

Australian Standard[®]

**Methods for the analysis of lead
sulfide concentrates—**

**Part 2: Determination of lead
content—Back titration of EDTA
after precipitation of lead sulfate**

[ISO title: Lead sulfide concentrates — Determination of lead
content— Back titration of EDTA after precipitation of lead sulfate]

This Australian Standard was prepared by Committee MN/5—Copper, Lead, Zinc, Gold and Silver Ores and Concentrates. It was approved on behalf of the Council of Standards Australia on 10 May 1996 and published on 5 August 1996.

The following interests are represented on Committee MN/5:

Australasian Institute of Mining and Metallurgy

Australian Lead Development Association

CSIRO, Division of Mineral and Processing Engineering

Minerals Council of Australia

The Royal Australian Chemical Institute

Review of Australian Standards. *To keep abreast of progress in industry, Australian Standards are subject to periodic review and are kept up to date by the issue of amendments or new editions as necessary. It is important therefore that Standards users ensure that they are in possession of the latest edition, and any amendments thereto.*

Full details of all Australian Standards and related publications will be found in the Standards Australia Catalogue of Publications; this information is supplemented each month by the magazine 'The Australian Standard', which subscribing members receive, and which gives details of new publications, new editions and amendments, and of withdrawn Standards.

Suggestions for improvements to Australian Standards, addressed to the head office of Standards Australia, are welcomed. Notification of any inaccuracy or ambiguity found in an Australian Standard should be made without delay in order that the matter may be investigated and appropriate action taken.

Australian Standard[®]

**Methods for the analysis of lead
sulfide concentrates—**

**Part 2: Determination of lead
content—Back titration of EDTA
after precipitation of lead sulfate**

PREFACE

This Standard was prepared by the Standards Australia Committee MN/5 on Copper, Lead, Zinc, Gold and Silver Ores and Concentrates as part of a programme of standardizing methods for the determination of elements of commercial interest in such materials.

The objective of this Standard is to provide those involved in the analysis of lead sulfide concentrates with a standardized method of determining lead content supported by precision data obtained from an inter-laboratory test programme.

It is identical with and has been reproduced from ISO 11441:1995, which has been prepared by ISO/TC 183 Copper, Lead and Zinc Ores and Concentrates. Australia holds the Chairmanship and Secretariat of ISO/TC 183 and has made a significant contribution to the preparation of ISO 11441.

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

- (a) Its number does not appear on each page of text and its identity is shown only on its cover and title page.
- (b) In the source text, 'this international Standard' should read 'this Australian Standard'.
- (c) A full point substitutes for a comma when referring to a decimal point.

The references to international Standards should be replaced by references, where appropriate, to the following Australian or Joint Australian/New Zealand Standards:

<i>Reference to International Standard or other publication</i>	<i>Australian or Joint Australian/New Zealand 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
385-1 Laboratory glassware—Burettes Part 1: General requirements	—
648 Laboratory glassware—One-mark pipettes	—
1042 Laboratory glassware—One-mark volumetric flasks	—
4787 Laboratory glassware—Volumetric glassware—Methods for use and testing of capacity	—

© Copyright — STANDARDS AUSTRALIA

Users of Standards are reminded that copyright subsists in all Standards Australia publications and software. Except where the Copyright Act allows and except where provided for below no publications or software produced by Standards Australia may be reproduced, stored in a retrieval system in any form or transmitted by any means without prior permission in writing from Standards Australia. Permission may be conditional on an appropriate royalty payment. Requests for permission and information on commercial software royalties should be directed to the head office of Standards Australia.

Standards Australia will permit up to 10 percent of the technical content pages of a Standard to be copied for use exclusively in-house by purchasers of the Standard without payment of a royalty or advice to Standards Australia.

Standards Australia will also permit the inclusion of its copyright material in computer software programs for no royalty payment provided such programs are used exclusively in-house by the creators of the programs.

Care should be taken to ensure that material used is from the current edition of the Standard and that it is updated whenever the Standard is amended or revised. The number and date of the Standard should therefore be clearly identified.

The use of material in print form or in computer software programs to be used commercially, with or without payment, or in commercial contracts is subject to the payment of a royalty. This policy may be varied by Standards Australia at any time.

CONTENTS

	<i>Page</i>
1 Scope	1
2 Normative references	1
3 Principle	1
4 Reagents	1
5 Apparatus	3
6 Sample	4
7 Procedure	4
8 Expression of results	5
9 Precision	6
10 Test report	7
 Annexes	
A Procedure for the preparation and determination of the mass of a predried test portion	8
B Flow sheet of the procedure for the acceptance of analytical values for test samples	10
C Derivation of precision equations	11

(PAGE iv IN THE HARD COPY IS BLANK)

AUSTRALIAN STANDARD

Lead sulfide concentrates — Determination of lead content — Back titration of EDTA after precipitation of lead sulfate**1 Scope**

This International Standard specifies a lead sulfate precipitation EDTA titrimetric method for the determination of lead content in lead sulfide concentrates.

The method is applicable to all lead sulfide concentrates with lead content in the range from 10 % (*m/m*) to 80 % (*m/m*).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements*.

ISO 648:1977, *Laboratory glassware — One-mark pipettes*.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks*.

ISO 4787:1984, *Laboratory glassware — Volumetric glassware — Methods for use and testing of capacity*.

ISO 9599:1991, *Copper, lead and zinc sulfide concentrates — Determination of hygroscopic moisture in the analysis sample — Gravimetric method*.

ISO Guide 35:1989, *Certification of reference materials — General and statistical principles*.

3 Principle

The stages which comprise the determination are described in 3.1 to 3.4 inclusive.

3.1 Dissolution

Dissolution by fusion with sodium peroxide.

3.2 Precipitation

Double precipitation of lead as a sulfate, with removal of bismuth if applicable.

3.3 EDTA dissolution

Dissolution of lead sulfate in a known volume of an alkaline EDTA solution.

3.4 Titration

Titration of EDTA in excess by a titrated zinc solution.

4 Reagents

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

4.1 Lead metal, minimum 99,99 % purity.

The surface of the metal shall be free from oxide prior to use and may be cleaned by immersing the metal in nitric acid solution (4.10) diluted 1 + 9 for 1 min, washed well with water followed by acetone and dried in an oven at 50 °C.