 12743.

8.2 Test sample

Prepare an air-equilibrated test sample and a hygroscopic moisture test sample in accordance with ISO 9599.

NOTE A test sample is not required if pre-dried test portions are used, see [Annex B](#).

9 Procedure

9.1 Number of determinations

Carry out the determination at least in duplicate, 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.

9.2 Test portion

Taking multiple increments, extract approximately 1,0 g from the test sample and weigh to the nearest 0,1 mg. At the same time as the 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 B](#) may be used to prepare pre-dried test portions directly from the laboratory sample.

Obtain an approximate content for the copper, zinc and iron in the sample as required in [9.7.3](#).

Estimate the content for the copper, zinc, and iron in the sample by ICP-OES, AAS, XRF, titration, etc.

9.3 Blank test

Carry out a blank test in parallel with the analysis using the same quantities of all reagents but omitting the sample. The result of silver, in μg , in the blank test will be subtracted from the result of samples.

The purpose of the blank test is to check the atmosphere used in the test, apparatus contamination and reagent purity.

9.4 Dissolution of the test portion

9.4.1 General

Decompose the test portion in accordance with [9.4.2](#) or [9.4.3](#).

9.4.2 Dissolution of the test portion with nitric acid and hydrochloric acid

Quantitatively transfer the test portion into a 300 ml beaker. Add cautiously 20 ml of dilute nitric acid (5.2.2). Cover with a watch glass and leave the sample at room temperature for 30 min. Then place the beaker on the hotplate and heat slowly from room temperature to 90 °C. After the test portion is decomposed, increase the temperature of the hotplate to approximately 120 °C and expel oxides of nitrogen. Rinse the watch glass with a small amount of water and collect the washing solutions to the dissolved solutions. Remove the watch glass, increase the temperature of the hotplate to approximately 150 °C and evaporate to approximately 10 ml. Add 5 ml of hydrochloric acid (5.2.3), heat gently at a hotplate temperature of 120 °C to 150 °C and evaporate until the liquid volume is 1 ml to 2 ml. Remove the beaker from the hotplate.

NOTE 1 Rapid heating can cause the free sulfur to deposit, the free sulfur holding some of the silver ions.

In the case of free sulfur, which is deposited during gentle heating, dissolution should be carried out as described in 9.4.3.

NOTE 2 Silver oxide can form if heating is continued to the point of dryness of the solution. Silver oxide is difficult to dissolve by the procedure described in 9.5.

Cover with watch glass again. Cool and cautiously add 20 ml of dilute hydrochloric acid (5.2.5). Heat the solution to boiling, then cool to room temperature. Rinse the watch glass with dilute hydrochloric acid (5.2.5) and collect the washing solutions to the dissolved solutions, then remove the watch glass. Filter the solution through an insoluble filter paper (6.5) into a clean volumetric flask that is specified in 9.6. Thoroughly wash the 300 ml beaker used to digest the test portion and filter paper with dilute hydrochloric acid (5.2.5) and collect the washing solutions into the same volumetric flask.

If acid-insoluble material is present, then treat this residue as described in 9.5, otherwise proceed to 9.6.

9.4.3 Alternative dissolution of the test portion

Quantitatively transfer the test portion into a 300 ml beaker. Add cautiously 20 ml of dilute nitric acid (5.2.2). Cover with a watch glass and place the beaker on the hotplate and heat gently until all nitrogen oxides are expelled. Cautiously add 5 ml of perchloric acid (5.2.8). Continue heating until dense fumes of perchloric acid condense and drain down the sides of the beaker. Remove the watch glass and evaporate to dryness, then remove the beaker from the hotplate immediately.

Ensure that the temperature of the hotplate is between 200 °C and 220 °C.

NOTE Silver oxide can form if heating is continued even after the point of dryness of the solution. Silver oxide is difficult to dissolve by the procedure described in 9.5.

Cover with watch glass again. Cool and cautiously add 20 ml of dilute hydrochloric acid (5.2.5). Heat the solution to boiling, then cool to room temperature. Rinse the watch glass with dilute hydrochloric acid (5.2.5) and collect the washing solutions to the dissolved solutions, then remove the watch glass. Filter the solution through an insoluble filter paper (6.5) into a clean volumetric flask that is specified in 9.6. Thoroughly wash the 300 ml beaker used to digest the test portion and filter paper with dilute hydrochloric acid (5.2.5) and collect the washing solutions into the same volumetric flask.

If acid-insoluble material is present, then treat this residue as described in 9.5, otherwise proceed to 9.6.

9.5 Dissolution of the insoluble residue

Quantitatively, transfer the acid-insoluble residue into a 200 ml polytetrafluoroethylene beaker (6.4) with a small quantity of water. Add 5 ml of nitric acid (5.2.1), 5 ml of dilute sulfuric acid (5.2.7) and 3 ml to 5 ml of hydrofluoric acid (5.2.9). Heat the solution until enough white sulfuric acid gas is evolved to remove the silicon dioxide, then allow to cool. Add 20 ml of dilute hydrochloric acid (5.2.5) and cover the beaker with a watch glass. Heat the solution to boiling, then cool to room temperature. Rinse the watch glass and collect the washing solutions to the dissolved solutions.

If the insoluble material remains after this treatment, filter through an insoluble filter paper (6.5). Then wash the insoluble material and the filter paper with dilute hydrochloric acid (5.2.5), and collect the filtrate and the washing solution to the dissolved solutions. Reject insoluble material and the filter paper.

In cases where the absence of silver in the acid-insoluble residue is confirmed, the procedure of dissolution of the insoluble residue may be omitted.

Proceed to 9.6.

9.6 Preparation of test solutions

9.6.1 General

Prepare the test solutions in accordance with 9.6.2 or 9.6.3.

9.6.2 Atomic absorption spectrometer (AAS)

Quantitatively transfer the solution from 9.4.2 or 9.4.3 and 9.5 to volumetric flask of the volume specified in Table 1. Dilute to about 3/4 with dilute hydrochloric acid (5.2.5). Allow the solution to cool to room temperature, then dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

Table 1 — Volume of test solution for AAS

Silver content of sample g/t	Volume of volumetric flask ml
< 400	200
400 to 800	500

9.6.3 Inductively coupled plasma optical emission spectrometer (ICP-OES)

Quantitatively transfer the solution from 9.4.2 or 9.4.3 and 9.5 to a 200 ml volumetric flask and pipette 5,00 ml yttrium standard internal solution (5.3.8). Dilute to about 3/4 with dilute hydrochloric acid (5.2.5). Allow the solution to cool to room temperature, then dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

9.7 Preparation of the calibration solutions

9.7.1 General

Prepare the calibration solutions in accordance with 9.7.2 or 9.7.3.

9.7.2 Atomic absorption spectrometer (AAS)

Using working silver standard B (5.3.3), prepare a series of calibration solutions as per Table 2. Transfer the appropriate volumes of working standard solutions using pipettes, into each 200 ml volumetric flasks containing the following:

- Sufficient matrix/interference standard to match the major element in the concentrate being measured. For copper sulfide concentrates, use the copper matrix/interference standard (5.3.4). For zinc sulfide concentrates, use the zinc matrix/interference standard (5.3.5).
- Sufficient iron matrix/interference standard (5.3.6) to match the iron content of the samples.

Dilute to the mark with dilute hydrochloric acid (5.2.5) and mix well.

In cases where silver is determined using AAS, if the interference from the matrix has not appeared, the addition of the matrix/interference standard may be omitted.