

SLOVENSKI STANDARD

SIST ISO 13658:2001

01-junij-2001

Prečiščeni koncentri sulfidov železa -- Določanje vsebnosti železa -- Hidroksidna precipitacija in EDTA titrimetrična metoda

Zinc sulfide concentrates -- Determination of zinc content -- Hydroxide precipitation and EDTA titrimetric method

Standard Preview

Concentrés sulfurés de zinc -- Dosage du zinc -- Méthode par précipitation d'hydroxydes et titrage à l'EDTA

[SIST ISO 13658:2001](https://standards.iteh.ai/catalog/standards/sist/c16f11c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001)

Ta slovenski standard je istoveten z:

ISO 13658:2000

ICS:

73.060.99

Druge rude

Other metalliferous minerals

SIST ISO 13658:2001

en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 13658:2001

<https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001>

INTERNATIONAL STANDARD

ISO
13658

First edition
2000-03-15

Zinc sulfide concentrates — Determination of zinc content — Hydroxide precipitation and EDTA titrimetric method

*Concentrés sulfurés de zinc — Dosage du zinc — Méthode par
précipitation d'hydroxydes et titrage à l'EDTA*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST ISO 13658:2001](https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001)

[https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-
1426c2735bbe/sist-iso-13658-2001](https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001)



Reference number
ISO 13658:2000(E)

© ISO 2000

ISO 13658:2000(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST ISO 13658:2001](https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001)

<https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001>

© ISO 2000

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 734 10 79
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

Foreword.....	iv
1 Scope	1
2 Normative references	1
3 Principle	2
4 Reagents	2
5 Apparatus	3
6 Sample	3
7 Procedure	4
8 Expression of results	6
9 Application to the trading of zinc concentrates	7
10 Accuracy	7
11 Test report	8
Annex A (informative) An examination of potentially interfering elements	9
Annex B (normative) Procedure for the preparation and determination of the mass of a predried test portion.....	10
Annex C (normative) Determination of zinc content of acid-insoluble and hydroxide precipitation residues by atomic absorption spectroscopy	12
Annex D (normative) Determination of cadmium by atomic absorption spectrometry.....	15
Annex E (normative) Flowsheet of the procedure for the acceptance of analytical values for test samples.....	18
Annex F (informative) Derivation of precision equations	19

ISO 13658:2000(E)

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13658 was prepared by Technical Committee ISO/TC 183, *Copper, lead and zinc ores and concentrates*.

Annexes B, C, D and E form a normative part of this International Standard. Annexes A and F are for information only.

ITEH STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 13658:2001
<https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001>

Zinc sulfide concentrates — Determination of zinc content — Hydroxide precipitation and EDTA titrimetric 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 a hydroxide precipitation and EDTA titrimetric method for the determination of the zinc content of zinc ores and concentrates containing 10 % (m/m) to 60 % (m/m) zinc.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1998, *Laboratory glassware — One-mark volumetric flasks*.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*.

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*.

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*.

ISO 9599:1991, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*.

ISO 12739:1997, *Zinc sulfide concentrates — Determination of zinc content — Ion-exchange/EDTA titrimetric method*.

ISO 13291:1997, *Zinc sulfide concentrates — Determination of zinc content — Solvent extraction and EDTA titrimetric method*.

ISO/Guide 35:1989, *Certification of reference materials — General and statistical principles*.

ISO 13658:2000(E)

3 Principle

Dissolution of a test portion of zinc concentrate in bromine and nitric acid. Dissolution of any remaining insoluble residue in hydrofluoric and sulfuric acids. Separation of aluminium, iron and manganese as hydroxides using precipitation with ammonia, ammonium chloride and ammonium persulfate. Separation of lead as sulfate. Copper is masked. Determination of zinc and cadmium together by complexometric titration (pH 5,5 to 5,7) in the presence of xylene orange. Independent determination of cadmium and subtraction from the zinc value.

NOTE An examination of potential interfering elements is contained in annex A.

4 Reagents

During the analysis, use only reagents of recognized analytical reagent grade and water that complies with grade 2 of ISO 3696.

4.1 Zinc metal, minimum purity 99,99 %

The surface of the metal shall be free from oxide prior to use and may be cleaned by immersing the metal in hydrochloric acid (4.6) for 1 min, washing well with water followed by an acetone rinse and dried in an oven at 50 °C.

4.2 Ammonium chloride

4.3 Bromine

4.4 Nitric acid (ρ_{20} 1,42 g/ml)

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

4.6 Hydrochloric acid (1+1)

iTeh STANDARD PREVIEW
(standards.iteh.ai)
SIST ISO 13658:2001
<https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-1426c2735bbe/sist-iso-13658-2001>

Add 500 ml of hydrochloric acid (4.5) to 500 ml of water.

4.7 Sulfuric acid (ρ_{20} 1,84 g/ml)

4.8 Dilute sulfuric acid (1+1)

Add carefully and slowly with stirring, 500 ml of sulfuric acid (4.7) to 500 ml of water.

4.9 Sulfuric acid (1 + 99)

Add 20 ml of sulfuric acid (4.8) to 1 000 ml of water.

4.10 Ammonia (ρ_{20} 0,89 g/ml)

4.11 Ammonium persulfate

4.12 Ammonia washing solution

Dissolve 20 g of ammonium chloride in 1 000 ml of aqueous ammonia solution (1 + 100).

4.13 Ethanol, pure or denatured

4.14 Acetic acid (ρ_{20} 1,05 g/ml)

4.15 Bromothymol blue indicator solution (1 g/l)

Dissolve 0,1 g of bromothymol blue in ethanol (4.13) and dilute to 100 ml.

4.16 Hexamethylenetetramine buffer solution (250 g/l)

Dissolve 250 g of hexamethylenetetramine (hexamine) in water. Add 60 ml of acetic acid (4.14) and dilute to 1 l.

4.17 Masking solution

Dissolve 50 g of ammonium fluoride and 100 g of sodium thiosulfate pentahydrate in water and dilute to 1 l.

4.18 Xylenol orange indicator solution (1 g/l)

Dissolve 0,1 g of the sodium salt of xylenol orange in water and dilute to 100 ml.

4.19 Iron(III) solution (10 mg Fe/ml)

Dissolve 72,3 g of iron(III) nitrate nonahydrate in water and dilute to 1 l.

4.20 EDTA standard solution (0,1 mol/l)

Dissolve 37,2 g of the di-sodium salt of ethylenediaminetetraacetic acid and 2 g of sodium hydroxide in water and dilute to 1 l.

iTeh STANDARD PREVIEW (standards.iteh.ai)

5 Apparatus

5.1 Class A volumetric glassware, complying with ISO 385-1, ISO 648 and ISO 1042 and used in accordance with ISO 4787.

[SIST ISO 13658:2001](https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-0416c2735bbe/sist-iso-13658-2001)

[https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-](https://standards.iteh.ai/catalog/standards/sist/c16fd1c2-5146-4d92-beb5-0416c2735bbe/sist-iso-13658-2001)

5.2 Balance, capable of being read to 0,1 mg.

5.3 Laboratory hotplate**5.4 pH meter****6 Sample****6.1 Test sample**

Prepare an air-equilibrated test sample in accordance with ISO 9599.

NOTE An air-equilibrated test sample is not required if predried test portions are to be used (see annex B).

6.2 Test portion

Taking multiple increments, extract a test portion of 0,5 g from the test sample and weigh to the nearest 0,1 mg. At the same time as the test portions are taken 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 predried test portions directly from the laboratory sample.

ISO 13658:2000(E)

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 Reagent blank

A reagent blank shall be determined. It is advisable to perform duplicate blank determinations every time an analysis is carried out on a laboratory sample. The blank samples are carried through the whole procedure, with the exception that no laboratory sample test portion is required. The average volume of EDTA titrant used for the reagent blanks is V_b .

7.3 Dissolution

Digest the sample as follows.

Transfer the test portion (6.2) to a 300 ml conical beaker. Moisten the material with about 5 ml of water and add 1 ml of bromine (4.3). Stand for 15 min, swirling the beaker and contents from time to time.

Cautiously add 15 ml of nitric acid (4.4). Place the beaker on a hotplate (5.3) and heat gradually until bromine and nitrogen oxides are expelled. Add 15 ml of sulfuric acid (4.8). Heat and evaporate the solution until the evolution of sulfuric acid fumes (approximately 5 ml).

NOTE Care needs to be taken to ensure no loss of material when fuming samples containing significant carbonaceous material, due to the sample climbing up the walls of the beaker.

Cool and cautiously add about 50 ml of water and heat the solution until boiling. Cool the solution to room temperature and filter it into a 500 ml conical beaker through a medium-speed cellulose filter paper. Wash the beaker and filter paper thoroughly with sulfuric acid (4.9). Collect the filtrate and washing solution in the same beaker.

Reserve the filter paper and acid-insoluble residue for determination of zinc by FAAS (as described in annex C) for the concentrate being analysed and the analyst performing the test, unless it has been statistically shown, by previous testing, that the zinc is completely soluble in the initial decomposition. If this step is not routinely performed, regular checks confirming the validity of its exclusion shall be carried out for each concentrate type by each analyst. Similarly, all documentation containing results where this step has been excluded shall contain a comment indicating this fact.

7.4 Separation of aluminium, iron and manganese as hydroxides

Add 15 g of ammonium chloride (4.2) to the solution and swirl gently to dissolve.

NOTE 1 If the iron content of the sample is only a few percent, then iron(III) solution (4.19) is added to the solution to increase the contained iron in solution to about 50 mg.

Heat to near boiling then add ammonia (4.10) to the solution until complete precipitation occurs and then 30 ml in excess. Add 1 g of ammonium persulfate (4.11) to the solution, heat until boiling and continue boiling for 1 min. Stand to allow the precipitate to settle out. Filter into another 500 ml beaker through a medium-speed cellulose filter paper and wash the precipitate several times with hot ammonia washing solution (4.12). Collect the filtrate and the washing solutions in the same beaker.

NOTE 2 Filtration will be slow for high lead content samples.

Wash the precipitate back into the original beaker without damaging the filter paper and dissolve it with 10 ml of hydrochloric acid (4.6). Add 5 g of ammonium chloride (4.2) to the solution. Repeat the precipitation by addition of ammonia (4.10) and ammonium persulfate (4.11). Boil, filter through the original filter paper and wash in the same manner as described above.

Collect the filtrate and washing solution in the same beaker used to collect the filtrate for the first separation and place the beaker on a hotplate (5.3). Heat gently and evaporate the solution to about 200 ml. Cool to room temperature.

Keep the hydroxide precipitate for later treatment.

7.5 Titration

7.5.1 Determination of the titration factor of the EDTA solution

NOTE 1 In order to obtain a relative accuracy of between 0,1 % and 0,2 %, it is necessary to standardize the EDTA solution with zinc at the same time and under the same conditions as the analysis. It is thus advisable for the calibration to follow the complete set of operating conditions set down for the analysis. Likewise, to improve the repeatability of the calibration, it is useful to prepare several zinc reference solutions.

The EDTA solution should be standardized as follows:

Weigh into three separate 500 ml beakers between 0,055 g and 0,31 g of high-purity zinc metal (4.1), to the nearest 0,1 mg, depending on the zinc content of the test sample. Record these masses as m_1 , m_2 and m_3 .

Add 10 ml of hydrochloric acid (4.6) and warm gently to aid the complete dissolution of the metal. Add 50 ml of water and 20 g of ammonium chloride (4.2), swirl gently to dissolve and dilute to about 200 ml with water.

Add 4 drops of bromothymol blue indicator solution (4.15) and neutralize by adding ammonia (4.10) dropwise until the yellow colour disappears. Add 7 drops of hydrochloric acid (4.5).

NOTE 2 A pH meter (5.4) may be used to adjust the pH to a value of 5,6 instead of using the bromothymol blue indicator. Care should be taken to ensure that the electrodes are washed off with water before continuing the procedure.

Add 20 ml of hexamethylenetetramine buffer solution (4.16) and 20 ml of masking solution (4.17). Add 10 drops of xylenol orange indicator (4.18) to the solution. Swirl to dissolve and titrate with EDTA standard solution (4.20) until a colour change to yellow occurs. Record the volumes as V_1 , V_2 and V_3 .

Calculate the intermediate factors, f_{i_x} , for each beaker using the following equation:

$$f_{i_x} = m_x / V_x \text{ for } x = 1 \text{ to } 3 \quad (1a)$$

where

f_{i_x} is the factor obtained from the titration;

m_x is the mass of zinc weighed, in grams;

V_x is the volume of EDTA solution, in millilitres.

If the range of values for f_{i_1} , f_{i_2} and f_{i_3} exceeds 0,000 01 g/ml, then repeat the standardization.

Otherwise calculate the mean factor as follows:

$$f = (f_{i_1} + f_{i_2} + f_{i_3}) / 3 \quad (1b)$$