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Copper, lead and zinc sulfide concentrates — Determination of cadmium —

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

Concentrés de sulfure de cuivre, de plomb et de zinc — Dosage du cadmium —

Partie 2: Méthode par digestion acide et spectroscopie d'émission atomique à plasma à couplage inductif

Reference number
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Foreword

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

A list of all parts in the ISO 19976 series can be found on the ISO website.

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Copper, lead and zinc sulfide concentrates — Determination of cadmium —

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

WARNING — The use of this document might involve hazardous materials, operations and equipment. It is the responsibility of the user of this document to establish appropriate health and safety practices.

1 Scope

This document specifies an acid digestion and inductively coupled plasma atomic emission spectrometric (ICP-AES) method for the determination of the mass fraction of cadmium in copper, lead and zinc sulfide concentrates as follows:

a) for copper sulfide concentrates, the method is applicable to the determination of mass fractions of cadmium from 0,01 % to 0,30 %;

b) for lead sulfide concentrates, the method is applicable to the determination of mass fractions of cadmium from 0,01 % to 0,30 %;

c) for zinc sulfide concentrates, the method is applicable to the determination of mass fractions of cadmium from 0,05 % to 1,00 %.

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 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, 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*

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

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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 sample is dissolved in hydrochloric acid, nitric acid and hydrofluoric acid with a final stage perchloric acid digestion. Yttrium is added as an internal standard to solutions of dissolved samples, reference materials and calibration solutions and cadmium determined by ICP-AES.

5 Reagents

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

5.1 Hydrochloric acid, concentrated (ρ_{20} 1,19 g/ml).

5.2 Hydrochloric acid, dilute 1+1. Slowly add 500 ml of concentrated hydrochloric acid (5.1) to 500 ml of water while stirring.

5.3 Hydrochloric acid, dilute 1+9. Slowly add 50 ml of concentrated hydrochloric acid (5.1) to 450 ml of water while stirring.

5.4 Nitric acid, concentrated (ρ_{20} 1,42 g/ml).

5.5 Hydrofluoric acid, concentrated (ρ_{20} 1,15 g/ml).

5.6 Perchloric acid, concentrated (ρ_{20} 1,70 g/ml).

5.7 Cadmium metal, minimum purity 99,99 %.

5.8 Cadmium, standard solution, 1 ml contains 1 mg of Cd.

Weigh, to the nearest 0,1 mg, 1,000 0 g of cadmium metal (5.7) into a 250 ml beaker. Add 10 ml of water and 5 ml of nitric acid (5.4). Cover and warm gently (if necessary) until the cadmium metal is dissolved. Heat to remove nitrogen oxides, then allow to cool, and add about 50 ml of water. Transfer the solution to a 1 000 ml one-mark volumetric flask, add 40 ml of hydrochloric acid (5.2), fill up nearly to the mark with water, mix and cool to room temperature. Then fill up exactly to the mark with water and mix again.

5.9 Cadmium, standard solution, 1 ml contains 100 µg of Cd.

Pipette 10,00 ml of cadmium standard solution (5.8) into a 100 ml one-mark volumetric flask. Add 20 ml of hydrochloric acid (5.2), fill up nearly to the mark with water, mix and cool to room temperature. Then fill up exactly to the mark with water and mix again.

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