

Zinc sulfide concentrates — Determination of zinc — Ion-exchange/EDTA titrimetric method

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National foreword

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Summary of pages

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Zinc sulfide concentrates — Determination of zinc — Ion-exchange/EDTA titrimetric method

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



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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 12739 was prepared by Technical Committee ISO/TC 183, *Copper, lead, zinc and nickel ores and concentrates*.

This second edition cancels and replaces the first edition (ISO 12739:1997), which has been technically revised.

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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 an ion-exchange/EDTA titrimetric method for the determination of the mass fraction of zinc in zinc 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*

3 Principle

The test portion of zinc concentrate is dissolved in hydrochloric, nitric and sulfuric acids. The acidity is adjusted to about 2 mol/l with respect to hydrochloric acid. Zinc is adsorbed on a strongly basic anion-exchange resin. Some interfering ions are removed by elution with 2 mol/l dilute hydrochloric acid. Zinc is eluted with an ammonia/ammonium chloride solution. Zinc is determined in the eluate by titration with EDTA at a pH of approximately 5,6 using xylenol-orange indicator.

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

The surface of the metal may be cleaned by immersing the metal in hydrochloric acid (4.3) diluted 1 + 9, for 1 min, then washed well with water followed by an acetone rinse and dried in an oven at 50 °C.

4.2 Xylenol-orange indicator (0,1 % m/m)

Mix 0,1 g of the sodium salt of xylenol orange with 100 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.

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

4.4 Hydrochloric acid, diluted (1 + 1)

Add 500 ml of hydrochloric acid (4.3) to 500 ml of water.

4.5 Hydrochloric acid, diluted (1 + 5)

Add 100 ml of hydrochloric acid (4.3) to 500 ml of water.

4.6 Nitric acid (ρ_{20} 1,42 g/ml)

4.7 Nitric acid, diluted (1 + 1)

Add 500 ml of nitric acid (4.6) to 500 ml of water.

4.8 Sulfuric acid, diluted (1 + 1)

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

4.9 Sulfuric acid, diluted (1 + 100)

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

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

4.11 Ammonia solution, diluted (7 + 100)

Add 70 ml of aqueous ammonia solution (ρ_{20} 0,89 g/ml) to 1 000 ml of water.

4.12 Ammonia/ammonium chloride solution

Dissolve 20 g of ammonium chloride in 1 l of dilute ammonia solution (4.11).

4.13 Hydrochloric acid/ascorbic acid solution

Dissolve 0,25 g of ascorbic acid in 100 ml of dilute hydrochloric acid (4.5). Prepare fresh on the day of use.

4.14 Ammonium fluoride solution (50 g/l)

Dissolve 50 g of ammonium fluoride in water and dilute to 1 l.

4.15 Sodium thiosulfate solution (100 g/l)

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

4.16 Buffer solution (pH = 5,5)

Dissolve 250 g of ammonium acetate and 25 ml of concentrated acetic acid (ρ_{20} 1,05 g/ml) in water and dilute to 1 l.

4.17 Para-nitrophenol indicator solution (2 g/l)

Dissolve 0,2 g of para-nitrophenol in water and dilute to 100 ml.

4.18 EDTA standard volumetric solution (0,1 mol/l)

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

5 Apparatus

5.1 Class A volumetric glassware complying with ISO 385, 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 Muffle furnace, capable of operating at 800 °C.

5.5 Platinum crucibles, of capacity 25 ml.

5.6 Ion exchange columns, having typical dimensions as shown in Annex A.

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 B).

6.2 Test portion

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