



BSI Standards Publication

**Copper and zinc sulfide concentrates —
Determination of fluorine content by
sodium hydroxide fusion and fluoride
ion selective electrode detection**

National foreword

This British Standard is the UK implementation of ISO 13548:2025.

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Standard

ISO 13548

**Copper and zinc sulfide
concentrates — Determination
of fluorine content by sodium
hydroxide fusion and fluoride ion
selective electrode detection**

*Concentrés sulfurés de cuivre et de zinc — Dosage du fluor
par fusion à l'hydroxyde de sodium et détection par électrode
sélective des ions fluorure*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Copper and zinc sulfide concentrates — Determination of fluorine content by sodium hydroxide fusion and fluoride ion selective electrode detection

WARNING — This document can involve hazardous materials and equipment. It is the responsibility of the user of this document to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This document specifies a procedure for the determination of fluorine in copper and zinc sulfide concentrates by sodium hydroxide fusion and fluoride ion selective electrode (FISE) detection. Fluoride ions are leached from the sodium hydroxide fusion and presented to the FISE. Potential interfering ions are masked by the use of a complexing agent and control of solution pH. If the aluminium content in the concentrate is up to a mass fraction of 4,5 %, there is no effect of aluminium in this method. However, if the aluminium content in the concentrate exceeds a mass fraction of 4,5 %, there is a possibility that it can affect the analytical results.

The method is applicable to fluorine in the following ranges:

- copper concentrates: mass fraction of 0,008 % to 0,3 %;
- zinc concentrates: mass fraction of 0,005 % to 0,1 %.

The results of the trueness test can be found in [Annex C](#).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 12743, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <https://www.electropedia.org/>

4 Principle

The test portion is decomposed by fusion with sodium hydroxide. Fluorine is then determined by ion selective electrode after masking of potential interfering ions by use of a complexing agent and pH control.

5 Reagents

During the analysis, only reagents of analytical grade and grade 2 water of ISO 3696 shall be used. Particular care should be taken to ensure all reagents are fluorine free, except for [5.2](#) and the solutions in [5.10](#), [5.11](#) and [5.12](#).

5.1 Sodium hydroxide (NaOH) pellets, AR grade.

5.2 Sodium fluoride (NaF > 99,90 %), AR grade.

5.3 Sodium nitrate (NaNO₃), AR grade.

5.4 Tri-sodium citrate dihydrate (Na₃C₆H₅O₇·2H₂O).

5.5 Nitric acid ($\rho_{20} = 1,38$ g/ml).

5.6 200 g/l tri-sodium citrate solution.

Dissolve 200 g of tri-sodium citrate dihydrate ([5.4](#)) in 1 000 ml of water.

5.7 Nitric acid (1+1).

5.8 Nitric acid (1+99).

5.9 5 g/l sodium hydroxide solution.

Dissolve 0,5 g of sodium hydroxide ([5.1](#)) in 100 ml of water.

5.10 Working fluorine standard (1 000 mg/l).

Dissolve $2,210 \times 100/A$ g (A is the purity of a mass fraction of NaF % of sodium fluoride ([5.2](#)), dried from between 500 °C to 550 °C for 1 h by using a platinum dish ([6.24](#)), in 400 ml of water. Transfer the solution quantitatively into a 1 000 ml plastic volumetric flask ([6.8](#)). Dilute to volume with water. Store in a labelled plastic bottle.

Alternatively, purchase a suitable high-quality prepared standard.

NOTE There are reference solutions with metrological traceability secured.

5.11 Working fluorine standard solution A (100 mg/l).

Pipette 10,0 ml of 1 000 mg/l fluorine standard solution ([5.10](#)) into a 100 ml plastic volumetric flask ([6.8](#)). Dilute to volume with water. Store in a labelled plastic bottle.

Alternatively, purchase a suitable high-quality prepared standard.

NOTE There are reference solutions with metrological traceability secured.

5.12 Working fluorine standard solution B (10 mg/l).

Pipette 10,0 ml of 100 mg/l fluorine standard solution ([5.11](#)) into a 100 ml plastic volumetric flask ([6.8](#)). Dilute to volume with water. Store in a labelled plastic bottle.