Australian Standard®

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

Part 8—DETERMINATION OF CALCIUM CONTENT

(FLAME ATOMIC ABSORPTION SPECTROMETRIC 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 28 June 1983 and published on 5 September 1983.

The following interests are represented on Committee MN/4:

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

PREFACE

This standard was prepared by the Association's Committee on Heavy Mineral Sands under the direction of the Minerals Standards Committee in order to provide a method for use in the settling of disputes arising from discrepancies between buyer and seller in the determination of calcium 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, Capel
Australian Laboratory Services
Laporte Australia Ltd
Mineral Deposits Ltd

First published 1983

This standard was issued in draft form for comment as DR 82145.

ISBN 0 7262 3083 9

STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

PART 8—DETERMINATION OF CALCIUM CONTENT (FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD)

1 SCOPE. This standard sets out a method for the determination of calcium in zircon sand concentrates using flame atomic absorption spectrometry.

2 APPLICATION. The method is applicable to zircon sand concentrates containing between 0.005 percent and 0.06 percent CaO.

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

AS 2134 Code of Practice for the Chemical Analysis of Materials by Flame Atomic Absorption Spectroscopy

AS 2162 Code of Practice for the Use of Volumetric Glassware

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 fusion with sodium tetrafluoroborate. Dissolution of the melt with sulphuric acid and hydrofluoric acid and removal of fluoride and silica by fuming. Aspiration of the solution into a nitrous oxide/acetylene flame and measurement of the atomic absorption at 422.7 nm.

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 tetrafluoroborate ($NaBF_4$).

NOTE: Sodium tetrafluoroborate of a suitable analytical reagent grade is commercially available. Alternatively a material with low impurity level may be prepared from the laboratory reagent grade by recrystallization (see Appendix A).

5.2.2 Zirconium dioxide (ZrO_2). Zirconium dioxide powder containing less than 0.0005 percent CaO.

5.3 Solutions.

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

5.3.2 Hydrofluoric acid (ρ_{20} 1150 kg/m^3).

5.3.3 Nitric acid (ρ_{20} 1420 kg/m^3), diluted 1 + 4.

5.4 Standard Calcium Solutions.

5.4.1 *Standard solution A* (1 mL ≡ 200 μg Ca). Dissolve 0.4995 g of calcium carbonate in 25 mL of nitric acid (5.3.3). Transfer to a 1000 mL volumetric flask, dilute to volume and mix.

5.4.2 *Standard solution B* (1 mL ≡ 20 μg Ca). Pipette 50 mL of solution A into a 500 mL volumetric flask. Dilute to volume and mix.

6 APPARATUS.

6.1 Ordinary laboratory apparatus.

6.2 Glassware. All glassware should be cleaned by soaking with chromic acid or nitric acid (diluted 1 + 2) then rinsing well with tap water and finally rinsing with distilled water.

6.3 Volumetric glassware. Grade A volumetric glassware complying with the relevant Australian standards to be used in accordance with AS 2162.

6.4 Platinum, or preferably platinum/gold, crucibles or basins. With a minimum capacity of 25 mL.