Australian Standard®

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

Part 1—DETERMINATION OF PHOSPHORUS 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.

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 phosphorus 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:

Associated Minerals Consolidated Ltd
Australian Mineral Development Laboratories
CSIRO, Division of Mineral Chemistry
Mineral Deposits Ltd
R.K Newman and Co. Pty Ltd

This standard requires reference to the following standards:

AS CK19	Code of Recommended Practice for the Chemical Analysis of Materials by Ultravoilet Visible Spectrophotometry
BS 3875	Optical Spectrophotometric Cells
BS 4237	Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry

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METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

PART 1—DETERMINATION OF PHOSPHORUS CONTENT (SPECTROPHOTOMETRIC METHOD)

1 SCOPE. This standard sets out a reduced molybdophosphate spectrophotometric method for the determination of the phosphorus content of zircon sand concentrates.

2 APPLICATION. The method is applicable to zircon sand concentrates containing between 0.01 percent and 0.5 percent P_2O_5.

3 PRINCIPLE. Decomposition of the test portion by fusion with sodium tetrafluoroborate. Dissolution of the melt with dilute sulphuric and boric acids and removal of fluoride and silica by evaporation.

Removal of zirconium from an aliquot by chloroform extraction of the cupferron complex. Addition of ammonium molybdate, reduction with ascorbic acid to form the molybdenum blue complex, and spectrophotometric measurement at approximately 825 nm.

4 REAGENTS.

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

4.2 Solids.

4.2.1 Sodium tetrafluoroborate ($NaBF_4$), containing less than 0.002 percent P_2O_5.

NOTE: If the P_2O_5 content of the sodium tetrafluoroborate is greater than 0.002 percent it should be recrystallized by the procedure given in Appendix A.

4.2.2 Ascorbic acid ($C_6H_8O_6$).

4.3 Solutions.

4.3.1 Chloroform ($CHCl_3$).

4.3.2 Sulphuric acid (ρ_{20} 1840 kg/m^3), diluted 1 + 1. To 500 mL of water carefully add with stirring 500 mL of sulphuric acid.

4.3.3 Sulphuric acid (ρ_{20} 1840 kg/m^3), diluted 1 + 39. To 975 mL of water carefully add with stirring 2 mL of sulphuric acid.

4.3.4 Boric acid (H_3BO_3) solution, 25 g/L.

4.3.5 Cupferron ($C_6H_5N(NO)ONH_4$) solution, 10 g/100 mL. Dissolve 10 g of pure reagent (white crystals or not darker than a pale buff colour) in 100 mL water.

4.3.6 Ammonium molybdate ($(NH_4)_6MO_7O_{24}.4H_2O$) solution, 20 g/L.

4.4 Standard Phosphorus Solutions.

4.4.1 Standard solution A (1 mL ≡ 1 200 µg P_2O_5). Dry potassium dihydrogen orthophosphate (KH_2PO_4) at 110°C for 1 h and cool in a desiccator. Dissolve 1.150 g in water, transfer to a 500 mL volumetric flask, dilute to volume and mix. (Solution A).

4.4.2 Standard solution B (1 mL ≡ 120 µg P_2O_5). Pipette 10 mL of solution A into a 100 mL volumetric flask. Dilute to volume and mix. (Solution B).

4.4.3 Standard solution C (1 mL ≡ 6 µg P_2O_5). Pipette 10 mL of solution B into a 200 mL volumetric flask. Dilute to volume and mix. (Solution C).

5 APPARATUS.

5.1 Ordinary laboratory apparatus.

5.2 Glassware. All glassware used in the test for the first time shall be cleaned before use by soaking overnight in chromic acid cleaning mixture and rinsing well with tap water and finally with distilled water. Such cleaned glassware shall be reserved and labelled 'P_2O_5 determination only'.

After each use, brief soaking with chromic acid cleaning mixture or nitric acid diluted 1 + 2 prior to final rinsing will be adequate. On no account should detergents, which may contain phosphates, be used.