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METHODS FOR THE ANALYSIS OF COPPER SULPHIDE CONCENTRATES

Part 1—DETERMINATION OF COPPER (SHORT IODIDE TITRIMETRIC METHOD)

STANDARDS ASSOCIATION OF AUSTRALIA
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The following interests are represented on Committee MN/5:

Australasian Institute of Mining and Metallurgy

Australian Lead Development Association

Australian Mineral Development Laboratories

Australian Mining Industry Council

CSIRO, Institute of Energy and Earth Resources

The Royal Australian Chemical Institute

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AUSTRALIAN STANDARD

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Part 1
DETERMINATION OF COPPER (SHORT IODIDE TITRIMETRIC METHOD)

AS 2917.1—1986

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PREFACE

This standard was prepared by the Association's Committee on Copper, Lead and Zinc Ores and Concentrates under the direction of the Minerals Standards Board as part of its program of standardizing methods for the determination of elements of commercial interest in such materials.

The short iodide method for titrimetric determination of copper in copper concentrates is so called because it provides a rapid method for the determination of copper. The method has been shown to be free from interference from all elements except high levels of bismuth.

In order to provide precision data, a comprehensive inter-laboratory test program was organized. Details on the precision of the method are reported in the standard.

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF COPPER SULPHIDE CONCENTRATES

PART 1—DETERMINATION OF COPPER (SHORT IODIDE TITRIMETRIC METHOD)

1 SCOPE. This standard sets out a titrimetric method (without separation of interfering elements) for the determination of the copper content of copper sulphide concentrates.

2 APPLICATION. The method is applicable to copper concentrates containing between 15 percent and 60 percent copper and up to 1 percent bismuth.

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 2134 Code of Practice for the Chemical Analysis of Materials by Flame Atomic Absorption Spectroscopy

AS 2162 Code of Practice for the Use of Volumetric Glassware

AS 2816 Copper Lead and Zinc Sulphide Concentrates—Determination of Hygroscopic Moisture in the Analysis Sample

AS 2850 Chemical Analysis—Interlaboratory Test Programs—Guide to the Planning and Conduct—For Determining Precision of Analytical Methods

AS 2862 Copper, Lead and Zinc Sulphide Concentrates—Sampling—
Part 1—Sampling from Moving Streams*
Part 2—Sampling from Stationary Situations*
Part 3—Preparation of Samples

4 PRINCIPLE. Decomposition of the concentrate in a sulphuric acid/nitric acid mixture. Addition of excess potassium iodide and titration with sodium thiosulphate in the presence of soluble starch indicator.

5 REAGENTS.

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

5.2 Solids.

5.2.1 *Copper metal.* Minimum 99.95 percent purity.

5.2.2 *Ammonium hydrogen difluoride.*

5.3 Solutions.

5.3.1 *Soluble starch solution.* Slurry 1 g of soluble starch powder with cold water, pour into 100 mL of boiling water and boil for 2 min. Prepare freshly each day.

NOTE: Alternatively commercial starch indicators may be prepared in accordance with the manufacturer's instructions. Vitex has been found suitable.

5.3.2 *Dilute sulphuric acid.* (500 mL/L).

CAUTION: Safety glasses should be worn during the following preparation.

To 500 mL of water add slowly, with stirring, 500 mL of sulphuric acid (ϱ_{20} 1840 kg/m^3). Cool, dilute to 1 L with water and mix.

5.3.3 *Dilute sulphuric acid.* (2 mL/L). To 900 mL of water add slowly, with stirring, 2 mL of sulphuric acid (ϱ_{20} 1840 kg/m^3). Cool, dilute to 1 L with water and mix.

5.3.4 *Nitric acid.* (ϱ_{20} 1420 kg/m^3).

5.3.5 *Dilute nitric acid.* (500 mL/L). To 500 mL of water add slowly, with stirring, 500 mL of nitric acid (5.3.4). Cool, dilute to 1 L with water and mix.

5.3.6 *Dilute hydrochloric acid.* (200 mL/L). To 800 mL of water add carefully, with stirring, 200 mL of hydrochloric acid (ϱ_{20} 1160 kg/m^3 to 1190 kg/m^3).

5.3.7 *Potassium iodide solution.* Add 500 g of potassium iodide to 350 mL of water. Warm and stir to dissolve. Allow to cool.

NOTE: This reagent should be prepared fresh daily.

5.3.8 *Sodium thiosulphate solution.* (20 g/L). Dissolve 20 g of sodium thiosulphate pentahydrate in 1 L of freshly boiled and cooled water. Add 0.2 g of sodium carbonate and stir to dissolve. Standardize by the procedure specified in Clause 8.6.

NOTE: It is preferable to allow the solution to stand for one week before standardization is carried out.

5.3.9 *Acetone.*

6 APPARATUS.

6.1 Ordinary laboratory glassware.

6.2 Grade A volumetric glassware.

6.3 Hotplate.

7 SAMPLES. Samples shall be taken in accordance with AS 2862.1 or AS 2862.2 and prepared in accordance with AS 2862.3.

8 PROCEDURE.

8.1 Number of determinations. It is recommended that duplicate determinations be carried out on each laboratory sample.

8.2 Blank test. Carry out a blank test in parallel with the analysis using the same procedure as for the analysis and the same quantities of all reagents but omitting the sample. The purpose of the blank test in this method is to check the quality of the reagents. If a blank titration is obtained, check all reagents and rectify the problem.

* In course of preparation.