

Zinc sulfide concentrates — Determination of zinc — Solvent extraction and EDTA titrimetric method

ICS 73.060.99

National foreword

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The UK participation in its preparation was entrusted to Technical Committee NFE/36, Copper, lead and zinc ores and concentrates, which has the responsibility to:

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- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep UK interests informed;
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Summary of pages

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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.

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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 13291 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 13291:1997), which has been technically revised.

Zinc sulfide concentrates — 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.