AS 2489.3—1981

Australian Standard®

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

Part 3—DETERMINATION OF TITANIUM CONTENT (SPECTROPHOTOMETRIC METHOD)

The following industrial, scientific and governmental organizations were officially represented on the committee entrusted with the preparation of this standard:

Australian Foundry Institute
Australian Mineral Development Laboratories
Chamber of Mines of W.A. (Incorporated)
CSIRO, Division of Mineral Chemistry
CSIRO, Division of Mineralogy
Mineral Sands Producers Association

This standard, prepared under the direction of Committee MN/4, Heavy Mineral Sands, was approved on behalf of the Council of the Standards Association of Australia on 28 July 1981, and was published on 14 September 1981.

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PREFACE

This standard was prepared by the Association's Committee on Heavy Mineral Sands in order to provide a method for use in settling disputes arising from discrepancies between buyer and seller in the determination of titanium in zircon sand concentrates.

The committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the method. Laboratories from the following organizations participated in the test program to provide the data given in Table 1:

Australian Mineral Development Laboratories
Cable Sands
Government Chemical Laboratories, W.A.
Jennings Mining
LaPorte Australia
Westralian Sands

This standard requires reference to the following standards:

AS CK 19	Code of Recommended Practice for the Chemical Analysis of Materials by Ultraviolet/Visible Spectrophotometry
BS 3875	Optical Spectrophotometric Cells
BS 4237	Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry

First published 1981

This standard was issued in draft form for public review as DR 80169.

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ISBN 0 7262 2356 5

STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

PART 3—DETERMINATION OF TITANIUM CONTENT (SPECTROPHOTOMETRIC METHOD)

1 SCOPE. This standard sets out a spectrophotometric method, using hydrogen peroxide for colour development, for the determination of the titanium content of zircon sand concentrates.

2 APPLICATION. The method is applicable to zircon sand concentrates with a titanium content, expressed as titanium dioxide, of 0.02 percent to 0.6 percent TiO_2. Iron interferes above 0.3 percent Fe_2O_3 content and titanium values should be corrected by using a compensating solution.

3 PRINCIPLE. Decomposition of the test portion by fusion with sodium tetraborate/potassium carbonate flux and dissolution of the melt with dilute sulphuric acid. Development of the titanium(IV) complex by the addition of hydrogen peroxide and measurement of the absorbance of the resultant yellow solution at a wavelength of approximately 410 nm.

4 REAGENTS.

4.1 General. During the analysis use only reagents of recognized analytical reagent grade and distilled water or water of equivalent purity.

4.2 Solids.

4.2.1 Sodium tetraborate, anhydrous ($Na_2B_4O_7$).

4.2.2 Potassium carbonate, anhydrous.

4.3 Solutions.

4.3.1 Sulphuric acid (ρ_{20} 1840 kg/m^3), diluted 1 + 1.

4.3.2 Sulphuric acid (ρ_{20} 1840 kg/m^3), diluted 1 + 6.

4.3.3 Hydrogen peroxide, 300 g/L.

4.4 Standard Titanium Solutions.

4.4.1 Standard solution A ($1\ mL \equiv 250\ \mu g\ TiO_2$). Dry standard titanium dioxide (NBS 154b, 99.74 percent TiO_2) at 110°C for 2 h. Transfer to a desiccator and allow to cool.

Weigh, to the nearest 0.0002 g, 0.2507 g of the dry titanium dioxide and transfer to a platinum crucible containing 2.5 g of sodium tetraborate (4.2.1) and 1.0 g of potassium carbonate (4.2.2). Mix intimately.

Heat the mixture vigorously until fusion is complete. Allow the melt to cool and then tap the crucible to transfer the melt to a 250 mL beaker. Add a little sulphuric acid (4.3.2) to the crucible and heat gently to remove any adhering melt. Carefully wash the contents of the crucible into the 250 mL breaker. Add 60 mL of sulphuric acid (4.3.1) and heat gently until the melt is completely dissolved.

Cool the solution and transfer it to a 1 L volumetric flask. Add 140 mL of sulphuric acid (4.3.1) and dilute to the mark with water. (Solution A).

4.4.2 Standard solution B ($1\ mL \equiv 50\ \mu g\ TiO_2$). Pipette 50.00 mL of standard solution A (4.4.1) into a 250 mL volumetric flask containing 60 mL of sulphuric acid solution (4.3.1) and dilute to the mark with water. (Solution B).

5 APPARATUS.

5.1 Ordinary laboratory apparatus.

5.2 Volumetric glassware. Grade A volumetric glassware complying with the relevant Australian standards shall be used throughout.

5.3 Crucibles. Platinum or platinum/gold crucibles of 25 mL minimum capacity with cover.

5.4 Laboratory stirrer-hotplate.

5.5 Spectrophotometer. A spectrophotometer suitable for measurement of absorbance in the region of 410 nm. In the absorbance range 0.0 to 1.0, absorbance measurements shall be repeatable to ± 0.002 absorbance units. Spectrophotometric practice shall be in accordance with AS CK19.