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Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic —

Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method

Concentrés sulfurés de cuivre, de plomb et de zinc — Dosage de l'arsenic —

Partie 1: Méthode par digestion acide, co-précipitation avec le fer et plasma induit par haute fréquence

Reference number
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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 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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

ISO 13547 consists of the following parts, under the general title *Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic:*

— *Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method*

— *Part 2: Acid digestion and inductively coupled plasma atomic emission spectrometric method*

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Copper, lead, zinc and nickel sulfide concentrates — Determination of arsenic —

Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric 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 an iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric (ICP-AES) method for the determination of the mass fraction of arsenic in copper, lead, zinc, and nickel sulfide concentrates as follows:

a) for copper sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 2,0 %;

b) for lead sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 1,0 %;

c) for zinc sulfide concentrates, the method is applicable to the determination of mass fractions of arsenic from 0,05 % to 0,6 %;

d) for nickel sulfide concentrates, the method is applicable to the determination of mass fraction of arsenic from 0,05 % to 1,0 %.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 4787, *Laboratory glassware — Volumetric instruments — Methods for testing of capacity and for use*

ISO 8466-2, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 2: Calibration strategy for non-linear second-order calibration functions*

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

ISO 12743:2006, *Copper, lead, zinc and nickel concentrates — Sampling procedures for determination of metal and moisture content*

ISO Guide 35:2006, *Reference materials — General and statistical principles for certification*

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3 Principle

The test portion is decomposed in nitric and sulphuric acids. Arsenic is coprecipitated in ammonia solution by the carriers of iron hydroxide to be separated from the matrix. The arsenic content is determined by comparison against iron-matched standards using ICP-AES.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and distilled water, or water of equivalent purity.

4.1 Sodium hydroxide (NaOH), AR grade.

4.2 Arsenic trioxide (As_2O_3), AR grade.

4.3 Iron(III) chloride hexahydrate ($FeCl_3 \cdot 6H_2O$), AR grade (<0,000 05 % arsenic).

4.4 Ammonium chloride (NH_4Cl), AR grade.

4.5 Ammonium acetate, AR grade.

4.6 Nitric acid (ρ_{20} 1,42 g/mL), AR grade.

4.7 Nitric acid, dilute (1 + 1).

Slowly, add 50 ml of nitric acid (4.6) to 50 ml of water while stirring.

4.8 Hydrochloric acid (ρ_{20} 1,16 g/mL), AR grade.

4.9 Hydrochloric acid, dilute (1 + 1).

Slowly add 50 ml of hydrochloric acid (4.8) to 50 ml of water while stirring.

4.10 Sulphuric acid (ρ_{20} 1,840 g/mL), AR grade.

4.11 Sulphuric acid, dilute (1 + 1).

Slowly add 50 ml of sulphuric acid (4.10) to 50 ml of water while stirring.

4.12 Sulphuric acid, dilute (1 + 50).

Slowly add 20 ml of sulphuric acid (4.10) to 1 000 ml of water while stirring.

The addition of sulphuric acid to water generates heat and shall be performed with adequate precautions.

4.13 Hydrofluoric acid (ρ_{20} 1,13 g/mL).

4.14 Aqueous ammonia (ρ_{20} 0,91 g/mL).

4.15 Ammonia solution (1 + 99).

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