

AS 3550.1—1988

Reconfirmed 2017

Australian Standard<sup>®</sup>

---

**METHODS FOR THE ANALYSIS OF  
WATERS**

**Part 1—DETERMINATION OF  
DISSOLVED SULPHIDE—  
SPECTROPHOTOMETRIC  
METHOD**

---

This Australian Standard was prepared by Committee CH/22, Methods for Examination of Waters. It was approved on behalf of the Council of the Standards Association of Australia on 26 May 1988 and published on 12 September 1988.

---

The following interests are represented on Committee CH/22:

Australian Construction Services, Department of Administrative Services  
Australian Government Analytical Laboratories  
Australian Mining Industry Council  
Confederation of Australian Industry  
Department of Environment, Tas.  
Department of Conservation, Forests and Lands  
Department of Health, N.S.W.  
Department of Local Government, Qld  
Department of Water Resources, N.S.W.  
Engineering and Water Supply Department, S.A.  
Environment Protection Authority, Vic.  
Ministry of Conservation, Vic.—Fisheries and Wildlife Division  
National Association of Testing Authorities, Australia  
National Health and Medical Research Council  
Royal Australian Chemical Institute  
Rural Water Commission, Vic.  
University of Sydney  
Water Board, Sydney

---

*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which subscribing members receive, and which gives details of new publications, new editions and amendments, and of withdrawn Standards.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.

---

*This Standard was issued in draft form for comment as DR 87247.*

STANDARDS AUSTRALIA

---

**RECONFIRMATION**

**OF**

**AS 3550.1—1988**

**METHODS FOR THE ANALYSIS OF WATERS**

**Part 1: DETERMINATION OF DISSOLVED SULPHIDE—  
SPECTROPHOTOMETRIC METHOD**

---

**RECONFIRMATION NOTICE**

Technical Committee EV-008 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

Certain documents referenced in the publication may have been amended since the original date of publication. Users are advised to ensure that they are using the latest versions of such documents as appropriate, unless advised otherwise in this Reconfirmation Notice.

Approved for reconfirmation in accordance with Standards Australia procedures for reconfirmation on 15 September 2017.

The following are represented on Technical Committee EV-008:

Australasian Land and Groundwater Association  
Australian Contaminated Land Consultants Association  
Australian Nuclear Science & Technology Organisation  
Australian Water Association  
Chem Centre  
Environmental Laboratory Industry Group  
National Association of Testing Authorities Australia  
National Measurement Institute  
Sydney Water Corporation

## NOTES

Australian Standard<sup>®</sup>

---

**METHODS FOR THE ANALYSIS OF  
WATERS**

**Part 1—DETERMINATION OF  
DISSOLVED SULPHIDE—  
SPECTROPHOTOMETRIC  
METHOD**

---

First published as AS 3550.1 — 1988.

## PREFACE

This Standard for the determination of sulphide in waters was prepared by the Association's Committee on Methods for Examination of Waters under the direction of the Chemical Standards Board.

Sulphide is present in some industrial wastes and is also generated in anoxic waters by the bacterial reduction of sulphate and the decomposition of organic matter. Hydrogen sulphide gas escaping from sulphide-rich waters is toxic and an odour nuisance.

---

 CONTENTS

	<i>Page</i>
1 SCOPE . . . . .	3
2 APPLICATION . . . . .	3
3 REFERENCED DOCUMENTS . . . . .	3
4 PRINCIPLE . . . . .	3
5 REAGENTS . . . . .	3
6 APPARATUS . . . . .	3
7 SAMPLES AND SAMPLING . . . . .	3
8 PROCEDURE . . . . .	4
9 CALCULATION . . . . .	4
10 PRECISION AND LIMIT OF DETECTION . . . . .	4
11 REPORT . . . . .	4
APPENDICES	
A INTERFERENCES . . . . .	5
B IODOMETRIC METHOD FOR THE STANDARDIZATION OF SULPHIDE SOLUTION . . . . .	6
C DETERMINATION OF DISSOLVED SULPHIDE—EXTRAC- TION SPECTROPHOTOMETRIC METHOD . . . . .	7
REFERENCES . . . . .	9

## © 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.

## STANDARDS ASSOCIATION OF AUSTRALIA

## Australian Standard

## METHODS FOR THE ANALYSIS OF WATERS

PART 1: DETERMINATION OF DISSOLVED SULPHIDE—  
SPECTROPHOTOMETRIC METHOD

**1 SCOPE.** This Standard sets out a spectrophotometric method for the determination of dissolved sulphide in waters.

**2 APPLICATION.** The method is applicable to natural and waste waters containing dissolved sulphide in the concentration range 0.1 mg/L to 50 mg/L. A more sensitive version of the method, involving solvent extraction, is given in Appendix C.

## NOTES:

1. Insoluble sulphides such as mercury(II) sulphide and copper(II) sulphide are not measured by this method.
2. Information on interferences by common cations and anions is given in Appendix A.

**3 REFERENCED DOCUMENTS.** The following documents are referred to in this Standard.

## AS

- 2031 Selection of containers and preservation of water samples for chemical and microbiological analysis  
Part 1: Chemical (AS 2031.1)
- 2162 Code of practice for the use of volumetric glassware
- 2850 Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct
- CK19 Code of recommended practice for the chemical analysis of materials by ultraviolet/visible spectrophotometry

**4 PRINCIPLE.** Bis(DMP)copper(II) ions (where DMP = 2,9-dimethyl-1,10-phenanthroline) are reduced by sulphide at pH 4.8 to the yellow-orange bis(DMP)copper(I) complex and the absorbance is measured at 454 nm.

**5 REAGENTS.**

**5.1 General requirements.** Unless otherwise specified, use analytical grade reagents and distilled water or water of equivalent purity.

**5.2 Solutions.**

**5.2.1 Acetic acid solution (0.5 mol/L).** Dilute 30 mL of glacial acetic acid ( $\rho_{20}$  1.05 g/mL) to 1 L.

**5.2.2 Sodium acetate solution (0.5 mol/L).** Dissolve 41 g of anhydrous sodium acetate ( $\text{CH}_3\text{COONa}$ ) in water and dilute to 1 L.

**5.2.3 Buffer solution pH 4.8.** Mix 400 mL of acetic acid solution (5.2.1) and 600 mL of sodium acetate solution (5.2.2). Check the pH and, if necessary, adjust by adding either acetic acid solution (5.2.1) or sodium acetate solution (5.2.2).

**5.2.4 Copper sulphate solution.** Dissolve 2.0 g of copper sulphate pentahydrate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) in water and dilute to 1 L.

**5.2.5 Copper-DMP reagent.** Dissolve 0.15 g of 2,9 dimethyl-1,10-phenanthroline (DMP) hydrochloride (neocuproine hydrochloride) in water, add 25 mL of copper sulphate solution (5.2.4) and 125 mL of buffer solution pH 4.8 (5.2.3). Dilute to 250 mL.

NOTE: This reagent should be used fresh but it may be stored for at least a week, provided it is kept away from light.

**5.2.6 Nitric acid (1 + 1).** Add 500 mL of nitric acid ( $\rho_{20}$  1.42 g/mL) to 500 mL of water.

**5.2.7 Deoxygenated water.** Boil water for 10 min and bubble nitrogen through it as it cools. Continue to bubble nitrogen through the water until it is required.

**5.3 Standard solutions.**

**5.3.1 Sulphide solution (approximately 200 mg  $\text{S}^{2-}$ /L).** Dissolve 0.75 g of sodium sulphide nonahydrate ( $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$ ) in a solution containing 25 mL of glycerol and approximately 300 mL of deoxygenated water (5.2.7) in a 500 mL volumetric flask. Dilute to volume with deoxygenated water (5.2.7). This solution shall be freshly prepared and standardized by using the method described in Appendix B just prior to use.

**5.3.2 Working standardsulphide solution (approximately 10 mg  $\text{S}^{2-}$ /L).** Pipette 5.0 mL of standardized sulphide solution (5.3.1) into a 100 mL volumetric flask containing 5 mL of glycerol and dilute to volume with deoxygenated water (5.2.7). This solution shall be used immediately.

**6 APPARATUS.** The following items of apparatus are required:

**6.1 Glassware.** Volumetric glassware shall comply with the relevant Australian Standards and be used in accordance with AS 2162. All glassware shall be thoroughly rinsed with nitric acid (5.2.6), and then with water before use.

**6.2 Polyethylene bottles.** 100 mL capacity.

**6.3 Spectrophotometer.** For use at 454 nm with a cell path length of 10 mm. Spectrophotometric practice shall be in accordance with AS CK19.

**7 SAMPLES AND SAMPLING.**

**7.1 General.** Sample containers shall be 100 mL polyethylene bottles, cleaned in accordance with AS 2031.1 and containing 10 mL portions of copper-DMP reagent (5.2.5). Reserve an additional bottle containing 10 mL of copper-DMP reagent for a field blank. Zinc acetate solution shall not be used as a preservative in this method.