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Copper concentrates — Determination of mercury content — Cold vapour atomic absorption spectrometric method

Concentrés de cuivre — Dosage du mercure — Méthode par spectrométrie d'absorption atomique de vapeur froide

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Foreword

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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 documents 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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 concentrates — Determination of mercury content — Cold vapour atomic absorption spectrometric method

WARNING — The use of this document can involve hazardous materials, operations 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 an acid digestion and vapour generation atomic absorption spectrometric method for the determination of the mercury content in copper sulfide concentrates.

This document is applicable to mass fraction of mercury between 5 µg/g and 65 µg/g in copper sulfide concentrates.

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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume 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 instruments — Methods for testing of capacity and for use*

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

ISO 12743:2018, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

ISO Guide 35, *Reference materials — Guidance for characterization and assessment of homogeneity and stability*

3 Terms and definitions

No terms and definitions are listed in this document.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

The test portion is decomposed by treatment with hydrochloric and nitric acid at a temperature between 60 °C and 80 °C followed by the addition of potassium permanganate as oxidizing agent. Subsequently, the potassium permanganate is reduced by hydroxylamine hydrochloride. The mercury

vapour is generated by vapour generation using tin (II) chloride as reduction agent. The equipment is set to measure the absorbance at 253,7 nm. The absorbances of the test and calibration solutions, including those of certified or other reference materials, are compared to determine the mercury content.

5 Reagents

During the analysis, use only reagents of recognized analytical grade and grade 2 water in accordance with ISO 3696.

Reagents shall be selected or purified for the lowest possible blank value.

5.1 Tin (II) chloride dehydrate, ($SnCl_2.2H_2O$), containing < 5 mg/g mercury.

5.2 Potassium permanganate ($KMnO_4$).

5.3 Hydroxylamine hydrochloride ($HONH_2.HCl$).

5.4 Mercury (II) chloride ($HgCl_2$).

5.5 Nitric acid, ρ = 1,42 g/ml.

5.6 Hydrochloric acid, ρ = 1,16 g/ml to 1,19 g/ml.

5.7 Sulfuric acid, ρ = 1,84 g/ml.

5.8 Sulfuric acid solution, diluted 1 + 5.

5.9 Sulfuric acid solution, diluted 1 + 9.

5.10 Aqua regia. Mix 300 ml of hydrochloric acid (5.6) and 100 ml nitric acid (5.5). Prepare freshly for each batch of mercury determination.

5.11 Tin (II) chloride solution, 100 g/l. Add 10 g of tin (II) chloride (5.1) to 80 ml sulfuric acid solution (5.9). Heat and swirl to dissolve. Cool the solution and dilute with deionized water to 100 ml and mix thoroughly.

Continuously stir the solution with a magnetic stirrer for at least 2 h before use and maintain stirring during analysis. Prepare weekly.

Hydrochloric acid may be used instead of sulfuric acid.

Alternative procedure:

Tin (II) chloride solution, 100 g/l: add 10 g of tin (II) chloride (5.1) to 20 ml of deionized water. Add continuously 60 ml of sulfuric acid solution (5.8). Heat and swirl to dissolve. Cool, dilute to 100 ml with deionized water and mix thoroughly.

5.12 Potassium permanganate ($KMnO_4$) solution, 2 g/l. Add 0,2 g of potassium permanganate (5.2) to 100 ml of deionized water. Store in a glass bottle.

5.13 Hydroxylamine hydrochloride solution, 20 g/l. Add 2 g of hydroxylamine hydrochloride (5.3) to 100 ml of deionized water.

5.14 Mercury trapping solution. Add 10 g of potassium permanganate (5.2) to 100 ml of water and mix well. This reagent is used to oxidize mercury vapour to its Hg^{2+} state and trap it in solution.

