AS 3550.6—1990
Reconfirmed 2017

Australian Standard®

Waters

Part 6: Determination of filtrable calcium—Flame atomic absorption spectrometric method

This Australian Standard was prepared by Committee CH/22, Methods for Examination of Waters. It was approved on behalf of the Council of Standards Australia on 16 June 1989 and published on 19 January 1990.

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Australian Government Analytical Laboratories

Australian Mining Industry Council

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RECONFIRMATION

OF
AS 3550.6—1990
Waters
Part 6: Determination of filtrable calcium—Flame atomic absorption spectrometric method

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

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NOTES

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First published as AS 2526—1982.
Revised and redesignated AS 3350.6—1990.

PUBLISHED BY STANDARDS AUSTRALIA
(STANDARDS ASSOCIATION OF AUSTRALIA)
1 THE CRESCENT, HOMEBUSH, NSW 2140
ISBN 0 7262 5901 2

PREFACE

This Standard was prepared by the Standards Australia Committee on Methods for Examination of Waters under the direction of the Chemical Standards Board to supersede AS 2526—1982, *Waters—Determination of calcium—Atomic absorption spectrometric method.* The method is technically identical to AS 2526, except for the restriction in the application range (to maintain linearity of the calibration graph), the recommendation on the use of cellulose acetate filters in the sampling procedure, and minor editorial changes. This Standard forms part of the AS 3550 series of Standards on the sampling and analysis of waters.

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STANDARDS AUSTRALIA

Australian Standard

Waters

Part 6: Determination of filtrable calcium—Flame atomic absorption spectrometric method

1 SCOPE. This Standard sets out a flame atomic absorption spectrometric method for the determination of filtrable calcium in natural and waste waters. A procedure for the determination of non-filtrable calcium is described in Appendix A. Waste-waters and raw water samples are likely to contain suspended materials and may require pretreatment before analysis. Details of such pretreatment are given in AS 2031.1 and in Appendix A of this Standard.

This method is applicable to the direct determination of calcium in the concentration range 0.2 mg/L to 4 mg/L in the test solution.

2 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
2031.1 Part 1: Chemical
2134 Recommended practice for chemical analysis by atomic absorption spectrometry
2134.1 Part 1: Flame atomic absorption spectrometry
2162 Code of practice for the use of volumetric glassware
2164 One-mark volumetric flasks
2165 Burettes and bulb burettes
2166 One-mark pipettes
2850 Chemical analysis — Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct

3 PRINCIPLE. A sample of water containing calcium is aspirated into a nitrous oxide-acetylene flame of an atomic absorption spectrometer and the absorption measured at 422.7 nm.

NOTE: In the nitrous oxide-acetylene flame the main interference is caused by ionization of calcium itself (10% to 20%). This is overcome by the addition of a more readily ionizable element such as caesium or potassium to the standards, blank and sample. Few other interferences occur but, where these are anticipated, a standard addition technique can be used.

4 REAGENTS AND MATERIALS.

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

4.2 Solutions.

4.2.1 *Nitric acid* (ρ_{20} 1.42 g/mL).

4.2.2 *Hydrochloric acid* (ρ_{20} 1.16 g/mL).

4.2.3 *Hydrochloric acid* (0.1 mol/L)—dilute 10 mL of hydrochloric acid (4.2.2) to 1 L.

4.2.4 *Ionization suppressor* — prepare an aqueous solution of either 25 g/L of caesium chloride (CsCl), or 38 g/L of potassium chloride (KCl).

4.3 Standard solutions.

4.3.1 *Stock calcium solution* (2.000 g Ca/L). Dry a sample of calcium carbonate ($CaCO_3$) at 150°C for 2 h. Transfer 4.995 g into a 1 L volumetric flask using about 200 mL of water. Cautiously add 12 mL of hydrochloric acid (4.2.2). When the calcium carbonate is dissolved, dilute to volume with water and store in a polyethylene container.

4.3.2 *Standard calcium solution* (100 mg Ca/L). Pipette 50.00 mL of stock calcium solution (4.3.1) to a 1 L volumetric flask and add 10 mL of hydrochloric acid (4.2.2). Dilute to volume with water and store in a polyethylene container.

5 APPARATUS.

5.1 Volumetric glassware — Volumetric flasks shall comply with AS 2164, burettes shall comply with AS 2165 and pipettes shall comply with AS 2166. Use of volumetric glassware shall be in accordance with AS 2162.

5.2 Atomic absorption spectrometer — to be used in accordance with AS 2134.1.

5.3 Calcium hollow-cathode lamp.

5.4 Support gas—Nitrous oxide—instrument grade.

5.5 Fuel gas—Acetylene—instrument grade.

CAUTION: ACETONE (ALWAYS PRESENT IN ACETYLENE CYLINDERS) ALTERS THE RESPONSE OF THE INSTRUMENT, AND SHOULD BE PREVENTED FROM ENTERING THE BURNER SYSTEM BY REPLACING THE CYLINDER WHEN THE PRESSURE HAS FALLEN TO 600 kPa. ACETONE CAN ALSO WEAKEN POLYVINYL CHLORIDE TUBING THAT CARRIES THE ACETYLENE TO THE BURNER, CAUSING A POTENTIALLY HAZARDOUS SITUATION.

6 SAMPLES AND SAMPLING. As soon as practicable after collection, the sample shall be filtered through a 0.45 ± 0.05 μm cellulose acetate membrane filter, and acidified with nitric acid (4.2.1) to pH less than 2. If the non-filtrable calcium is to be determined, record the volume filtered (V_1) and retain the residue for treatment in accordance with Appendix A. Selection of containers and preservation of samples shall be in accordance with AS 2031.1.

7 PROCEDURE.

7.1 Optimization. The instrument settings for the 422.7 nm calcium wavelength, and for a nitrous oxide- acetylene flame shall be optimized in accordance with AS 2134.1.

7.2 Calibration. In preparing a calibration graph, the range of working standards shall be chosen in such

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