



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

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



This Australian Standard® was prepared by Committee MN-005, Copper, Lead, Zinc and Nickel Ores and Concentrates. It was approved on behalf of the Council of Standards Australia on 26 August 2015.
This Standard was published on 29 September 2015.

The following are represented on Committee MN-005:

- Australasian Institute of Mining and Metallurgy
 - CSIRO
 - Minerals Council of Australia
-

This Standard was issued in draft form for comment as DR AS ISO 13547.1:2015.

Standards Australia wishes to acknowledge the participation of the expert individuals that contributed to the development of this Standard through their representation on the Committee and through the public comment period.

Keeping Standards up-to-date

Australian Standards® are living documents that reflect progress in science, technology and systems. To maintain their currency, all Standards are periodically reviewed, and new editions are published. Between editions, amendments may be issued.

Standards may also be withdrawn. It is important that readers assure themselves they are using a current Standard, which should include any amendments that may have been published since the Standard was published.

Detailed information about Australian Standards, drafts, amendments and new projects can be found by visiting www.standards.org.au

Standards Australia welcomes suggestions for improvements, and encourages readers to notify us immediately of any apparent inaccuracies or ambiguities. Contact us via email at mail@standards.org.au, or write to Standards Australia, GPO Box 476, Sydney, NSW 2001.

Australian Standard[®]

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

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

First published as AS ISO 13547.1:2015.

COPYRIGHT

© Standards Australia Limited

All rights are reserved. No part of this work may be reproduced or copied in any form or by any means, electronic or mechanical, including photocopying, without the written permission of the publisher, unless otherwise permitted under the Copyright Act 1968.

Published by SAI Global Limited under licence from Standards Australia Limited, GPO Box 476, Sydney, NSW 2001, Australia

ISBN 978 1 76035 253 0

PREFACE

This Standard was prepared by the Standards Australia Committee MN-005 Copper, Lead, Zinc and Nickel Ores and Concentrates.

The objective of this Standard is to specify 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.

This Standard is identical with, and has been reproduced from ISO 13547-1:2014, *Copper, lead, zinc and nickel sulfide concentrates—Determination of arsenic, Part 1: Iron hydroxide concentration and inductively coupled plasma atomic emission spectrometric method*.

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 or Australian/New Zealand Standards, as follows:

<i>Reference to International Standard</i>		<i>Australian Standard</i>	
ISO		AS	
9599	Copper, lead and zinc sulfide concentrates—Determination of hygroscopic moisture in the analysis sample—Gravimetric method	2816	Copper, lead and zinc sulfide concentrates—Determination of hygroscopic moisture in the analysis sample—Gravimetric method
		2862	Copper, lead, zinc and nickel concentrates—Sampling
12743	Copper, lead, zinc and nickel concentrates—Sampling procedures for determination of metal and moisture content	2862.1	Part 1: Sampling procedures for determination of metal and moisture content

Only normative references that have been adopted as Australian or Australian/New Zealand Standards have been listed.

The term ‘normative’ has been used in this Standard to define the application of the annexes to which it applies. A ‘normative’ annex is an integral part of a Standard.

CONTENTS

1	Scope	1
2	Normative references	1
3	Principle	2
4	Reagents	2
5	Apparatus	3
6	Sample	4
6.1	Laboratory sample	4
6.2	Test sample	4
6.3	Test portion	4
7	Procedure	4
7.1	Number of determinations	4
7.2	Blank test	4
7.3	Dissolution of the test portion	4
7.4	Removal of lead sulfate	5
7.5	Dissolution of the insoluble residue	5
7.6	Separation of arsenic	5
7.7	Preparation of test solutions	6
7.8	Preparation of calibration solutions	6
7.9	Preparation of arsenic calibration curve	6
7.10	Determination of arsenic content in test solutions	6
8	Expression of results	7
9	Precision	7
9.1	Expression of precision	7
9.2	Procedure for obtaining the final result	8
9.3	Between-laboratories precision	8
9.4	Check of trueness	9
10	Test report	10
Annex A (normative) Procedure for the preparation and determination of the mass of a pre-dried test portion		11
Annex B (normative) Flowsheet of the procedure for the acceptance of analytical values for test samples		13
Bibliography		14

NOTES

AUSTRALIAN STANDARD

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*

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 (As₂O₃), AR grade.

4.3 Iron(III) chloride hexahydrate (FeCl₃•6H₂O), AR grade (<0,000 05 % arsenic).

4.4 Ammonium chloride (NH₄Cl), 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).