
**Copper, lead and zinc sulfide
concentrates — Determination of
cadmium —**

**Part 2:
Acid digestion and inductively
coupled plasma atomic emission
spectrometric method**

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*Concentrés de sulfure de cuivre, de plomb et de zinc — Dosage du
cadmium*

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*Partie 2: Méthode par digestion acide et spectroscopie d'émission
atomique à plasma à couplage inductif*



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Contents

	Page
Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Principle	2
5 Reagents	2
6 Apparatus	3
7 Sample	3
7.1 Laboratory sample.....	3
7.2 Test sample.....	3
7.3 Test portion.....	4
8 Procedure	4
8.1 Number of determinations.....	4
8.2 Blank test.....	4
8.3 Dissolution of the test portion.....	4
8.4 Preparation of test solutions.....	4
8.5 Preparation of calibration solutions.....	4
8.6 Preparation of cadmium calibration curve.....	5
8.7 Determination of cadmium content in test solutions.....	5
9 Expression of results	5
10 Precision	6
10.1 Expression of precision.....	6
10.2 Method for obtaining the final result.....	6
10.3 Between-laboratories precision.....	7
10.4 Check of trueness.....	7
10.4.1 General.....	7
10.4.2 Type of CRM or reference material.....	8
11 Test report	8
Annex A (informative) Procedure for the preparation and determination of the mass of a pre-dried test portion	9
Annex B (normative) Procedure for obtaining the final result	11
Bibliography	12

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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

A list of all parts in the ISO 19976 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Copper, lead and zinc sulfide concentrates — Determination of cadmium —

Part 2: Acid digestion and inductively coupled plasma atomic emission spectrometric method

WARNING — The use of this document might involve hazardous materials, operations and equipment. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

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

- a) for copper sulfide concentrates, the method is applicable to the determination of mass fractions of cadmium from 0,01 % to 0,30 %;
- b) for lead sulfide concentrates, the method is applicable to the determination of mass fractions of cadmium from 0,01 % to 0,30 %;
- c) for zinc sulfide concentrates, the method is applicable to the determination of mass fractions of cadmium from 0,05 % to 1,00 %.

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 385, *Laboratory glassware — Burettes*

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, 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*

ISO Guide 35, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

4 Principle

The test sample is dissolved in hydrochloric acid, nitric acid and hydrofluoric acid with a final stage perchloric acid digestion. Yttrium is added as an internal standard to solutions of dissolved samples, reference materials and calibration solutions and cadmium determined by ICP-AES.

5 Reagents

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

5.1 Hydrochloric acid, concentrated (ρ_{20} 1,19 g/ml).

5.2 Hydrochloric acid, dilute 1+1. Slowly add 500 ml of concentrated hydrochloric acid (5.1) to 500 ml of water while stirring.

5.3 Hydrochloric acid, dilute 1+9. Slowly add 50 ml of concentrated hydrochloric acid (5.1) to 450 ml of water while stirring.

5.4 Nitric acid, concentrated (ρ_{20} 1,42 g/ml).

5.5 Hydrofluoric acid, concentrated (ρ_{20} 1,15 g/ml).

5.6 Perchloric acid, concentrated (ρ_{20} 1,70 g/ml).

5.7 Cadmium metal, minimum purity 99,99 %.

5.8 Cadmium, standard solution, 1 ml contains 1 mg of Cd.

Weigh, to the nearest 0,1 mg, 1,000 0 g of cadmium metal (5.7) into a 250 ml beaker. Add 10 ml of water and 5 ml of nitric acid (5.4). Cover and warm gently (if necessary) until the cadmium metal is dissolved. Heat to remove nitrogen oxides, then allow to cool, and add about 50 ml of water. Transfer the solution to a 1 000 ml one-mark volumetric flask, add 40 ml of hydrochloric acid (5.2), fill up nearly to the mark with water, mix and cool to room temperature. Then fill up exactly to the mark with water and mix again.

5.9 Cadmium, standard solution, 1 ml contains 100 µg of Cd.

Pipette 10,00 ml of cadmium standard solution (5.8) into a 100 ml one-mark volumetric flask. Add 20 ml of hydrochloric acid (5.2), fill up nearly to the mark with water, mix and cool to room temperature. Then fill up exactly to the mark with water and mix again.

5.10 Cadmium, standard solution, 1 ml contains 10 µg of Cd.

Pipette 10,00 ml of cadmium standard solution (5.9) into a 100 ml one-mark volumetric flask. Add 18 ml of hydrochloric acid (5.2), fill up nearly to the mark with water, mix and cool to room temperature. Then fill up exactly to the mark with water and mix again.

5.11 Yttrium, standard solution, 1 ml contains 1 mg of Y.

Weigh, to the nearest 0,1 mg, 1,269 9 g of yttrium trioxide (≥99,99 %) and transfer to a 250 ml beaker. Add 100 ml of hydrochloric acid (5.3) and heat gently until the metal is dissolved, then cool to room temperature. Transfer to a 1 000 ml one-mark volumetric flask, fill up nearly to the mark with hydrochloric acid (5.3), mix and cool to room temperature, then fill up exactly to the mark with hydrochloric acid (5.3) and mix again.

5.12 Yttrium, standard solution, 1 ml contains 200 µg of Y.

Pipette 50,00 ml of yttrium standard solution (5.11) into a 250 ml one-mark volumetric flask. Add 40 ml of hydrochloric acid (5.2), fill up nearly to the mark with water, mix and cool to room temperature, then fill up exactly to the mark with water and mix again.

6 Apparatus

Ordinary laboratory equipment and the following.

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

It is permissible to use other measuring instruments, approved types, auxiliary devices and materials whose technical and metrological characteristics are not inferior to those mentioned above.

6.2 Polytetrafluoroethylene beaker, of capacity 200 ml, or glass-carbon beaker.

6.3 Analytical balance, sensitive to 0,1 mg.

6.4 ICP-AES. The emission wavelength should be set to 226,502 nm.

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

7 Sample

7.1 Laboratory sample

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

7.2 Test sample

Prepare an air-equilibrated 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).

1) This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

7.3 Test portion

Taking multiple increments, extract approximately 0,50 g from the test sample and weigh to the nearest 0,1 mg. At the same time as 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](#) can be used to prepare pre-dried test portions directly from the laboratory sample.

8 Procedure

8.1 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.

8.2 Blank test

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

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8.3 Dissolution of the test portion

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Transfer the test portion into a 200 ml polytetrafluoroethylene beaker (6.2) and moisten with 5 ml of water.

Add 10 ml of hydrochloric acid (5.1), and heat for 5 min at 60 °C to 70 °C. Add 10 ml of nitric acid (5.4) and heat gently. Add 3 ml of hydrofluoric acid (5.5), then heat gradually until the solution is about 5 ml at 60 °C to 70 °C. Add 3 ml of perchloric acid (5.6), cover with a polytetrafluoroethylene lid, heat the solution until the strong white fumes evolve, then remove from the hotplate and cool. Wash the polytetrafluoroethylene lid and sides of the beaker with a minimum volume of water and add 2 ml of perchloric acid (5.6). Continue heating until the strong white fumes have evolved completely, then cool.

Add 40 ml of hydrochloric acid (5.2) [for lead concentrate samples, add 50 ml of hydrochloric acid (5.3)]. Wash down the cover and sides of the beaker, heat until the onset of boiling, then allow to cool.

8.4 Preparation of test solutions

Transfer the solution (8.3) and pipette 5,00 ml yttrium standard solution (5.12) to a 200 ml one-mark volumetric flask, dilute to the mark with water and mix thoroughly. For lead concentrate samples, filter the solution through an insoluble filter paper (6.5), then wash the precipitate with hydrochloric acid (5.3). Collect the filtrate in a 200 ml one-mark volumetric flask, and pipette 5,00 ml yttrium standard solution (5.12), dilute to the mark with hydrochloric acid (5.3) and mix thoroughly.

8.5 Preparation of calibration solutions

Using cadmium standard, prepare a series of calibration standards as per [Table 1](#). Transfer 0,0 ml and 2,00 ml of cadmium standard (5.10) and 1,00 ml, 5,00 ml, 10,00 ml, 20,00 ml and 30,00 ml of cadmium standard (5.9), then transfer 2,50 ml yttrium standard solution (5.12) into 100 ml volumetric flasks, dilute to the mark with hydrochloric acid (5.3) and mix thoroughly.

Table 1 — Preparation of calibration solutions^a

Concentration of cadmium standard µg/ml	Volume of cadmium standard ml	Concentration of cadmium µg/ml	Mass of cadmium contained µg
10	0,00	0,00	0
10	2,00	0,20	20
100	1,00	1,00	100
100	5,00	5,00	500
100	10,00	10,00	1 000
100	20,00	20,00	2 000
100	30,00	30,00	3 000

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

8.6 Preparation of cadmium calibration curve

Set up the ICP-AES (6.4) according to the guidelines set out in 6.4. Adjust the instrument read-out scale to zero. Aspirate each calibration solution, as prepared in 8.5, through the ICP-AES and record the emission intensities. Manually or electronically plot a graph of the average emission intensities versus mass of cadmium (in µg) and repeat errant standards if necessary.

Use ISO 8466-2 as a guide to determine the acceptability of the calibration curve.

8.7 Determination of cadmium content in test solutions

Immediately after calibrating the ICP-AES (6.4), determine the cadmium content in the test solutions from 8.5. Adjust the instrument read-out scale to zero. Aspirate the first duplicate of each sample through the ICP-AES and record the emission intensities, checking the zero regularly.

Check the calibration of the instrument, then aspirate the second duplicate of each sample in the reverse order through the ICP-AES and record the emission intensities, checking the zero regularly.

Determine the mass of cadmium in each test sample (F_{Cd} , in µg) from the curve determined in 8.7.

9 Expression of results

The mass fraction of cadmium in the test portion, w_{Cd} , expressed as a percentage, is given in Formula (1):

$$w_{Cd} = \frac{(F_{Cd} - F_{Cdblank})}{m \times 10^6} \times \frac{100}{100 - H} \times 100 \quad (1)$$

where

F_{Cd} is the mass of cadmium found in the test sample aliquot, in µg;

$F_{Cdblank}$ is the mass of cadmium found in the blank test, in µg;

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

H is the hygroscopic moisture content of the sample, expressed as a percentage, determined using ISO 9599.

NOTE If pre-dried test samples are used, $H = 0$.