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Reconfirmed 2017

Australian Standard®

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
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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.

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

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Australian Water Association
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NOTES

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METHODS FOR THE ANALYSIS OF WATERS

Part 1—DETERMINATION OF DISSOLVED SULPHIDE—SPECTROPHOTOMETRIC METHOD

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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.

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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 4.5 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 (ρ_{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 (CH_3COONa) 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 ($CuSO_4.5H_2O$) 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 (ρ_{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 S^{2-}/L*). Dissolve 0.75 g of sodium sulphide nonahydrate ($Na_2S.9H_2O$) 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 standard sulphide solution* (*approximately 10 mg 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.