Australian Standard®

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

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

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.

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

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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1 SCOPE. This standard sets out a gravimetric method (in conjunction with a reduced molybdosilicate 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 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 (Na_2CO_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 (Na_2O_2), fine powder.

5.2.3 Borate-carbonate flux. Mix intimately in a clean mortar, 5 parts by mass of anhydrous sodium tetraborate ($Na_2B_4O_7$) and 3 parts by mass of anhydrous potassium carbonate (K_2CO_3).

5.3 Solutions.

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

5.3.2 Sulphuric acid (ρ_{20} 1840 kg/m$_3$) diluted 1 + 3.

5.3.3 Hydrofluoric acid (ρ_{20} 1160 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 [$(NH_4)_6Mo_7O_{24}.4H_2O$] 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 ($C_6H_8O_6$) 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 ≡ 500 μg 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 ≡ 20 mg SiO_2). Measure 10.00 mL of standard solution A into a 250 mL volumetric flask, dilute to volume with water, and mix.