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WATERS—DETERMINATION OF BORON—CURCUMIN SPECTROPHOTOMETRIC METHOD

STANDARDS ASSOCIATION OF AUSTRALIA
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THE FOLLOWING SCIENTIFIC, INDUSTRIAL AND GOVERNMENTAL ORGANIZATIONS and departments were officially represented on the committee which prepared this standard:

- Australian Mining Industry Council
- Australian Water Resources Council
- Confederation of Australian Industry
- CSIRO, Division of Mineralogy
- Department of Housing and Construction
- Department of Science and the Environment
- Department of the Environment of Tasmania
- Engineering and Water Supply Department, S.A.
- Environment Protection Authority (Vic.)
- Health Commission of N.S.W.
- Melbourne and Metropolitan Board of Works
- Metropolitan Water Sewerage and Drainage Board, N.S.W.
- Ministry of Conservation (Vic.)—Fisheries and Wildlife Division
- National Association of Testing Authorities, Australia
- National Health and Medical Research Council
- Royal Australian Chemical Institute
- Technical Association of the Australian and New Zealand Pulp and Paper Industry (Apitta)
- Water Resources Commission, N.S.W.
- Water Quality Council of Queensland

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AUSTRALIAN STANDARD

WATERS—DETERMINATION OF BORON—CURCUMIN SPECTROPHOTOMETRIC METHOD

AS 2384—1980

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PREFACE

This standard was prepared by the Association's Committee on Methods for Examination of Waters under the direction of the Chemical Standards Board in order to provide a method for determining the concentration of boron in waters.

The level of boron is critical in irrigation waters because of its effect on citrus fruits; also from the health aspect it may indicate contamination, particularly from irrigation return waters.

This standard requires reference to the following Australian standards:

AS 2031	Recommendations for the Selection of Containers and Preservation of Water Samples for Chemical and Microbiological Analysis Part 1—Chemical
AS CK19	Code of Recommended Practice for the Chemical Analysis of Materials by Ultraviolet Visible Spectrophotometry

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1 SCOPE. This standard sets out a method for the determination of concentration of boron in waters. It is suitable for use with fresh and saline waters and wastewaters.

2 APPLICATION. The method is directly applicable to most waters having a boron level in the range 0.1 mg/L to 2.0 mg/L. The upper range can be readily extended by dilution and the minimum detectable level can be decreased by the use of longer pathlength spectrophotometric cells and/or preconcentration of the sample.

NOTES:

1. A level of calcium and magnesium hardness exceeding 100 mg/L expressed as calcium carbonate ($CaCO_3$) produces a residue of insoluble salts which imparts a turbidity to the final ethanolic solution. The interference can be removed by filtration (Clause 8.3 (e)) or by removal of the calcium and magnesium from the sample with a cation exchange resin prior to analysis (Clause 7.2).
2. Nitrate levels in excess of 20 mg nitrogen per litre interfere by oxidation of the curcumin reagent.
3. Fluoride interferes at a level in excess of 5 mg fluoride per litre, which is higher than the level generally found in natural waters.
4. Iron(III) interferes at a level in excess of 100 mg iron per litre.
5. Molybdenum compounds interfere at a level in excess of 10 mg molybdenum per litre.
6. Although titanium, niobium, tantalum and zirconium compounds give red colours with curcumin, there is no interference at the levels normally found in natural waters.
7. Where the sample contains sufficient suspended matter to cause blockage of the ion exchange column, filtration or centrifugation is required prior to the determination.

3 PRINCIPLE. Boron is reacted with curcumin in the presence of oxalic acid to form a red complex which is measured spectrophotometrically at 540 nm.

4 REACTION. The reaction between boron (as boric acid) and curcumin proceeds at a measurable rate only when the curcumin molecule is protonated and this requires the presence of a strong acid. In the protonated form, however, curcumin is unstable and slowly decomposes with moderate heating. The technique utilized in this standard employs the formation of the curcumin-boron-oxalate complex by the controlled evaporation of an acidified sample of water in the presence of the curcumin reagent. For optimum performance of the method, careful attention must be paid to the sample volume, reagent volumes and concentrations, the temperature of evaporation, and the shape, size and composition of evaporating vessels, because the following reaction conditions must be provided:

(a) Acidity must increase during evaporation to protonate the curcumin. Curcumin changes from pale yellow to deep red when protonated.

(b) Protonation must occur early enough to allow for complete reaction with the boric acid before dryness is reached.

(c) The protonated curcumin must not be formed too early otherwise significant decomposition of the reagent would occur at the temperature employed for evaporation.

Protonated curcumin has an absorption maximum at 555 nm and therefore it is necessary to dissociate excess reagent on completion of the reaction and prior to colour measurement. Excess mineral acid is removed by evaporation and excess protonated curcumin dissociates when the deposit is redissolved in ethanol.

5 REAGENTS.

5.1 General Requirements. Use only analytical grade reagents and water purified so as to give a negligible blank.

5.2 Reagents.

5.2.1 *Ethanol* (950 mL/L).

5.2.2 *Curcumin reagent.* Dissolve 0.040 g of finely ground curcumin ($C_{21}H_{20}O_6$, M_r* 368.39) and 5.0 g of oxalic acid ($C_2H_2O_4$, M_r 90.04) in 80 mL ethanol (5.2.1). Add 4.2 mL of concentrated hydrochloric acid (ρ_{20} 1160 kg/m^3) and make the solution up to 100 mL with ethanol (5.2.1) in a 100 mL volumetric flask.

NOTE: This reagent is stable for several days if stored at 4°C although the absorbance of standards may increase slightly with the time of storage of the reagent.

5.2.3 *Reagents for removal of calcium and magnesium hardness by ion exchange.*

(a) *Strongly acid cation exchange resin.*

(b) *Hydrochloric acid* (200 mL/L). Dilute 200 mL of concentrated hydrochloric acid (ρ_{20} 1160 kg/m^3) to 1 L.

5.2.4 *Stock boron solution* (100 mg boron per litre). Dissolve 0.5716 g of anhydrous boric acid (H_3BO_3) in water and dilute to 1 L in a volumetric flask.

NOTE: As boric acid loses mass on drying at 105°C, it should be used without heating. Care is required to reseal the bottle to prevent absorption of moisture.

5.2.5 *Standard boron solution* (1 mg boron per litre). Dilute 10.00 mL of stock boron solution (5.2.4) to 1 L in a volumetric flask.

6 APPARATUS.

6.1 For Sample Preparation.

6.1.1 *Glass tube,* 500 mm long, 12 mm internal diameter, fitted with a porous glass frit and a tap.

6.1.2 *Suitable membrane filter holder and filters* (mean pore size 0.45 ± 0.05 µm), attached to a 100 mL glass vacuum flask.

6.2 For Colour Development.

6.2.1 *Evaporating dishes,* 100 mL to 150 mL capacity, made of porcelain, boron-free glass, silica, polytetrafluoroethylene (PTFE) or platinum. The dishes used shall have a semicircular profile and be identical in shape, size and material of construction.

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*Relative molecular mass.