

Australian Standard<sup>®</sup>

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**METHODS FOR THE ANALYSIS OF  
ZIRCON SAND CONCENTRATES**

**Part 10—DETERMINATION OF  
SILICON CONTENT  
(COMBINED  
GRAVIMETRIC AND  
SPECTROPHOTOMETRIC  
METHOD)**

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This Australian standard was prepared by Committee MN/4, Heavy Mineral Sands. It was approved on behalf of the Council of the Standards Association of Australia on 29 March 1984 and published on 2 July 1984.

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The following interests are represented on Committee MN/4:

- Australian Foundry Institute
- Chamber of Mines of W.A. (Incorporated)
- CSIRO, Division of Mineral Chemistry
- CSIRO, Division of Mineralogy
- NSW Chamber of Mines and Extractive Industries
- Oil and Colour Chemists Association Australia

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## PREFACE

This standard was prepared by the Association's Committee on Heavy Mineral Sands under the direction of the Minerals Standards Board in order to provide a method for use in the settling of disputes arising from discrepancies between buyer and seller in the determination of silicon 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 obtain the results given in Table 1:

- ARM Laboratories
- Associated Minerals Consolidated Limited, Southport
- Australian Laboratory Services
- CSIRO, Division of Mineral Chemistry
- Government Chemical Laboratories, W.A.

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## STANDARDS ASSOCIATION OF AUSTRALIA

## Australian Standard

## METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

PART 10—DETERMINATION OF SILICON CONTENT  
(COMBINED GRAVIMETRIC AND SPECTROPHOTOMETRIC METHOD)

**1 SCOPE.** This standard sets out a gravimetric method (in conjunction with a reduced molybdsilicate spectrophotometric method) for the determination of the silicon content of zircon sand concentrates.

**2 APPLICATION.** The method is applicable to zircon sand concentrates containing between 30 percent and 35 percent  $\text{SiO}_2$ .

**3 REFERENCED DOCUMENTS.** The following standard is referred to in this standard:  
BS 4237 Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry.

**4 PRINCIPLE.** Decomposition of the test portion by sintering with a mixture of sodium carbonate and sodium peroxide. Leaching and evaporation with sulphuric acid, dilution, addition of gelatin, and filtration of silica. Ignition, treatment with hydrofluoric acid, evaporation to dryness, and calculation of silica content from the loss of mass. Spectrophotometric determination of residual soluble silica in the silica filtrate.

**5 REAGENTS.**

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

**5.2 Solids.**

**5.2.1** Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) anhydrous, fine powder.

NOTE: The water content should be less than 1 percent, determined by heating at 500°C for 1 h. Dry under the same conditions if necessary.

**5.2.2** Sodium peroxide ( $\text{Na}_2\text{O}_2$ ), fine powder.

**5.2.3** Borate-carbonate flux. Mix intimately in a clean mortar, 5 parts by mass of anhydrous sodium tetraborate ( $\text{Na}_2\text{B}_4\text{O}_7$ ) and 3 parts by mass of anhydrous potassium carbonate ( $\text{K}_2\text{CO}_3$ ).

**5.3 Solutions.**

**5.3.1** Sulphuric acid ( $\rho_{20}$  1840  $\text{kg/m}^3$ ) diluted 1 + 1.

**5.3.2** Sulphuric acid ( $\rho_{20}$  1840  $\text{kg/m}^3$ ) diluted 1 + 3.

**5.3.3** Hydrofluoric acid ( $\rho_{20}$  1160  $\text{kg/m}^3$ ).

**5.3.4** Sodium hydroxide solution (250 g/L). Prepare in a plastics beaker. Cool and store in a plastics container.

**5.3.5** Gelatin solution (0.50 percent m/V). Dissolve 5 g of gelatin and a pea-sized crystal of thymol in 600 mL of water with warming, cool, dilute to 1 L and mix.

**5.3.6** Ammonium molybdate [ $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ ] solution, 10 percent m/V. Dissolve 10 g of ammonium molybdate in 70 mL of water with warming if necessary. Cool, dilute to 100 mL and mix.

Store in a plastics container and discard when precipitation occurs.

**5.3.7** Ascorbic acid ( $\text{C}_6\text{H}_8\text{O}_6$ ) solution, 5 percent m/V. Dissolve 2.5 g of ascorbic acid in 50 mL of water. Prepare freshly for each series of tests.

**5.4 Standard silica solutions.**

**5.4.1** *Standard solution A* (1 mL  $\equiv$  500  $\mu\text{g}$   $\text{SiO}_2$ ). Ignite in a platinum crucible approximately 0.12 g of pure silica at 1000°C for 30 min and cool in a desiccator. Weigh 0.100 g into a platinum crucible, add 1 g of anhydrous sodium carbonate and fuse at 1000°C until a clear melt is obtained. Cool, leach in water and transfer the solution to a 200 mL volumetric flask. Dilute to volume with water, and mix. Store in a plastics container.

**5.4.2** *Standard solution B* (1 mL  $\equiv$  20 mg  $\text{SiO}_2$ ). Measure 10.00 mL of standard solution A into a 250 mL volumetric flask, dilute to volume with water, and mix.