



Copper sulfide concentrates— Determination of copper content— Titrimetric methods



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Australian Standard[®]

**Copper sulfide concentrates—
Determination of copper content—
Titrimetric methods**

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PREFACE

This Standard was prepared by the Standards Australia Committee MN-005, Copper, Lead, Zinc and Nickel Ores and Concentrates, to supersede AS 2917.1—1994.

The objective of this Standard is to specify two titrimetric methods for the determination of the copper content of copper sulfide concentrates in the range 15 % (m/m) to 50 % (m/m), using sodium thiosulfate after separation (method 1) or without separation (method 2) of copper from interfering elements.

This Standard is identical with, and has been reproduced from ISO 10258:2015, *Copper sulfide concentrates Determination of copper content Titrimetric methods*.

As this Standard is reproduced from an International Standard, the following applies:

- (a) In the source text ‘this International Standard’ should read ‘this Australian Standard’.
- (b) A full point substitutes for a comma when referring to a decimal marker.

References to International Standards should be replaced by references to Australian Standards, as follows:

<i>Reference to International Standard</i>		<i>Australian Standard</i>	
ISO		AS	
1042	Laboratory glassware—One-mark volumetric flasks	2164	Laboratory glassware—One-mark volumetric flasks
9599	Copper, lead and zinc sulfide concentrates—Determination of hygroscopic moisture content of the analysis sample—Gravimetric method	2816	Copper, lead and zinc sulfide concentrates—Determination of hygroscopic moisture in the analysis sample—Gravimetric method

Only normative references that have been adopted as Australian Standards have been listed.

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the annexes to which they apply. A ‘normative’ annex is an integral part of a Standard, whereas an ‘informative’ annex is only for information and guidance.

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NOTES

Copper sulfide concentrates—Determination of copper content— Titrimetric methods

1 Scope

This International Standard specifies two titrimetric methods for the determination of the copper content of copper sulfide concentrates in the range 15 % (m/m) to 50 % (m/m), using sodium thiosulfate after separation (method 1) or without separation (method 2) of copper from interfering elements.

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

3 Principle

3.1 Method 1 (Long iodide method)

A test portion is decomposed in nitric and sulfuric acids, and arsenic, antimony, and tin are removed by treatment with hydrobromic acid. Copper is separated from interfering elements by precipitation of copper sulfide with sodium thiosulfate. The precipitate is dissolved in nitric and sulfuric acids, ammonium hydrogen difluoride is added to eliminate interference of residual iron, and excess potassium iodide is also added. Free iodine isolated by reaction between iodide ions and copper(II) ions is titrated with sodium thiosulfate using soluble starch as the indicator.

3.2 Method 2 (Short iodide method)

A test portion is decomposed in nitric and sulfuric acids, and arsenic, antimony, and tin are removed by treatment with hydrobromic acid. Ammonium hydrogen difluoride is added to eliminate interference of iron, and excess potassium iodide is also added. Free iodine isolated by reaction between iodide ions and copper(II) ions is titrated with sodium thiosulfate using soluble starch as the indicator.

4 Reagents

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

4.1 Copper metal, minimum purity 99,99 %.

4.2 Potassium iodide.

4.3 Ammonium hydrogen difluoride.**4.4 Sulfuric acid**, diluted 1 + 1.

Slowly add 500 ml of concentrated sulfuric acid (ρ_{20} 1,84 g/ml) to 500 ml of water, while stirring and cooling.

4.5 Sulfuric acid, diluted 1 + 999.

Add 1 ml of dilute sulfuric acid (4.4) to 500 ml of water.

4.6 Nitrit acid, concentrated (ρ_{20} 1,42 g/ml).**4.7 Nitrit acid**, diluted 1 + 1.

Slowly add 500 ml of concentrated nitric acid (4.6) to 500 ml of water.

4.8 Hydrofluoric acid (ρ_{20} 1,14 g/ml).**4.9 Bromine.****4.10 Bromine water**, saturated.**4.11 Hydrobromic acid** (ρ_{20} 1,50 g/ml).**4.12 Acetic acid**, diluted 1 + 3.

Slowly add 25 ml of glacial acetic (ρ_{20} 1,05 g/ml) to 75 ml of water.

4.13 Nitration mixture.

Slowly add 250 ml of concentrated sulfuric acid (ρ_{20} 1,84 g/ml) to 250 ml of concentrated nitric acid (4.6).

4.14 Ammonium hydrogen difluoride, 250 g/l solution.**4.15 Sodium carbonate**, 20 g/l solution.**4.16 Sodium thiosulfate pentahydrate**, 200 g/l solution.**4.17 Potassium thiocyanate**, 100 g/l solution.**4.18 Starch**, 2 g/l solution.

Moisten 1 g of soluble starch with cold water, slowly pour into 500 ml of hot water while stirring, and boil for about 1 min.

4.19 Ethanol.**4.20 Standard solutions.**

Standard solutions should be prepared at the same ambient temperature as that at which the determinations will be conducted.

4.20.1 Sodium thiosulfate, standard volumetric solution (20 g/l).

4.20.1.1 Preparation

Dissolve 20 g of sodium thiosulfate (pentahydrate) in 1 l of freshly boiled and cooled water. Add 0,2 g of sodium carbonate, stir to dissolve and allow to stand for at least one day. Standardize this solution as specified in [4.20.1.2](#).

4.20.1.2 Standardization

Clean a piece of copper metal ([4.1](#)) by immersing it in warm dilute acetic acid ([4.12](#)). Wash the copper thoroughly with water followed by ethanol ([4.19](#)) and allow to dry in air. Weigh into three separate 400 ml conical beakers to the nearest 0,1 mg, a mass of clean copper metal which approximates the copper content in the test portion. Record these masses as m_1 , m_2 , and m_3 .

Dissolve the copper using 10 ml of dilute nitric acid ([4.7](#)) followed by 5 ml of dilute sulfuric acid ([4.4](#)). Heat to evaporate to dryness. Add 40 ml of water, heat to dissolve the soluble salts, and cool. Continue the standardization as specified in [7.3.4](#) for method 1 and in [7.4.2](#) for method 2. Record the volumes of sodium thiosulfate solution used in the titration as V_1 , V_2 , and V_3 .

The standardization factor of the standard volumetric solution varies with the volume of sample solution, mass of potassium iodide, mass of copper, and temperature of solution. The same volume of solution and mass of potassium iodide as those used for the standardization should be used for the analysis of the test portion. The temperatures of standardization and determination should be essentially the same.

Calculate the standardization factors f_1 , f_2 , and f_3 using the following formulae:

$$f_1 = \frac{m_1}{V_1} \quad (1)$$

$$f_2 = \frac{m_2}{V_2} \quad (2)$$

$$f_3 = \frac{m_3}{V_3} \quad (3)$$

Calculate, to four significant figures, the mean standardization factor f for the sodium thiosulfate standard volumetric solution, provided that the range of the values of f_1 , f_2 , and f_3 does not exceed 10^{-5} g Cu/ml. If this range is exceeded, repeat the standardization.

4.20.2 Copper, standard solution (0,1 mg/ml).

Weigh, to the nearest 0,1 mg, 0,1 g of copper metal ([4.1](#)) into a 200 ml beaker, decompose with 10 ml of dilute nitric acid ([4.7](#)). Heat to remove nitrogen oxides, cool, and add about 50 ml of water. Transfer to a 1 000 ml volumetric flask, fill up nearly to the mark with water, mix and cool to room temperature; then fill up exactly to the mark and mix again.

5 Apparatus

Ordinary laboratory equipment and the following.

5.1 Volumetric glassware, of class A complying with ISO 385, ISO 648, and ISO 1042, and used in accordance with ISO 4787.

5.2 Analytical balance, sensitive to 0,1 mg.

5.3 Platinum crucibles.