Australian Standard®

METHODS FOR THE ANALYSIS OF ZIRCON SAND CONCENTRATES

Part 2—DETERMINATION OF IRON 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 iron 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 Tables 1 and 2:

Associated Minerals Consolidated Ltd
Australian Laboratory Services Pty Ltd
Mineral Deposits Ltd
Westralian Sands Ltd

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.

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PART 2—DETERMINATION OF IRON CONTENT (SPECTROPHOTOMETRIC METHOD)

1 SCOPE. This standard sets out a spectrophotometric method using the tris (1,10-phenanthroline) iron(II) complex for the determination of the iron content of zircon sand concentrates.

2 APPLICATION. The method is applicable to zircon sand concentrates containing between 0.02 percent and 0.4 percent Fe_2O_3. Aluminium does not interfere.

3 PRINCIPLE. Decomposition of the test portion by—

(a) fusion with sodium tetraborate 10-water/potassium carbonate mixture followed by dissolution of the melt with sulphuric acid; or

(b) fusion with anhydrous sodium tetraborate/potassium carbonate mixture followed by dissolution of the melt with sulphuric acid; or

(c) fusion with anhydrous sodium tetrafluoroborate followed by dissolution of the melt in sulphuric acid and fuming to remove fluorides.

NOTE: Method (a) is the preferred method of decomposition.

Reduction of the iron to iron(II) and formation of the tris(1,10-phenanthroline)iron(II) complex at a pH of 2.5 to 3.0 followed by spectrophotometric measurement of the coloured complex at a wavelength of approximately 505 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 tetraborate, anhydrous ($Na_2B_4O_7$).

4.2.2 Sodium tetraborate, 10-water ($Na_2B_4O_7.10H_2O$).

4.2.3 Potassium carbonate (K_2CO_3).

4.2.4 Sodium tetrafluoroborate ($NaBF_4$).

NOTE: Sodium tetrafluoroborate may not be readily available in analytical reagent grade. A material of suitably low impurity level may be prepared from the laboratory reagent grade by recrystallization (see Appendix A).

4.2.5 Zirconium dioxide (ZrO_2). Zirconium dioxide powder containing less than 0.001 percent iron.

4.3 Solutions.

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

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

4.3.3 Hydrochloric acid (ρ_{20} 1160 kg/m^3), diluted 1 + 1.

4.3.4 Ascorbic acid solution, 10 g/100 mL (freshly prepared).

4.3.5 Ammonia solution (ρ_{20} 880 kg/m^3), diluted 1 + 1.

4.3.6 1,10-phenanthroline hydrate solution ($C_{12}H_8N_2.H_2O$), 0.5 g/100 mL. Dissolve 0.5 g of reagent in 100 mL of hydrochloric acid (1 + 99).

4.4 Standard Iron Solutions.

4.4.1 Standard solution A (1 mL ≡ 1 mg Fe_2O_3). Dissolve 0.6994 g of Specpure iron wire in 10 mL of hydrochloric acid (4.3.3). Transfer to a 1000 mL volumetric flask, dilute to volume and mix. (Solution A).

4.4.2 Standard solution B (1 mL ≡ 0.10 mg Fe_2O_3). Pipette 50 mL of solution A into a 500 mL volumetric flask. Dilute to volume and mix. (Solution B).

5 APPARATUS.

5.1 Ordinary laboratory apparatus.

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