

Australian Standard[®]

Zinc sulfide concentrates—Chemical analysis

Part 1: Determination of zinc—Solvent extraction and EDTA titrimetric method



This Australian Standard® was prepared by Committee MN-005, Copper, Lead, Zinc and Nickel Ores and Concentrates. It was approved on behalf of the Council of Standards Australia on 24 January 2008.
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- CSIRO Minerals
- Minerals Council of Australia

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- Minerals industry analytical laboratories
-

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Part 1: Determination of zinc—Solvent extraction and EDTA titrimetric method

Originated as AS 2678.1—1984.
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PREFACE

This Standard was prepared by the Standards Australia Committee MN-005, Copper, Lead, Zinc and Nickel Ores and Concentrates, to supersede AS 2678.1—1999.

The objective of this Standard is to provide those involved in the analysis of zinc concentrates with a standardized method for determining zinc, supported by precision data obtained from an interlaboratory test program.

The objective of this revision is to adopt the latest edition of the corresponding International Standard.

This Standard is identical with, and has been reproduced from ISO 13291:2006, *Zinc sulfide concentrates—Determination of zinc—Solvent extraction and EDTA titrimetric method*.

As this Standard is reproduced from an International Standard, the following applies:

- (a) Its number appears on the cover and title page while the International Standard number appears only on the cover.
- (b) In the source text ‘this International Standard’ should read ‘this Australian Standard’.
- (c) A full point substitutes for a comma when referring to a decimal marker.
- (d) Substitute ‘L’ for ‘l’ as the unit for litres.

References to International Standards should be replaced by references to Australian Standards, as follows:

<i>Reference to International Standard</i>	<i>Australian 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

Only international references that have been adopted as Australian Standards have been listed.

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the annex to which they apply. A ‘normative’ annex is an integral part of a Standard, whereas an ‘informative’ annex is only for information and guidance.

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AUSTRALIAN STANDARD

Zinc sulfide concentrates—Chemical analysis**Part 1:****Determination of zinc—Solvent extraction 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 solvent extraction/titrimetric method for the determination of the mass fraction of zinc in zinc sulfide concentrates.

The method is applicable to zinc sulfide concentrates having a mass fraction of zinc in the range from 11 % to 62 %.

2 Normative references

The following referenced documents are indispensable for the application 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 — One-mark pipettes*

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

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

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

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

ISO Guide 35, *Reference materials — General and statistical principles for certification*

3 Principle

A test portion of zinc concentrate is dissolved in bromine and nitric acid. Any remaining insoluble residue is dissolved in sulfuric, nitric and hydrofluoric acids. A zinc thiocyanate complex is selectively extracted into methyl isobutyl ketone, after screening of some elements with thiourea and citrate ions. Partially extracted cadmium is screened with iodide ions before the final titration as explained in Annex C. Cobalt is extracted and determined separately, if present at concentration levels exceeding 0,05 %. Zinc is determined by titration with EDTA solution at pH 5,5.

4 Reagents

During the analysis, only reagents of recognized analytical grade and water that complies with grade 2 of ISO 3696 shall be used.

4.1 Zinc metal, minimum purity 99,99 %, free from oxide prior to use.

The surface of the metal may be cleaned by immersing the metal in hydrochloric acid (4.2), diluted 1 + 9 for 1 min, then washing well with water, followed by an acetone rinse and drying in an oven at 50 °C.

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

4.3 Bromine.

4.4 Nitric acid, (ρ_{20} 1,42 g/ml).

4.5 Hydrofluoric acid, (ρ_{20} 1,13 g/ml to 1,15 g/ml).

4.6 Hydrochloric acid, (ρ_{20} 1,16 g/ml to 1,19 g/ml) diluted (1 + 4).

Add 200 ml of hydrochloric acid (4.2) to 800 ml of water.

4.7 Sulfuric acid, diluted (1 + 1).

Add carefully and slowly, with stirring, 500 ml of sulfuric acid (ρ_{20} 1,84 g/ml) to 500 ml of water.

4.8 Ammonia, (ρ_{20} 0,89 g/ml).

4.9 4-methyl-2-pentanone (methyl isobutyl ketone).

4.10 Ethanol, pure or denatured.

4.11 Screening solution.

Dissolve 60 g of thiourea, 100 g of di-ammonium citrate and 200 g of ammonium thiocyanate in water and dilute to 1 l. Filter if necessary.

4.12 Sodium fluoride solution (20 g/l).

Dissolve 20 g of sodium fluoride in water. Dilute to 1 l.

4.13 Thiourea solution (100 g/l).

Dissolve 100 g of thiourea in water and dilute to 1 l.

4.14 Buffer solution.

Dissolve 250 g of hexamethylenetetramine in water. Add 60 ml of acetic acid (ρ_{20} 1,05 g/ml) and dilute to 1 l.

4.15 EDTA standard volumetric solution (0,05 mol/l).

Dissolve 18,6 g of the di-sodium salt of ethylenediaminetetraacetic acid dihydrate (EDTA) in water. Dilute to 1 l.

4.16 Potassium iodide solution (1 000 g/l).

Dissolve 100 g of potassium iodide in water and dilute to 100 ml.

Prepare fresh on the day of use.

4.17 Xylenol orange indicator (1 % *m/m*).

Mix 1 g of the sodium salt of xylenol orange with 99 g of potassium nitrate crystals, by gently grinding in a ceramic mortar with a pestle. Mixing is considered complete when the colour is uniform throughout.

4.18 Iron stock solution.

Dissolve 45 g of iron(III) nitrate nonahydrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ in water and dilute to 1 l.

5 Apparatus

Ordinary laboratory equipment and the following.

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

5.2 Platinum crucibles, of 25 ml capacity.

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

5.4 Oven, with its temperature controlled at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

5.5 Muffle furnace, having a maximum required operating temperature higher than $800\text{ }^{\circ}\text{C}$.

5.6 Laboratory hotplate.

6 Sample

6.1 Test sample

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

NOTE A test sample is not required if predried test portions are to be used (see Annex A).

6.2 Test portion

Taking multiple increments, extract a test portion from the test sample in such a manner that it is representative of the dish or tray. Weigh, to the nearest 0,1 mg, 2,5 g of test sample. At the same time as the test portion is weighed, 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 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.