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WATERS—DETERMINATION OF FILTRABLE SYNTHETIC ANIONIC SURFACTANTS—COPPER-ETHYLENEDIAMINE FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

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The following interests are represented on Committee CH/22:

Australian Mining Industry Council
Confederation of Australian Industry
Department of Conservation, Forests and Lands
Department of Environment, Tas.
Department of Health, N.S.W.
Department of Housing and Construction
Department of Local Government, Qld.
Department of Science
Engineering and Water Supply Department, S.A.
Environment Protection Authority, Vic.
Melbourne and Metropolitan Board of Works
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Ministry of Conservation (Victoria)—Fisheries and Wildlife Division
National Association of Testing Authorities, Australia
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Royal Australian Chemical Institute
Rural Water Commission, Vic.
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Water Resources Commission, N.S.W.

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PREFACE

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

Synthetic anionic surfactants are determined in waters because they may be present in concentrations which are harmful to aquatic biota or produce undesirable foaming. This method is simpler than the methylene blue method and suffers fewer interferences (Appendix A). The response to the method of linear alkylsulphates of different chain lengths correlates more closely with the foaming characteristics of these compounds than does their response to either the methylene blue or azure A methods (Appendix B).

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WATERS—DETERMINATION OF FILTRABLE SYNTHETIC ANIONIC SURFACTANTS—COPPER-ETHYLENEDIAMINE FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD

1 SCOPE. This Standard sets out a flame atomic absorption spectrometric method for the determination of filtrable synthetic anionic surfactants in waters.

2 APPLICATION. The method is applicable to natural and waste waters that contain synthetic anionic surfactants in the concentration range 0.02 mg/L to 0.5 mg/L (expressed as linear alkylbenzene sulphonate, (LAS)). The lower limit of the method depends on instrument sensitivity (see Note to Clause 9.2(1)) and precision (Clause 12). The upper limit may be extended by taking smaller test sample volumes.

NOTE: Information on interferences by common cations and anions is given in Appendix A.

3 REFERENCED DOCUMENTS. The following Standards are referred to in this Standard:

AS 2031.1 Selection of Containers and Preservation of Water Samples for Chemical and Microbiological Analysis.
Part 1: Chemical.

AS 2134.1 Recommended Practice for Chemical Analysis by Atomic Absorption Spectrometry
Part 1: Flame Atomic Absorption Spectrometry.

AS 2162 Code of Practice for the Use of Volumetric Glassware.

AS 2407 Separating Funnels and Dropping Funnels.

AS 2850 Chemical Analysis—Inter-laboratory Test Programs—Guide to the Planning and Conduct—For Determining Precision of Analytical Method(s).

4 PRINCIPLE. At pH 5 to 9, neutral complexes form between synthetic surfactant anions and the bis(ethylenediamine)copper(II) ion. These complexes are extracted into chloroform and the copper they contain is back-extracted into dilute acid. The copper in the acid extract is determined by flame atomic absorption spectrometry.

5 REACTION. Formation of the extractable complexes may be represented by the following example:

$$[Cu(en)_2(H_2O)_2]^{2+} + 2RSO_2O^- \longrightarrow [Cu(en)_2(RSO_2O)_2] + 2H_2O$$

extracting cation — surfactant sulphonate anion — extracted complex

where

en = ethylenediamine ($H_2NCH_2CH_2NH_2$)

R = hydrocarbon group.

NOTE: Further information on the nature of the extracting agent and the extracted species is given in Appendix C.

6 REAGENTS.

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

6.2 Solutions.

6.2.1 *Chloroform.*

SAFETY WARNING. Care should be taken to avoid breathing chloroform vapours (carry out the handling operations in a fume cupboard) and to avoid contact with the skin.

6.2.2 *Copper-ethylenediamine reagent.* Dissolve 62 g of copper sulphate pentahydrate ($CuSO_4.5H_2O$), 50 g of ammonium sulphate ($(NH_4)_2SO_4$) and 45 g of ethylenediamine in water and dilute to 1 L.

6.2.3 *Hydrochloric acid (approx. 0.1 mol/L).* Dilute 10 mL of hydrochloric acid (ϱ_{20} 1.16 g/mL) to 1 L.

6.2.4 *Nitric acid (1 + 4).* Add 200 mL of nitric acid (ϱ_{20} 1.42 g/mL) to 800 mL of water.

6.3 Standard solutions.

6.3.1 *Stock standard LAS solution (approx. 1000 mg/L).* Weigh, to the nearest 0.1 mg, sufficient LAS standard to give on dilution, a solution containing approximately 1000 mg LAS/L. Calculate the concentration of the stock standard solution to four significant figures from the following formula:

$$c_{stock\,(mg/L)} = \frac{c \times m \times M \times 1000}{V_1}$$

where

c = concentration of LAS in the LAS standard, in millimoles per gram (mmol/g)

m = mass of LAS standard weighed, in grams

M = molecular weight of the LAS, as the sulphonic acid

V_1 = volume to which the LAS is diluted, in millilitres.

NOTES:

1. Enquiries regarding the supply of LAS standard may be directed to the Curator of Standards, Australian Government Analytical Laboratory, 11 William Street, Melbourne, Victoria 3000. LAS standard is supplied as a solution of the triethanolamine salt of Dobane 124AS sulphonate (molecular weight of the LAS, as the sulphonic acid = 323).
2. The stock standard LAS solution is stable for one week when stored at 4°C in the dark.