

Australian Standard[®]

Refractories and refractory materials— Chemical analysis

Part 1: Silica refractories



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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 1: Silica refractories

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PREFACE

This Standard was prepared by the Standards Australia Committee MN-007, Refractories and Refractory Materials, as a revision of AS 2503.1—1996, *Refractories and refractory materials—Chemical analysis*, Part 1: *Silica refractories*. It deals with the chemical analysis of silica refractory materials.

Other parts of this Standard dealing with the chemical analysis of the other refractory materials, are as follows:

Part 2: Aluminosilicate refractories

Part 3: High alumina materials

Part 4: Dolomites and magnesites

Part 5: Chrome-bearing materials

The Committee acknowledges the documented work of ISO/TC 33, Refractories, and the reference Standard BS 1902, *Methods of testing refractory materials*, which were used as source materials in the development of this Standard.

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 1: Silica refractories

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE

This Standard sets out methods for the sampling, preparation of samples and analysis of silica refractories and 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 composition of silica refractories and the associated form of expression.

NOTE: A schematic presentation of the analytical procedures in this Standard is given in Appendix A.

TABLE 1.1
COMPOSITION OF SILICA REFRACTORY MATERIALS

Constituent	Expressed as	Range, percent
Silicon dioxide	SiO ₂	≥93
Aluminium oxide	Al ₂ O ₃	0 to 5
Calcium oxide	CaO	0 to 4
Iron(III) oxide	Fe ₂ O ₃	0 to 3
Potassium oxide	K ₂ O	0 to 0.25
Titanium(IV) dioxide	TiO ₂	0 to 2
Magnesium oxide	MgO	0 to 0.5
Phosphorus pentoxide	P ₂ O ₅	0 to 0.25
Manganese(II) oxide	MnO	0 to 0.25
Sodium oxide	Na ₂ O	0 to 0.25
Lithium oxide	Li ₂ O	0 to 0.25

1.2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS
1152 Specification for test sieves

AS	
2497	Procedures for acceptance testing of refractory products
2497.1	Part 1: Batch procedure
2780	Refractories and refractory materials—Glossary of terms
3753	Recommended practice for the chemical analysis by ultraviolet/visible spectrophotometry
AS/NZS	
2243	Safety in laboratories (series)

1.3 DEFINITIONS

For the purpose of this Standard, the definitions given in AS 2780 and that below apply.

1.3.1 Silica refractory

A refractory or refractory material in which the silicon dioxide content is not less than 93% (*m/m*).

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 silicon dioxide 0.3% max.
- (b) For aluminium oxide or other constituent amounting to between
1% and 5% (*m/m*) 0.2% max.
- (c) For any constituent amounting to less than 1% (*m/m*) 0.05% max.

1.5 PRINCIPLE

1.5.1 Loss on ignition

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

1.5.2 Determination of silicon dioxide and phosphorus pentoxide (see Section 4)

The sample is fused with a potassium carbonate/sodium carbonate mixture and the melt is dissolved in hydrochloric acid with the addition of a small amount of sulfuric acid. 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 sulfuric acids, is again ignited to constant mass. The product is fused with potassium pyrosulfate and the solution of the melt added to the filtrate obtained earlier. After dilution to a standard volume, the silicon dioxide and phosphorus remaining in the filtrate are determined spectrophotometrically, the silicon dioxide by a method based on the formation of a molybdenum blue colour, and the phosphorus with ammonium metavanadate and ammonium molybdate.

NOTE: See flow chart in Paragraph A1, Appendix A.

1.5.3 Determination of aluminium, iron, titanium and manganese oxides (see Section 5)

A sample is decomposed with hydrofluoric and sulfuric acids and evaporated to dryness. After further addition of sulfuric acid, the mixture is ignited and the silicon dioxide removed by evaporation to dryness. The residue is fused with a sodium carbonate and boric acid mixture. It is then dissolved in hydrochloric acid. Interfering elements are removed from an aliquot by precipitation with cupferron followed by extraction with chloroform, the aluminium being determined gravimetrically with 8-hydroxyquinoline or volumetrically with EDTA. Iron, titanium and manganese oxides are determined spectrophotometrically on further aliquots of the solution using respectively 1,10-phenanthroline, hydrogen peroxide and ammonium persulfate.

NOTE: See flow chart in Paragraph A2, Appendix A.

1.5.4 Determination of calcium, magnesium, sodium, potassium and lithium oxides (see Section 6)

A further sample is reacted with nitric, perchloric and hydrofluoric acids and then evaporated to dryness. The residue is dissolved in hydrochloric acid. Calcium and magnesium oxides are determined by titrating aliquots with EDTA, and the alkali metal oxides are then determined on a further aliquot using a flame photometer.

NOTE: See flow chart in Figure A3, Appendix A.

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.

1.7 TEST REPORT

The test report shall include the following particulars:

- (a) Identification of the sample by means of a description or code number.
- (b) The results expressed in accordance with the method given in the appropriate calculation clause and with due regard to the specified reliability as given by Clause 1.4.
- (c) Any unusual features noted during the determination which would contribute to the correct interpretation of the results.
- (d) A description of any operation not included in this Australian Standard, or regarded as optional, which might affect the results.
- (e) Reference to this Australian Standard, i.e. AS 2503.1.

SECTION 2 SAMPLING AND SAMPLE PREPARATION

2.1 SCOPE OF SECTION

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

2.2 GROSS SAMPLING

Methods for obtaining a suitable sample 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

Bricks shall be prepared 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 and 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

Ground materials shall be prepared as follows:

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

2.3.2 Preliminary treatment

The ground samples shall first be treated 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, and close to, the sample. Remove 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 and divide the sample 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 should be followed since use of a hardened steel percussion mortar, as in the procedure for portion A, may introduce iron contamination which cannot be removed by the use of a magnet.

2.3.3 Preparation of sample for analysis

2.3.3.1 Portion A—to be used for the main analysis

Portion A shall be prepared as follows:

- (a) Crush in a hardened steel percussion mortar to pass a 500 μ m test sieve complying with AS 1152.
- (b) Mix and divide the sample to obtain a 10 g to 15 g sample.

- (c) Crush this sample in a hardened steel percussion mortar to pass a 125 µm test sieve complying with AS 1152 and remove any particles of extraneous iron with a magnet as before (see Clause 2.3.2(a)).

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

Portion B shall be prepared as follows:

- (a) Crush in a pure alumina mortar to pass a 500 µm test sieve complying with AS 1152.
- (b) Mix and divide the sample to obtain a 10 g to 15 g sample.
- (c) Grind this sample in a pure alumina mortar to pass a 125 µm test sieve complying with AS 1152.

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

SECTION 3 DETERMINATION OF LOSS ON IGNITION

3.1 SCOPE OF SECTION

This Section sets out the method for determining the loss on ignition of the test sample prepared in accordance with Section 2.

3.2 APPARATUS

3.2.1 Balance

Capable of weighing up to 30 g to the nearest 0.001 g.

3.2.2 Muffle furnace

Capable of maintaining any set temperature in the range 1000°C to 1200°C to the nearest 25°C.

3.2.3 Drying oven

Capable of maintaining any set temperature in the range 110°C to 150°C to the nearest 2°C.

3.3 PROCEDURE

The procedure shall be as follows:

- (a) Dry between 10 g and 15 g of the appropriate analysis sample (2.3.3) to constant mass at 110°C.
- (b) Weigh, to the nearest 0.001 g, approximately 1 g of this sample into a tared platinum crucible (m_1).
- (c) Place the crucible in the furnace at approximately 200°C and slowly raise the temperature to 1025°C.
- (d) Ignite to constant mass at this temperature. Record the final mass (m_2) of crucible and sample to the nearest 0.001 g.

NOTE: Ignition for 1 h is usually sufficient.

- (e) Calculate the percentage loss on ignition from the following equation:

$$\% \text{ Loss on ignition } (m/m) = \frac{100(m_1 - m_2)}{(m_1 - M)} \quad \dots 3.1$$

where

m_1 = mass of crucible plus sample before ignition, (3.3(b)), in grams

m_2 = mass of crucible plus sample after ignition, (3.3(d)), in grams

M = mass of crucible, in grams

SECTION 4 DETERMINATION OF SILICON DIOXIDE AND PHOSPHORUS PENTOXIDE

4.1 SCOPE OF SECTION

This Section sets out methods for determining the silicon dioxide and phosphorus pentoxide contents of the test sample prepared in accordance with Section 2.

4.2 REAGENTS

4.2.1 General

All reagents shall be of analytical reagent grade and distilled or demineralized water shall be used throughout the analysis.

4.2.2 Solid reagents

4.2.2.1 *Potassium carbonate/sodium carbonate, anhydrous, mixed in equimolecular proportions*

4.2.2.2 *Potassium pyrosulfate*

4.2.2.3 *Sodium carbonate, anhydrous*

4.2.3 Solutions

4.2.3.1 *Ammonia solution (ρ_{20} 0.88 g/mL)*

4.2.3.2 *Ammonia solution (500 mL/L)*

Dilute 500 mL of ammonia solution (4.2.3.1) to 1 L.

4.2.3.3 *Ammonium metavanadate solution (2.5 g/L)*

4.2.3.4 *Ammonium molybdate solution (100 g/L)*

Dissolve 50 g of ammonium molybdate, $(\text{NH}_4)_6 \text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, in water, filtering if necessary and dilute to 500 mL. Store in a polyethylene bottle. Discard after 4 weeks, or earlier if an appreciable deposit is observed.

4.2.3.5 *Hydrochloric acid (ρ_{20} 1.18 g/mL)*

4.2.3.6 *Hydrochloric acid (500 mL/L)*

Dilute 500 mL of hydrochloric acid (4.2.3.5) to 1 L.

4.2.3.7 *Hydrochloric acid (200 mL/L)*

Dilute 200 mL of hydrochloric acid (4.2.3.5) to 1 L.

4.2.3.8 *Hydrochloric acid (50 mL/L)*

Dilute 50 mL of hydrochloric acid (4.2.3.5) to 1 L.

4.2.3.9 *Hydrofluoric acid (ρ_{20} 1.13 g/mL)*

4.2.3.10 *Nitric acid (ρ_{20} 1.4 g/mL)*

4.2.3.11 *Nitric acid (330 mL/L)*

Dilute 330 mL of nitric acid (4.2.3.10) to 1 L.

4.2.3.12 *Sulfuric acid (500 mL/L)*

Cautiously prepare 1 L from concentrated sulfuric acid (ρ_{20} 1.84 g/mL).