

Australian Standard[®]

**METHODS FOR THE ANALYSIS OF
RUTILE**

**Part 1—DETERMINATION OF
TITANIUM CONTENT—
TITRIMETRIC 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 4 November 1986 and published on 5 January 1987.

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
 - Government Chemical Laboratories, W.A.
 - N.S.W 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 settling disputes arising from discrepancies between buyer and seller in the determination of titanium in rutile.

The Committee organized an interlaboratory test program to obtain information on the precision of the method. The following laboratories participated in the test program to provide the data given in Table 1:

ARM Laboratories
 Australian Laboratory Services
 CSIRO, Division of Mineral Chemistry
 Government Chemical Laboratories, W.A.
 SCM Chemicals Ltd
 Westralian Sands Ltd



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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS OF RUTILE

PART 1—DETERMINATION OF TITANIUM CONTENT—TITRIMETRIC METHOD

1 SCOPE. This standard sets out a titrimetric method using aluminium reduction and ammonium iron(III) sulphate titration for the determination of the titanium content of rutile.

The method is applicable to rutile concentrates containing in excess of 90 percent TiO_2 . The presence of chromium, vanadium and niobium, each at concentrations of up to 1 percent (expressed as oxide) has no effect on the result.

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

AS 2884	Heavy Mineral Sand Concentrates— Sampling Part 1—Sampling from Moving Streams* Part 2—Sampling from Stationary Situations* Part 3—Preparation of Samples
ISO 4791/1	Laboratory apparatus—Vocabulary relating to apparatus made essentially from glass, porcelain or vitreous silica— Part 1: Names for items of apparatus.

3 PRINCIPLE. Sintering of the test portion with sodium peroxide followed by leaching and filtration to remove chromium and vanadium. Two-stage wet ashing of the filter paper and titanium product with sulphuric acid and sodium hydrogen sulphate (or sodium pyrosulphate) and fusion of the product with sodium hydrogen sulphate (or sodium pyrosulphate). Dissolution in sulphuric and hydrochloric acids and reduction of the titanium with aluminium metal in the presence of citric acid added to overcome interference from niobium. Titration with ammonium iron(III) sulphate solution using potassium thiocyanate indicator.

4 REAGENTS.

4.1 General. During the analysis, use only reagents of recognized analytical reagent grade (except for 4.2.3 and 4.2.4), and only distilled water or water of equivalent purity.

4.2 Solids.

4.2.1 Sodium peroxide (Na_2O_2), fine powder.

NOTE: Sodium peroxide should be kept away from humidity and should not be used once it has begun to agglomerate.

4.2.2 Sodium hydrogen sulphate (NaHSO_4), anhydrous or sodium pyrosulphate ($\text{Na}_2\text{S}_2\text{O}_7$) powder.

4.2.3 Iron powder, laboratory reagent grade.

4.2.4 Aluminium metal, foil, wire, rod or thin sheet, laboratory reagent grades, or high-purity aluminium welding rod.

NOTES:

- Cut foil into 1 g pieces and fold each piece into a tight pack about 13 mm square. Cut thin sheet (0.5 mm to 0.7 mm) into

pieces, each of 1 g mass. Cut wire (3 mm diameter) or rod (3.2 mm diameter) into 1 g lengths. Alternatively, wire or rod may be used as a single 4 g length formed into a helix or bent into any shape through more than one plane.

- The aluminium must be tested for rate of solubility under the conditions of the test and 4 g must dissolve completely in 15 to 30 min. The titre of the blank test should not exceed 0.2 mL and must be consistent throughout the lot being used.

4.2.5 Citric acid ($\text{H}_3\text{C}_6\text{H}_5\text{O}_7 \cdot \text{H}_2\text{O}$).

4.3 Solutions.

4.3.1 Hydrochloric acid ρ_{20} 1160 to 1190 kg/m^3 .

4.3.2 Sulphuric acid ρ_{20} 1840 kg/m^3 .

4.3.3 Sulphuric acid ρ_{20} 1840 kg/m^3 , diluted 1 + 1.

4.3.4 Sulphuric acid ρ_{20} 1840 kg/m^3 , diluted 1 + 9.

4.3.5 Iron solution 1 g Fe/100 mL. Dissolve 1 g of iron powder (4.2.3) in 5 mL of hydrochloric acid (4.3.1) diluted with 5 mL of water in a covered beaker with heating. Add 10 mL of water, then add slowly 1.5 mL of hydrogen peroxide (30 percent) and heat to decompose any excess. Cool, dilute with water to 100 mL and mix.

4.3.6 Potassium thiocyanate solution, 20 g/100 mL.

4.3.7 Ammonium iron(III) sulphate [$\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$] solution, 0.13 M. Transfer to a 3 L flat-bottom Florence flask marked at 2 L, 125.3 g of ammonium iron(III) sulphate. Add approximately 1.8 L of water and 30 mL of sulphuric acid (4.3.2) and stir until dissolved. Add potassium permanganate solution 3 g/100 mL, dropwise, until slight excess is present then heat until boiling freely. Cover, allow to cool thoroughly and dilute to the 2 L mark. Store in a brown glass bottle or away from light.

4.4 Nitrogen, oxygen-free grade.

5 APPARATUS. The following apparatus is required:

NOTE: The apparatus and techniques specified in this standard for the reduction and titration of titanium have been found to be satisfactory. Other apparatus and techniques may be substituted provided that the reduction reaction is well controlled and adequate precautions are taken to prevent adventitious oxidation of reduced titanium. If a 500 mL conical flask is used for the reduction in conjunction with the use of aluminium metal in the form of wire or rod, a single length of the metal (4.2.4) may be preferable in order to confine the reaction to the lower region of the flask.

5.1 Ordinary laboratory apparatus.

5.2 Zirconium crucibles, 45 mm diameter, approximately 25 mL capacity.

NOTE: Between each use, crucibles should be burnished with 600 type wet-and-dry emery paper to ensure good release of sinters.

* In course of preparation.