International 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

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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/

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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 ($Na_3C_6H_5O_7 \cdot 2H_2O$).

5.5 Nitric acid (ρ20 = 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 × 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.

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