
**Copper, lead, zinc and nickel sulfide
concentrates — Determination of
arsenic —**

Part 2:

**Acid digestion and inductively coupled
plasma atomic emission spectrometric
method**

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*Concentrés sulfurés de cuivre, de plomb et de zinc — Dosage de
l'arsenic*

*Partie 2: Méthode par digestion acide et plasma induit par haute
fréquence*



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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

ISO 13547 consists of the following parts, under the general title *Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic*:

- *Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method*
- *Part 2: Acid digestion and inductively coupled plasma atomic emission spectrometric method*

Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic —

Part 2:

Acid digestion and inductively coupled plasma atomic emission 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 an acid digestion and inductively coupled plasma atomic emission spectrometric (ICP-AES) method for the determination of the mass fraction of arsenic in copper, lead, zinc, and nickel sulfide concentrates as follows:

- a) for copper sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 2,0 %;
- b) for lead sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 1,0 %;
- c) for zinc sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 0,6 %;
- d) for nickel sulfide concentrates, the method is applicable to the determination of mass fraction of arsenic from 0,05 % to 1,0 %.

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 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 4787, *Laboratory glassware — 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 and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*

ISO 12743:2006, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

3 Principle

The test portion is decomposed in nitric and sulphuric acids and arsenic is determined by comparison against matrix-matched standards using ICP-AES.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled water or water of equivalent purity.

4.1 Sodium hydroxide (NaOH), AR grade.

4.2 Arsenic trioxide (As₂O₃), AR grade.

4.3 Iron(III) chloride hexahydrate (FeCl₃•6H₂O), AR grade (<0,000 05 % Arsenic).

4.4 Copper metal (≥99,99 %).

4.5 Zinc metal (≥99,99 %).

4.6 Lead metal (≥99,99 %).

4.7 Nickel metal (≥99,99 %).

4.8 Ammonium acetate, AR grade.

4.9 Nitric acid (ρ₂₀ 1,42 g/ml).

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4.10 Nitric acid, dilute (1 + 1).

Slowly add 50 ml of nitric acid (4.9) to 50 ml of water while stirring.

4.11 Hydrochloric acid (ρ₂₀ 1,16 g/ml).

4.12 Hydrochloric acid, dilute (1 + 1).

Slowly add 50 ml of hydrochloric acid (4.11) to 50 ml of water while stirring.

4.13 Sulphuric acid (ρ₂₀ 1,84 g/ml).

4.14 Sulphuric acid, dilute (1 + 1).

Slowly add 50 ml of sulphuric acid (4.12) to 50 ml of water while stirring.

The addition of sulphuric acid to water generates heat and shall be performed with adequate precautions.

4.15 Sulphuric acid, dilute (1 + 50).

Slowly and with stirring, add 20 ml of sulphuric acid (4.12) to 1 000 ml of water.

4.16 Hydrofluoric acid (ρ₂₀ 1,13 g/ml).

4.17 Sodium hydroxide solution (20 % w/v).

Weigh 20,0 g of sodium hydroxide (4.1) in a polytetrafluoroethylene beaker (5.5). Add 50 ml of water and allow the solid to dissolve. Dilute the solution to 100 ml and store in a labelled polyethylene container.

4.18 Saturated solution of potassium chlorate in nitric acid.

Superfluous potassium chlorate is added into the nitric acid and then the deposited solution is allowed to form the saturated solution.

4.19 Ammonium acetate (25 % w/v).

Dissolve 25,0 g of ammonium acetate (4.8) into 100 ml water.

4.20 Arsenic standard, 1 ml contains 1 mg of As.

Weigh 1 320,3 g of arsenic trioxide (4.2) into a 400 ml beaker and add 5 ml of sodium hydroxide solution (4.17). Warm slightly to dissolve. When dissolution is complete, cool and add 40 ml of nitric acid (4.9). Transfer to a 1 000 ml volumetric flask containing 60 ml of nitric acid (4.9). Fill up nearly to the mark with water, mix, and equilibrate at room temperature, then fill up exactly to the mark and mix again. Store in a labelled container.

Alternatively, purchase a suitable high quality prepared standard.

Before use, compare this International Standard against a traceable National Standard to ensure suitability for use.

4.21 Arsenic standard, 1 ml contains 0,2 mg of As.

Pipette 40 ml of arsenic standard (4.20) into a 200 ml volumetric flask containing 2 ml hydrochloric acid (4.11). Fill up nearly to the mark with water, mix, and equilibrate at room temperature, then fill up exactly to the mark and mix again. Store in a labelled glass container.

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

Dissolve 2,5 g of copper metal (4.4) with 50 ml of warm dilute nitric acid (4.10). Evaporate to approximately 25 ml in order to remove nitrogen oxides. Transfer to a 100 ml volumetric flask, add 5 ml concentrated hydrochloric acid, 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.

4.23 Zinc matrix/interference standard (32,5 mg/ml).

Dissolve 3,25 g of zinc metal (4.5) with 50 ml of warm dilute hydrochloric acid (1+1). Transfer to 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.

4.24 Iron matrix/interference standard (10 mg/ml).

Dissolve 48,4 g iron (III) chloride hexahydrate (4.3) in 200 ml of water. Add 10 ml of hydrochloric acid (4.11) and make up to 1 000 ml with water and mix.

4.25 Lead matrix/interference standard (37,5 mg/ml).

Dissolve 3,75 g of lead metal (4.6) by adding 40 ml of water and 20 ml of nitric acid (4.9) and heat to dissolve. Transfer to 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.

4.26 Nickel matrix/interference standard (10 mg/ml).

Dissolve 1,0 g of nickel metal (4.7) in 50 ml of dilute nitric acid (4.10) and heat until all nitrogen oxides are expelled. The volume will be about 25 ml. Transfer to a 100 ml volumetric flask. Add 25 ml of hydrochloric acid (4.11) and 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 Apparatus

All laboratory glassware and equipment shall be shown to be free of arsenic contamination.

5.1 **Balance**, sensitive to $\pm 0,1$ mg.

5.2 **Normal laboratory glassware**, complying with ISO 648 and ISO 1042 and used in accordance with ISO 4787.

5.3 **Inductively coupled plasma atomic emission spectrometer (ICP-AES)**.

The emission wavelength should be set to 193,696 nm or 197,197 nm.

5.4 **Insolubles filter paper**, Whatman®¹⁾ No. 40 or equivalent.

5.5 **Polytetrafluoroethylene beaker**, 200 ml capacity.

6 Sampling and sample preparation

6.1 Laboratory sample

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

As arsenic can evaporate from samples at elevated temperatures, consideration should be given to preparing a separate chemical analysis sample as described in ISO 12743:2006, 16.2.

6.2 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 to be used (see Annex A).

6.3 Test portion

Taking multiple increments, extract approximately 0,5 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 A may be used to prepare pre-dried test portions directly from the laboratory sample.

Obtain an approximate concentration for the copper, lead, zinc, and iron in the sample as required in step 7.7.

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO.

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 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 the same quantities of all reagents, but omitting the sample. The purpose of the blank test is to check for contamination of the apparatus used in the test and the purity of the reagents.

7.3 Dissolution of the test portion

Quantitatively, transfer the test portion into a 300 ml narrow-necked conical beaker. Moisten with 5 ml of water.

Add cautiously 15 ml of the saturated solution of potassium chlorate in nitric acid (4.18) and place the conical beaker on the hotplate and heat gently until all nitrogen oxides are expelled. Add 15 ml of dilute sulphuric acid (4.14) and heat the solution until strong white fumes have evolved, then cool.

If the residue appears dark (indicating the presence of carbon), slowly add a small amount of nitric acid (4.9) to the hot solution until the solution becomes colourless, then heat until strong white fumes have evolved.

Add 50 ml of water and bring to boil, then allow to cool at room temperature.

Filter the solution through an insoluble filter paper (5.4) in a 250 ml conical beaker. Wash the 300 ml conical beaker used to digest the sample and filter paper thoroughly with sulphuric acid (4.15) and collect the washing solutions in the same 250 ml conical beaker.

If acid insoluble material is present, then treat this residue as the procedure in 7.5, otherwise proceed to step 7.6.

In the case of lead concentrates, proceed to step 7.4.

7.4 Removal of lead sulfate

Quantitatively, transfer the precipitate into a 250 ml conical beaker with small amount of deionised water. Add 15 ml of ammonium acetate (4.19) and place the conical beaker on the hotplate and heat until dissolution of the PbSO_4 .

Cool the solution to room temperature and filter through an insoluble filter paper (5.4), then wash the insoluble residue with sulphuric acid (4.15). Reject the filtrate and washing solution. If acid insoluble material is present, then treat this residue as the procedure in 7.5, otherwise proceed to step 7.6.

7.5 Dissolution of the insoluble residue

Quantitatively, transfer the acid insoluble residue into a 200 ml polytetrafluoroethylene beaker (5.5) with a small quantity of deionised water. Add 5 ml of nitric acid (4.9), 5 ml of dilute sulphuric acid (4.14), and 3 ml to 5 ml of hydrofluoric acid (4.16). Heat the solution until the evolution of white sulphuric acid fumes to remove silicon dioxide. Allow to cool, then dissolve the soluble salts with 10 ml of deionised water and 10 ml of nitric acid (4.9). Proceed to step 7.6.