

Australian Standard[®]

**WATERS—
DETERMINATION OF
URANIUM(VI)—
SPECTROPHOTOMETRIC
METHOD**

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The following interests are represented on Committee CH/22:

- Australian Mining Industry Council
 - Australian Water Resources Council
 - Confederation of Australian Industry
 - CSIRO, Division of Energy Chemistry
 - Department of Health, N.S.W.
 - Department of Housing and Construction
 - Department of Home Affairs and the Environment
 - Department of the Environment, Tasmania
 - Department of Science and Technology
 - Engineering and Water Supply Department, S.A.
 - Environment Protection Authority (Victoria)
 - Melbourne and Metropolitan Board of Works
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 - National Association of Testing Authorities, Australia
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PREFACE

This standard was prepared by the Association's Committee on Methods for Examination of Waters.

It describes a method in which the concentration of uranium(VI) is determined spectrophotometrically either directly or, when the concentration of interfering metal ions is too high for effective masking, by employing a simple chelating resin separation pretreatment.

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

for

WATERS—DETERMINATION OF URANIUM(VI)—SPECTROPHOTOMETRIC METHOD

1 SCOPE. This standard sets out a spectrophotometric method for the determination of filtrable uranium(VI) in waters.

2 APPLICATION. The method is directly applicable to fresh waters with uranium(VI) in the range 0.12 mg/L to 16 mg/L and to saline waters and seawater with uranium(VI) in the range 0.3 mg/L to 40 mg/L.

A preliminary chelating resin separation is applicable to polluted waters and waste waters with uranium(VI) in the range 0.12 mg/L to 16 mg/L.

NOTES:

1. The inclusion of the chelating resin separation step reduces the interference effects of many heavy metals, e.g. 2 mg of iron(III), 5 mg of copper(II) and 5 mg of chromium(III) in the portion applied to the column can be tolerated.

Phosphate interferes because it complexes uranium, thus reducing its retention on the resin. Up to 30 µg phosphate can be tolerated in the test portion, and up to 500 µg phosphate can be present when 1 mg of thorium(IV) is added to preferentially complex the phosphate.

2. The test portion should contain less than 0.01 mg of vanadium(V). However, up to 0.4 mg of vanadium(V) can be tolerated by reduction to vanadium(IV) with ascorbic acid.

3. Iron(III) interferes, and this effect is increased when magnesium(II) is present because it reduces the ability of the complexing solution to mask iron(III). The test portion should contain both less than 15 mg of magnesium(II) and less than 0.5 mg of iron(III).

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

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|---------|---|
| AS 2031 | Recommendations for the Selection of Containers and Preservation of Water Samples for Chemicals and Microbiological Analysis
Part 1—Chemical |
| AS 2162 | Code of Practice for the Use of Volumetric Glassware |
| AS CK19 | Code of Recommended Practice for the Chemical Analysis of Materials by Ultraviolet Visible Spectrophotometry |
| BS 3875 | Specification for Optical Spectrophotometric Cells. |

4 PRINCIPLE.

4.1 Direct spectrophotometric method (Reference 1). 4-(2-pyridylazo) resorcinol (PAR) forms strongly-coloured, water-soluble complexes with many metal ions. The 1:1 uranium(VI)-PAR complex formed at pH 8 is stable and its absorbance is measured at 530 nm.

As PAR is non-selective as a chromogenic reagent, interfering metal ions must be effectively masked to provide a direct determination of uranium(VI). The chelating agent (1,2-cyclohexylenedinitrilo) tetraacetic acid (CyDTA) forms stable complexes with most metals and prevents their reaction with PAR, but uranium(VI) reacts weakly with CyDTA and subsequent complex formation with PAR is only slightly affected. Sulphosalicylate and fluoride are also added to the masking solution to aid in the complexing of aluminium(III) and thorium(IV) respectively.

4.2 Chelating resin separation (Reference 2). When the concentration of the interfering metal ions is too high for effective masking (e.g. for polluted or waste waters), uranium(VI) may be separated by a simple chelating resin procedure. In the presence of CyDTA, most metals are complexed in solution and are not retained by a column of iminodiacetate chelating resin. Uranium(VI) is, however, adsorbed on the resin and can subsequently be eluted with dilute acid.

5 MATERIALS AND REAGENTS.

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

5.2 Reagents.

5.2.1 Sodium hydroxide (1.0 mol/L). Dissolve 40 g of sodium hydroxide in 200 mL of water. Cool and dilute to 1 L. Store in a polyethylene container.