DRAFT INTERNATIONAL STANDARD ISO/DIS 23201

ISO/TC **226**

Secretariat: **SN**

Voting begins on
2013-04-08

Voting terminates on
2013-07-08

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Aluminium oxide primarily used for production of aluminium — Determination of trace elements — Wavelength dispersive X-ray fluorescence spectrometric method

Oxyde d'aluminium utilisé pour la production d'aluminium — Détermination d'éléments traces — Spectrométrie de fluorescence des rayons X par dispersion en longueur d'onde

ICS 71.100.10

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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Foreword

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ISO 23201 was prepared by Technical Committee ISO/TC 226, *Materials for the production of primary aluminium*.

Introduction

This Standard is based on Australian Standard AS 2879.7–1997 “Determination of trace elements–Wavelength dispersive X-ray fluorescence spectrometric method” 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 standardised, validated procedure that will ensure the integrity of the analysis.

Aluminium oxide primarily used for production of aluminium — 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. These elements are expressed as the oxides Na2O, SiO2, Fe2O3, CaO, TiO2, P2O5, V2O5, ZnO, MnO, Ga2O3, K2O, CuO, Cr2O3 and NiO on an un-dried sample 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
Ga2O3	0,001	to	0,020
K2O	0,001	to	0,010
CuO	0,001	to	0,010
Cr2O3	0,001	to	0,010
NiO	0,001	to	0,010

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

AS 2563, *Wavelength dispersive X — ray fluorescence spectrometers — Determination of precision*

AS 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*

ISO 2927, *Aluminium oxide primarily used for the production of aluminium — Sampling*

3 Principle

A portion of the aluminium oxide test sample is incorporated, via fusion, into a borate glass disc using a casting technique. X ray fluorescence measurements are made on this disc.

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 Al2O3 matrix, will have negligible effect within the scope of the method.

Intensity measurements are corrected for spectrometer drift.

A certified reference material, (see Annex E) is used to verify the calibration.

4 Reagents and materials

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

See Annex F for comments on flux purity.

4.2 Aluminium oxide (Al_2O_3), high purity, nominally 99,999 % Al_2O_3. Prepared by heating to 1200 ± 25 °C for 2 h and cooling in a desiccator.

To ensure the high purity Al_2O_3 is not contaminated with analyte elements, analyse it before use by preparing a blank disc and measuring net intensities for each analyte element.

The methods for measurement of blank discs given in 7.4.5 and Annex A may be used.

If contamination with silica remains, see A.3.

4.3 Sodium tetraborate ($Na_2B_4O_7$), nominally 99,99 % $Na_2B_4O_7$. Prepared by heating to 650 ± 25°C for 4 h minimum and cooling in a desiccator.

4.4 Silicon dioxide (SiO_2), nominally 99,9 % SiO_2. Prepared by heating to 1200 ± 25°C for 2 h and cooling in a desiccator.

4.5 Iron(III) oxide (Fe_2O_3), nominally 99,9 % Fe_2O_3. Prepared by heating to 1000 ± 25°C for a minimum of 1 h and cooling in a desiccator.

4.6 Calcium carbonate ($CaCO_3$), nominally 99,9 % $CaCO_3$. Prepared by heating to 105 ± 5 °C for 1 h and cooling in a desiccator.

4.7 Titanium dioxide (TiO_2), nominally 99,9 % TiO_2. Prepared by heating to 1000 ± 25 °C for a minimum of 1 h and cooling in a desiccator.

4.8 Ammonium dihydrogen orthophosphate ($NH_4H_2PO_4$), nominally 99,9 % $NH_4H_2PO_4$. Prepared by heating to 105 ± 5 °C for 1 h and cooling in a desiccator.

4.9 Vanadium pentoxide (V_2O_5), nominally 99,9 % V2O5. Prepared by heating to 600 ± 25 °C for 1 h and cooling in a desiccator.

4.10 Zinc oxide (ZnO), nominally 99,9 % ZnO. Prepared by heating to 1000 ± 25 °C for a minimum of 1 h and cooling in a desiccator.

4.11 Manganese oxide (Mn_3O_4), nominally 99,9 % pure. Heat manganese dioxide (MnO_2) for 24 h at 1000 □ 25 °C in a platinum crucible and cool in a dessicator. The resultant material is Mn_3O_4. Crush the lumpy material to a fine powder and store in a dessicator.

4.12 Gallium oxide (Ga_2O_3), nominally 99,9 % Ga2O3. Prepared by heating to 1000 ± 25°C for a minimum of 1 h and cooling in a desiccator.

4.13 Potassium carbonate (K_2CO_3), nominally 99,9 % K_2CO_3. Prepared by heating to 600 ± 25 °C for a minimum of 2 h and cooling in a desiccator.

4.14 Copper oxide (CuO), nominally 99,9 % CuO. Prepared by heating to 1000 ± 25 °C for a minimum of 1 h and cooling in a desiccator.

4.15 Chromium(III) oxide (Cr_2O_3), nominally 99,9 % Cr2O3. Prepared by heating to 1000 ± 25 °C for a minimum of 1 h and cooling in a desiccator.

4.16 Nickel(II) oxide (NiO), nominally 99,9 % NiO. Prepared by heating to 1000 ± 25 °C for a minimum of 1 h and cooling in a desiccator.

5 Apparatus

5.1 Platinum crucible, non wetting, platinum or platinum alloy with a lid and having a capacity compatible with the bead requirements of the holders in use. Typical crucibles are 25 mL to 40 mL

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 cleaning platinum ware and removing silica is given in A2.

5.2 Desiccator, provided with an efficient, non-contaminating desiccant.

NOTE 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 ± 25°C.

5.4 Platinum mould, non wetting, platinum or platinum-alloy, circular-shaped of the type shown in Figure 1 and with dimensions compatible with sample holders employed in the particular spectrometer used. An example of a 35 mm mould is given in Figure 1.

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