

Australian Standard[®]

Refractories and refractory materials— Chemical analysis

Part 3: High alumina materials



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- Australasian Ceramic Society
 - Australasian Institute of Mining and Metallurgy
 - Bureau of Steel Manufacturers of Australia
 - CSIRO Manufacturing and Infrastructure Technology
 - Cement Industry Federation
 - Institute of Refractories Engineers
 - Refractories Manufacturers Association of Australia
 - The University of New South Wales
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Refractories and refractory materials— Chemical analysis

Part 3: High alumina materials

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PREFACE

This Standard was prepared by the Standards Australia Committee MN-007, Refractories and Refractory Materials, to supersede AS 2503.3—1985. It describes the chemical analysis of high alumina refractory materials.

Other parts of the Standard describing the chemical analysis of other refractory materials in the series as follows:

Part 1: Silica refractories

Part 2: Aluminosilicate refractories

Part 4: Dolomites and magnesites

Part 5: Chrome-bearing materials

In preparing this Standard, the Committee drew extensively upon the corresponding work of ISO/TC 33, Refractories, and the methods specified in BS 1902, *Methods of testing refractory materials*. The Committee also took into consideration the present practices of Australian industry and testing laboratories, details being established or verified, where necessary, by reference to the staff of refractories testing laboratories.

The objective of this revision is to bring the Standard into alignment with current style.

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

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STANDARDS AUSTRALIA

Australian Standard

Refractories and refractory materials—Chemical analysis

Part 3: High alumina materials

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE

This Standard sets out methods for the sampling, preparation of sample and analysis of high alumina refractories and high alumina refractory materials. Procedures are described for determining—

- (a) the loss on ignition; and
- (b) the chemical composition, viz the amount of silicon, phosphorus, aluminium, iron, titanium, manganese, calcium, magnesium, sodium, potassium and lithium present, expressed as the oxides of these elements.

Table 1.1 illustrates the typical range of composition of high alumina refractories and the associated form of expression. This Standard applies to materials whose compositions are within that range.

TABLE 1.1
TYPICAL COMPOSITION OF HIGH
ALUMINA REFRACTORY MATERIALS

Constituent	Range, percent
Aluminium oxide as Al_2O_3	$\geq 46^*$
Silicon dioxide as SiO_2	≤ 55
Calcium oxide as CaO	≤ 5
Iron (I II) oxide as Fe_2O_3	≤ 5
Potassium oxide as K_2O	≤ 5
Titanium dioxide as TiO_2	≤ 3
Magnesium oxide as MgO	≤ 3
Phosphorus pentoxide as P_2O_5	≤ 1.5
Manganese oxide as MnO	≤ 1.0
Sodium oxide as Na_2O	≤ 0.5
Lithium oxide as Li_2O	≤ 0.5

* To determine aluminium oxide contents of less than 46 percent (*m/m*) see AS 2503, Parts 1 and 2.

1.2 REFERENCED DOCUMENTS

The following Standards are referred to in this Standard:

AS

1152	Specification for test sieves
2497	Procedures for acceptance testing of refractory products
2497.1	Part 1: Batch procedure
2503	Refractories and refractory materials—Chemical analysis
2503.1	Part 1: Silica refractories
2503.2	Part 2: Aluminosilicate refractories
2780	Refractories and refractory materials—Glossary of terms
3753	Recommended practice for the chemical analysis of materials by ultraviolet/visible spectrophotometry

AS/NZS

2243	Safety in laboratories (series)
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1.3 DEFINITION

For the purpose of this Standard, the following definitions in AS 2780.

1.4 REPEATABILITY

Duplicate determinations shall be carried out in all cases. The analyses shall be repeated if the difference between duplicate determinations exceeds the following limits:

- (a) For aluminium oxide or other constituent exceeding 30 percent (*m/m*) 0.3 percent max.
- (b) For silicon dioxide or other constituent between 10 percent and 30 percent (*m/m*) 0.2 percent max.
- (c) For other constituents of between 5 percent and 10 percent (*m/m*) 0.1 percent max.
- (d) For other constituents amounting to less than 5 percent (*m/m*) 0.05 percent max.

1.5 PRINCIPLE

1.5.1 Loss on ignition (see Section 3)

A sample is heated to constant mass at 1025°C and the loss on ignition is determined by mass difference.

1.5.2 Determination of silicon, phosphorus, aluminium, iron (III), titanium, manganese, calcium and magnesium oxides (see Section 4)

The sample is fused with a potassium carbonate/sodium carbonate mixture in the presence of boric acid and the melt is dissolved in a sorbitol/hydrochloric acid/sulphuric acid mixture. The mixture is evaporated, digested with further hydrochloric acid and the silicon dioxide separated by filtration.

The residue is ignited, in a previously ignited and weighed crucible, to constant mass and after treatment with hydrofluoric and sulphuric acids, is again ignited to constant mass. The product is fused with potassium pyrosulphate and the solution of the melt added to the filtrate obtained earlier. The resulting solution is diluted to a Standard volume.

Residual silicon, phosphorus, iron (III), titanium and manganese oxides are determined spectrophotometrically on aliquots of the stock solution using respectively, ammonium molybdate, ammonium molybdate plus ammonium meta-vanadate, 1, 10-phenanthroline, hydrogen peroxide and ammonium persulphate.

Aluminium oxide is determined volumetrically with EDTA on a further aliquot after removal of interfering elements by precipitation with cupferron and extraction with chloroform.

Calcium and magnesium oxides are determined by titrating aliquots of the stock solution with EDTA, any interfering elements being complexed with triethanolamine.

1.5.3 Determination of sodium, potassium and lithium oxides (see Section 5)

Another sample is reacted with sulphuric and hydrofluoric acids and then evaporated to dryness. The residue is dissolved in dilute sulphuric acid and the alkali metal oxides are determined on an aliquot using a flame photometer.

1.6 SAFETY

For information on laboratory safety, reference should be made to the relevant parts of AS/NZS 2243.

Reagents used may be hazardous or toxic and reference should be made to the appropriate Material Safety Data Sheets.

SECTION 2 SAMPLING AND SAMPLE PREPARATION

2.1 SCOPE OF SECTION

This Section sets out methods of sampling and the subsequent preparation of the sample for analysis.

2.2 GROSS SAMPLING

Methods for obtaining a suitable sample of a shaped refractory for the analytical procedures outlined below are described in AS 2497.1.

2.3 PREPARATION OF SAMPLE

2.3.1 Crushing

2.3.1.1 Bricks

The procedure shall be as follows:

- (a) Break up half of each brick, including any skin (see Note) with the aid of a jaw crusher so that the crushed sample will pass a 4.00 mm test sieve complying with AS 1152.

NOTE: Discard any fuel ash present as it will give misleading results.

- (b) Reduce the sample mass to about 400 g by mixing, coning and quartering, or by the use of a mechanical sample divider. Crush in an iron mortar to pass a 2.00 mm test sieve complying with AS 1152.

2.3.1.2 Ground materials

The procedure shall be as follows:

- (a) Mix, cone and quarter, or use a mechanical sample divider, to reduce the gross sample to about 400 g.
- (b) Crush, if necessary, as with brick samples, to pass a 2.00 mm test sieve.

2.3.2 Samples for analysis

2.3.2.1 Preliminary treatment

The procedure shall be as follows:

- (a) Place a piece of clean white paper over the end of a pot magnet and slowly pass it backward and forward over, but close to, the crushed and/or ground sample. Remove and discard any attached iron particles from the magnet, remix the sample and repeat the process. Continue until no more iron is removed from the sample.
- (b) Mix, cone and quarter, or use a mechanical sample divider, to obtain two portions, A and B, of about 50 g each.

NOTE: In cases where an iron determination is required, the procedure for portion B (2.3.2.3) should be followed since use of a hardened steel percussion mortar, as in the procedure for portion A (2.3.2.2), may introduce iron contamination which cannot be removed by the use of a magnet.

2.3.2.2 *Portion A—to be used for the main analysis*

The procedure shall be as follows:

- (a) Crush sample in a hardened steel percussion mortar to pass a 500 mm test sieve complying with AS 1152.
- (b) Mix, cone and quarter, or use a mechanical sample divider, to obtain a 1 g to 15 g sample.
- (c) Crush this sample in a hardened steel percussion mortar to pass a 125 mm test sieve complying with AS 1152 and remove any particles of extraneous iron with a magnet as before (Clause 2.3.2.1(a)).

2.3.2.3 *Portion B—to be used where iron determination is required*

The procedure shall be as follows:

- (a) Crush in a pure alumina mortar to pass a 500 mm test sieve complying with AS 1152.
- (b) Mix, cone and quarter, or use a mechanical sample divider to obtain a 10 g to 15 g sample.
- (c) Grind this sample in a pure alumina mortar to pass a 125 mm test sieve complying with AS 1152.

NOTE: A non-metallic sieve (e.g. nylon bolting cloth) is preferable at this stage.