

Australian Standard[®]

Alumina

**Part 7: Determination of trace
elements—Wavelength dispersive
X-ray fluorescence spectrometric
method**

This Australian Standard was prepared by Committee MN/9, Alumina and Materials used in Aluminium Production. It was approved on behalf of the Council of Standards Australia on 30 September 1996 and published on 5 January 1997.

The following interests are represented on Committee MN/9:

Australasian Institute of Mining and Metallurgy

Australian Aluminium Council

Minerals Council of Australia

Royal Australian Chemical Institute

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RECONFIRMATION

OF

AS 2879.7—1997

Alumina

**Part 7: Determination of trace elements—Wavelength dispersive X-ray fluorescence
spectrometric method**

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NOTES

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PREFACE

This Standard was prepared by the Standards Australia Committee on Alumina and Materials used in Aluminium Production, to provide an XRF method for the analysis of alumina.

The objective of this Standard is to provide those responsible for the analysis of smelting-grade alumina with a standardized, validated procedure that will ensure the integrity of the analysis.

The term 'informative' has been used in this Standard to define the application of the appendix to which it applies. An 'informative' appendix is for information and guidance only.

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STANDARDS AUSTRALIA

Australian Standard

Alumina

Part 7: Determination of trace elements—Wavelength dispersive
X-ray fluorescence spectrometric method

1 SCOPE This Standard sets out a wavelength dispersive X-ray fluorescence spectrometric method for the analysis of aluminium oxide for trace amounts of any or all of the following elements: sodium, silicon, iron, calcium, titanium, phosphorus, vanadium, zinc, manganese, gallium, potassium, copper, chromium and nickel (expressed as the oxides Na_2O , SiO_2 , Fe_2O_3 , CaO , TiO_2 , P_2O_5 , V_2O_5 , ZnO , MnO , Ga_2O_3 , K_2O , CuO , Cr_2O_3 and NiO on an as-received basis).

The method is applicable to smelting-grade aluminium oxide. The concentration range covered for each of the components is given in Table 1.

TABLE 1
APPLICABLE CONCENTRATION
RANGE

Component	Concentration range %
Na_2O	0.10 to 1.00
SiO_2	0.005 to 0.05
Fe_2O_3	0.005 to 0.05
CaO	0.01 to 0.10
TiO_2	0.001 to 0.010
P_2O_5	0.001 to 0.050
V_2O_5	0.001 to 0.010
ZnO	0.001 to 0.010
MnO	0.001 to 0.010
Ga_2O_3	0.001 to 0.020
K_2O	0.001 to 0.010
CuO	0.001 to 0.010
Cr_2O_3	0.001 to 0.010
NiO	0.001 to 0.010

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

AS

2508 Safe storage and handling information cards for hazardous materials

2508.8.013 Part 8.013: Hydrofluoric acid (aqueous)

2563 Wavelength dispersive X-ray fluorescence spectrometers—Determination of precision

2706 Numeric values—Rounding and interpretation of limiting values

AS	
2850	Chemical analysis—Interlaboratory test programs—For determining precision of analytical method(s)—Guide to the planning and conduct
ISO	
802	Aluminium oxide primarily used for the production of aluminium—Preparation and storage of test samples
2927	Aluminium oxide primarily used for the production of aluminium—Sampling

3 PRINCIPLE The specimen upon which X-ray fluorescence measurements are made is prepared by incorporating the test portion of the aluminium oxide sample, via fusion, into a borate glass disc using a casting technique.

Calibration is carried out using synthetic standards prepared from pure chemicals using a two-point regression. Matrix corrections may be employed but, because of the low levels at which the analytes are present in the Al_2O_3 matrix, will have negligible effect within the scope of the method. A certified reference material, e.g. NBS 699, is used to verify the calibration.

Intensity measurements are corrected for spectrometer drift.

4 REAGENTS AND MATERIALS

4.1 Flux A mixture of 12 parts lithium tetraborate to 22 parts lithium metaborate. This flux is available commercially.

4.2 Aluminium oxide (Al_2O_3), nominally 99.999% Al_2O_3 . Heat to 1200°C for 2 h and cool in a desiccator.

Every effort shall be made to find a high-purity Al_2O_3 that is not contaminated with analyte elements. To this end, pure aluminas shall be checked before use by preparing as blank discs and measuring net intensities for each analyte element.

NOTE: The methods for measurement of blank discs given in Clause 7.4.5 and Appendix A may be used.

Pure aluminas that give the lowest net intensities for analytes shall be used.

NOTE: If contamination with silica occurs, see Paragraph A3.

4.3 Sodium tetraborate ($\text{Na}_2\text{B}_4\text{O}_7$), nominally 99.99% $\text{Na}_2\text{B}_4\text{O}_7$. Heat to 650°C for 4 h minimum and cool in a desiccator.

4.4 Silicon dioxide (SiO_2), nominally 99.9% SiO_2 . Heat to 1200°C for 2 h and cool in a desiccator.

4.5 Iron(III) oxide (Fe_2O_3), nominally 99.9% Fe_2O_3 . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

4.6 Calcium carbonate (CaCO_3), nominally 99.9% CaCO_3 . Heat to 105°C for 1 h and cool in a desiccator.

4.7 Titanium dioxide (TiO_2), nominally 99.9% TiO_2 . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

4.8 Ammonium dihydrogen orthophosphate ($\text{NH}_4\text{H}_2\text{PO}_4$), nominally 99.9% $\text{NH}_4\text{H}_2\text{PO}_4$. Heat to 105°C for 1 h and cool in a desiccator.

4.9 Vanadium pentoxide (V_2O_5), nominally 99.9% V_2O_5 . Heat to 600°C for 1 h and cool in a desiccator.

4.10 Zinc oxide (ZnO), nominally 99.9% ZnO . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

4.11 Manganese oxide (Mn_3O_4), nominally 99.9% pure. Heat manganese dioxide (MnO_2) for 24 h at 1000°C in a platinum crucible, and cool. Crush the lumpy material to a fine powder, heat for 1 h at 550°C and cool in a desiccator. The resultant material is Mn_3O_4 .

4.12 Gallium oxide (Ga_2O_3), nominally 99.9% Ga_2O_3 . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

4.13 Potassium carbonate (K_2CO_3), nominally 99.9% K_2CO_3 . Heat to 600°C for a minimum of 2 h and cool in a desiccator.

4.14 Copper oxide (CuO), nominally 99.9% CuO . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

4.15 Chromium(III) oxide (Cr_2O_3), nominally 99.9% Cr_2O_3 . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

4.16 Nickel(II) oxide (NiO), nominally 99.9% NiO . Heat to 1000°C for a minimum of 1 h and cool in a desiccator.

5 APPARATUS

5.1 Crucible—a non-wetting, platinum-alloy crucible with lid.

NOTE: The capacity of the crucible should be compatible with the bead requirements of the holders in use; it will be typically 25 mL to 40 mL.

Platinum crucibles shall be free of all elements to be determined.

NOTE: Silica has been found to be a common contaminant and a suggested method for removing silica from platinum ware is given in Paragraph A2.

5.2 Desiccator—provided with an efficient, non-contaminating desiccant. Pelletized molecular sieves have been found satisfactory.

5.3 Electric furnace—fitted with an automatic temperature controller and capable of maintaining a temperature of 1200°C.

5.4 Mould—a non-wetting, circular-shaped, platinum-alloy mould of the type shown in Figure 1. The actual dimensions of the mould will be determined by the sample holders employed in the particular spectrometer used. An example of a 35 mm mould is given in Figure 1.

The surfaces of platinum moulds shall be free of all elements to be determined.

NOTE: Silica has been found to be a common contaminant, and a suggested method for conditioning platinum ware is given in Paragraph A2.

5.5 X-ray fluorescence spectrometer—any conventional wavelength dispersive, vacuum path X-ray fluorescence spectrometer, provided that the performance of the instrument has been checked and found to comply with the precision requirements given in AS 2563.

5.6 Vibratory mill—having grinding components that do not contaminate the intermediate calibration glass (ICG) with analyte elements. Alumina, tungsten carbide and zirconia grinding components have been found to be satisfactory, but care should be taken to ensure that contaminants from the grinding equipment do not affect the analysis.

5.7 Fusion equipment—an electric furnace capable of maintaining a temperature of 1100°C. Alternatively, commercially available automatic fusion machines may be used to prepare fused glass discs.

It is recommended that platinum-tipped tongs be used.

6 SAMPLING AND SAMPLES Samples shall be taken in accordance with ISO 2927 and prepared in accordance with ISO 802.