

Australian Standard™

Zinc sulfide concentrates—Chemical analysis

Part 3: Determination of zinc content—Hydroxide precipitation and EDTA titrimetric method

[ISO title: Zinc sulfide concentrates—Determination of zinc content—Hydroxide precipitation and EDTA titrimetric method]

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Australasian Institute of Mining and Metallurgy

CSIRO Minerals

Minerals Council of Australia

The Royal Australian Chemical Institute

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RECONFIRMATION

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Zinc sulfide concentrates—Chemical analysis

Part 3: Determination of zinc content—Hydroxide precipitation and EDTA

titrimetric method

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NOTES

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PREFACE

This Standard was prepared by the Standards Australia Committee MN-005, Copper, Lead, Zinc, Gold and Silver Ores and Concentrates as part of a programme of standardizing methods for the determination of elements of commercial interest in such materials.

The objective of this Standard is to provide those involved in the analysis of zinc sulfide concentrates with a standardized method of determining gold content supported by precision data obtained from an inter-laboratory test programme.

This Standard is identical with and has been reproduced from ISO 13658:2000, *Zinc sulfide concentrates—Determination of zinc content—Hydroxide precipitation and EDTA titration method*, which has been prepared by ISO/TC 183 Copper, Lead and Zinc Ores and Concentrates. Australia holds the Chairmanship and Secretariat of ISO/TC 183 and has made a significant contribution to the preparation of ISO 13658.

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References to International Standards should be replaced by references to equivalent Australian or Australian/New Zealand Standards, as follows:

<i>Reference to International Standard</i>		<i>Australian/New Zealand Standard</i>	
ISO		AS	
9599	Copper, lead and zinc sulfide concentrates—Determination of hygroscopic moisture in the analysis sample—Gravimetric method	2816	Copper, lead and zinc sulfide concentrates—Determination of hygroscopic moisture in the analysis sample—Gravimetric method
385	Laboratory glassware—Burettes	—	
385-1	Part 1: General requirements	—	
648	Laboratory glassware—One-mark pipettes	—	
1042	Laboratory glassware—One-mark volumetric flasks	—	
3696	Water for analytical laboratory use—specification and test methods	—	
4787	Laboratory glassware—Volumetric glassware—Methods for use and testing of capacity	—	
5725-2	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		AS	
12739	Zinc sulfide concentrates— Determination of zinc content— Ion- exchange/EDTA titrimetric method	2678	Zinc sulfide concentrates— Chemical analysis
		2678.2	Part 2: Determination of zinc content—Ion-exchange method
13291	Zinc sulfide concentrates— Determination of zinc content— Solvent extraction and EDTA titrimetric method	2678.1	Part 1: Determination of zinc content—Solvent extraction and EDTA titrimetric method
Guide 35	Certification of reference materials— General and statistical principles	—	—

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AUSTRALIAN STANDARD

Zinc sulfide concentrates—Chemical analysis**Part 3: Determination of zinc content—Hydroxide precipitation and EDTA titrimetric method****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*.

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)

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.

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.

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.

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.