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

Part 1:

Flame atomic absorption spectrometric method

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

Partie 1: Spectrométrie d'absorption atomique dans la flamme

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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).

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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*.

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 1:
Flame atomic absorption 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 a flame atomic absorption spectrometric 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 0,30 %.

2 Normative reference

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 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

Dissolve the test portion in hydrochloric acid and nitric acid. Add hydrofluoric acid to remove silicon. Then add perchloric acid and determine the iron concentration in the test portion using an atomic absorption spectrometer (AAS) with a deuterium lamp for background correction at 228,8 nm.

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, diluted 1+1.

Slowly add 500 ml of concentrated hydrochloric acid (5.1) to 500 ml of water, while stirring.

5.3 Hydrochloric acid, diluted 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 Cadmium calibration solutions.

Prepare a series of calibration solutions by adding, from a pipette, 0,0 ml, 2,00 ml, 4,00 ml, 6,00 ml, 7,00 ml, 8,00 ml, 9,00 ml and 10,00 ml of cadmium standard solution (5.10) into a series of 100 ml one-mark volumetric flasks. Dilute to the mark with hydrochloric acid (5.3) and mix thoroughly.

These standards contain 0,0 µg, 0,2 µg, 0,4 µg, 0,6 µg, 0,7 µg, 0,8 µg, 0,9 µg and 1,0 µg of cadmium 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.

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 AAS, equipped with a cadmium hollow cathode lamp and deuterium lamp for background correction.

Recommended instrument conditions:

- flame: air-acetylene;
- wavelength: 228,8 nm;
- slit width: 0,2 nm.

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

7 Samples

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).

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[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.

8.3 Determination

8.3.1 Dissolution of test portion

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 perchloric acid (5.6) repeatedly until the residue is not present. Heat until the strong white fumes have exhausted completely, then allow to 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 and then heat until the onset of boiling, then allow to cool.

8.3.2 Preparation of test solutions

Transfer the solution (8.3.1) 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)]. Wash the precipitate with hydrochloric acid (5.3) and collect the filtrate in a 200 ml one-mark volumetric flask. Dilute to the mark with hydrochloric acid (5.3) and mix thoroughly. Then transfer an appropriate aliquot of this solution (see Table 1) to the corresponding one-mark volumetric flask. Add an appropriate amount of hydrochloric acid 5.2) as indicated in Table 1, 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.

The dilutions shown in Table 1 will provide concentrations of cadmium falling within the range of the calibration solutions.

Table 1 — Dilution guide for test solution

Expected mass fraction of cadmium in sample w_{Cd}/%	Aliquot to take V_1/ml	Hydrochloric acid (5.2) to be added ml	Volume after dilution V_2/ml
$0{,}01 \leq w_{Cd} \leq 0{,}03$	–	–	–
$0{,}03 < w_{Cd} \leq 0{,}08$	20,00	6,0	50
$0{,}08 < w_{Cd} \leq 0{,}30$	10,00	18,0	100

8.3.3 Adjustment of the AAS

Fit the cadmium hollow-cathode lamp into the apparatus (6.4), switch on the current and allow to stabilize. Adjust the current, the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the wavelength in the region of 228,8 nm to obtain maximum absorbance. Adjust the pressure of air and acetylene according to the characteristics of the aspirator burner.

8.3.4 Atomic absorption measurements

Aspirate the calibration solutions (5.11) and the final test solution (see 8.3.2) into the AAS (6.4) and measure the absorbance. As a guide, the instrument should be as near as is practical to a linear relationship between absorbance and concentration.

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 cadmium.

8.4 Check test

Make a preliminary check of the apparatus by preparing a solution of standard material or a synthetic sample containing a known amount of cadmium and of composition similar to the material to be analysed, and carry out the procedure as specified in 8.3.

9 Expression of results

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

$$w_{Cd} = \frac{(\rho - \rho_0) \cdot V \cdot V_2}{m \cdot V_1 \times 10^6} \times \frac{100}{100 - H} \times 100 \qquad (1)$$

where

ρ is the concentration, in micrograms per millilitre, of cadmium in the test solution obtained from the calibration curve;

ρ_0 is the concentration, in micrograms per millilitre, of cadmium in the blank solution;

V is the total volume, in millilitres, of test solution before transferring the aliquot solution;

V_1 is the volume, in millilitres, of aliquot solution to be transferred;