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Australian Standard®

ALUMINIUM ORES—DETERMINATION OF ALUMINIUM, SILICON, IRON, TITANIUM AND PHOSPHORUS CONTENTS—WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRIC METHOD

This Australian standard was prepared by Committee MN/3, Aluminium Ores. It was approved on behalf of the Council of the Standards Association of Australia on 6 July 1982 and published on 18 October 1982.

The following interests were represented on the committee responsible for the preparation of this standard:

- CSIRO, Division of Mineral Chemistry
- CSIRO, Division of Soils
- Producers of aluminium ores
- Processors of aluminium ores
- Producers of aluminium products

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PREFACE

This standard was prepared by the Association's Committee on Aluminium Ores under the direction of the Minerals Standards Committee. It is the first standard of its type in which an absolute method has been prepared for the analysis of a material by X-ray fluorescence spectrometry.

X-ray fluorescence spectrometry is in common use for in-house control of the quality of aluminium ore. The method contained in this standard is similar to that used in many laboratories analysing aluminium ores by X-ray fluorescence spectrometry.

The standard has been prepared to enable X-ray fluorescence methods to be specified in contracts.

The results of the test program have shown that the X-ray fluorescence method compares favourably with the results obtained using traditional wet chemical methods.

The committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the method. The following laboratories participated in the test program:

Analabs Pty Ltd
The Australian Mineral Development Laboratories
Australian National University, School of Geology
The Broken Hill Proprietary Co. Ltd, Central Research Laboratories
Bureau of Mineral Resources, Geology and Geophysics
CSIRO, Division of Soils
CSR Central Laboratory
Government Chemical Laboratories, Western Australia
Macquarie University, School of Earth Sciences
Queensland Alumina Limited
SGS Australia Ltd
University of Adelaide, School of Geology
The Zinc Corporation, Limited

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Australian Standard

ALUMINIUM ORES—DETERMINATION OF ALUMINIUM, SILICON, IRON, TITANIUM AND PHOSPHORUS CONTENTS—WAVELENGTH DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRIC METHOD

1 SCOPE AND APPLICATION.

1.1 Scope. This standard sets out a method for the determination of the contents of aluminium, silicon, iron, titanium, and phosphorus (expressed as the oxides Al_2O_3, SiO_2, Fe_2O_3, TiO_2, and P_2O_5) in aluminium ores by wavelength dispersive X-ray fluorescence spectrometry. The method has been designed to cope with the high and variable loss on ignition of aluminium ores.

1.2 Application. The method is applicable to all normal aluminium ores regardless of mineralogical type. The concentration range covered for each of the components is given in Table 1.

TABLE 1

RANGE OF APPLICATION OF THE METHOD

Component	Concentration range percent
Al_2O_3	30—65
SiO_2	1—25
Fe_2O_3	2—50
TiO_2	0.5—8
P_2O_5	0.02—5
Loss on ignition	10—35

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

AS 2563	Wavelength Dispersive X-ray Fluorescence Spectrometers—Methods of Test for Determination of Precision
AS XXXX	Aluminium Ores—Determination of Hygroscopic Moisture in Analytical Samples—Gravimetric Method*
BS 4237	Report on Reproducibility of Methods of Chemical Analysis Used in the Iron and Steel Industry
ISO/DP 6137	Aluminium Ores—Methods of Sampling
ISO/DP 6140	Aluminium Ores—Preparation of Samples

3 PRINCIPLE. The specimen on which X-ray fluorescence measurements are made is prepared by incorporating the test portion of the aluminium ore sample, via fusion, into a borate glass disc using a quench pressing procedure. By using such a specimen, particle size effects, which constitute a major problem when a compacted test portion (briquette) is used, are eliminated.

X-ray fluorescence measurements are based on the 'line only' principle. Backgrounds are not measured on each specimen, but are determined on several blank specimens at the line positions.

Calibration is carried out using pure chemicals. Final results are obtained after matrix corrections for interelement effects.

Hygroscopic moisture is determined separately on the laboratory sample in accordance with AS XXXX, but loss on fusion is determined as part of the preparation of the glass specimen that is used for X-ray fluorescence analysis.

4 REAGENTS AND MATERIALS.

4.1 General. During the analysis, only reagents of recognized high purity shall be used.

NOTE: Some reagents of sufficient purity, in particular aluminium oxide, are undesirable because of their hygroscopic nature. Select a reagent with a very low water absorption capacity.

4.2 Silicon dioxide (SiO_2), nominally 99.999 percent SiO_2.

4.3 Aluminium oxide (Al_2O_3), nominally 99.999 percent Al_2O_3.

4.4 Synthetic Calibration Sample.

4.4.1 *Reagents.*

4.4.1.1 *Silicon dioxide* (SiO_2) (4.2).

4.4.1.2 *Aluminium oxide* (Al_2O_3) (4.3).

4.4.1.3 *Iron(III) oxide* (Fe_2O_3), nominally 99.999 percent Fe_2O_3.

4.4.1.4 *Titanium dioxide* (TiO_2), nominally 99.999 percent TiO_2.

NOTE: Phosphorus is a common impurity in TiO_2 and a reagent low in phosphorus must be selected. The selected reagent should be checked as even nominally high purity reagents can be highly contaminated, e.g. a 99.999 percent TiO_2 grade reagent has been found to contain about 0.5 percent P_2O_5.

4.4.1.5 *Ammonium dihydrogen orthophosphate* ($NH_4H_2PO_4$).

4.4.1.6 *Calcium carbonate* ($CaCO_3$).

4.4.2 *Preparation.* The masses of the reagents used in preparing the synthetic calibration sample are given in Table 2. Prior to weighing, however, the iron (III) oxide and titanium dioxide shall be heated at 1000°C for a minimum of 1 h, while the ammonium dihydrogen orthophosphate and calcium carbonate shall be dried at 105°C for a similar time. Since aluminium oxide and silicon dioxide can retain water at 1000°C, they shall be heated at 1200°C for a minimum of 2 h. The cooling of all reagents shall be carried out in a desiccator with self-indicating silica gel desiccant.

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* In course of preparation as DR 82025.