

Australian Standard[®]

**Refractories and refractory
materials—Chemical analysis**

Part 5: Chrome-bearing materials

This Australian Standard was prepared by Committee MN/7, Refractories and Refractory Materials. It was approved on behalf of the Council of Standards Australia on 22 December 1988 and published on 18 August 1989.

The following interests are represented on Committee MN/7:

Bureau of Steel Manufacturers of Australia
CSIRO, Division of Materials Science and Technology
Electricity Supply Association of Australia
Refractories Manufacturers Association of Australia
University of New South Wales

Review of Australian Standards. *To keep abreast of progress in industry, Australian Standards are subject to periodic review and are kept up to date by the issue of amendments or new editions as necessary. It is important therefore that Standards users ensure that they are in possession of the latest edition, and any amendments thereto.*

Full details of all Australian Standards and related publications will be found in the Standards Australia Catalogue of Publications; this information is supplemented each month by the magazine 'The Australian Standard', which subscribing members receive, and which gives details of new publications, new editions and amendments, and of withdrawn Standards.

Suggestions for improvements to Australian Standards, addressed to the head office of Standards Australia, are welcomed. Notification of any inaccuracy or ambiguity found in an Australian Standard should be made without delay in order that the matter may be investigated and appropriate action taken.

This Standard was issued in draft form for comment as DR 87190.

Australian Standard[®]

**Refractories and refractory
materials—Chemical analysis**

Part 5: Chrome-bearing materials

First published as part of AS R28—1965.
Revised and redesignated AS 2503.5—1989.

PREFACE

This Standard was prepared by the Standards Australia Committee on Refractories and Refractory Materials, as a further part of this series of Standards to supersede AS R28—1965, *Methods for the sampling and chemical analysis of refractories and refractory materials*. Other parts of this series, which deal with the essentially classical methods of analysis of refractory materials, are as follows:

AS 2503 *Refractories and refractory materials—Chemical analysis*

Part 1: *Silica refractory materials*

Part 2: *Aluminosilicate refractory materials*

Part 3: *High alumina materials*

Part 4: *Dolomites and magnesites*

A further part dealing with the determination of carbon in carbon-bearing refractory materials is in course of preparation.

This Standard incorporates a revision of format to bring it into line with the requirements of SAA MP34, *Guide to the layout and preparation of standard methods of chemical analysis* and current drafting practices of Standards Australia.

In preparing this Standard, the corresponding work of ISO/TC 33, Refractories, and BS 1902, *Methods of testing refractory materials* were extensively drawn upon. Consideration was also given to the present practices of Australian industry and testing laboratories, details being established or verified, where necessary, by reference to the staff of laboratories who routinely conduct refractory analysis.

This Standard utilizes titrimetric, UV-visible spectrometric and flame photometric methods of analysis and includes flowsheets depicting the scheme of analysis (see Appendix C). A parallel series of Standards dealing with analysis by atomic absorption methods is under consideration.

© Copyright — STANDARDS AUSTRALIA

Users of Standards are reminded that copyright subsists in all Standards Australia publications and software. Except where the Copyright Act allows and except where provided for below no publications or software produced by Standards Australia may be reproduced, stored in a retrieval system in any form or transmitted by any means without prior permission in writing from Standards Australia. Permission may be conditional on an appropriate royalty payment. Requests for permission and information on commercial software royalties should be directed to the head office of Standards Australia.

Standards Australia will permit up to 10 percent of the technical content pages of a Standard to be copied for use exclusively in-house by purchasers of the Standard without payment of a royalty or advice to Standards Australia.

Standards Australia will also permit the inclusion of its copyright material in computer software programs for no royalty payment provided such programs are used exclusively in-house by the creators of the programs.

Care should be taken to ensure that material used is from the current edition of the Standard and that it is updated whenever the Standard is amended or revised. The number and date of the Standard should therefore be clearly identified.

The use of material in print form or in computer software programs to be used commercially, with or without payment, or in commercial contracts is subject to the payment of a royalty. This policy may be varied by Standards Australia at any time.

CONTENTS

	<i>Page</i>
SECTION 1. SCOPE AND GENERAL	
1.1 SCOPE	4
1.2 REFERENCED DOCUMENTS	4
1.3 PRINCIPLE	4
1.4 REPEATABILITY	5
1.5 DEFINITIONS	5
SECTION 2. SAMPLING AND SAMPLE PREPARATION	
2.1 SCOPE OF SECTION	6
2.2 GROSS SAMPLING	6
2.3 PREPARATION OF SAMPLE	6
SECTION 3. DETERMINATION OF LOSS ON IGNITION	
3.1 SCOPE OF SECTION	7
3.2 APPARATUS	7
3.3 PROCEDURE	7
SECTION 4. DETERMINATION OF SILICON	
4.1 SCOPE OF SECTION	8
4.2 REAGENTS	8
4.3 APPARATUS	8
4.4 PROCEDURE	8
SECTION 5. DETERMINATION OF CHROMIUM	
5.1 SCOPE OF SECTION	10
5.2 REAGENTS	10
5.3 APPARATUS	10
5.4 PROCEDURE	10
SECTION 6. DETERMINATION OF TITANIUM, IRON, ALUMINUM, MANGANESE, CALCIUM AND MAGNESIUM	
6.1 SCOPE	11
6.2 REAGENTS	11
6.3 APPARATUS	13
6.4 PROCEDURE	13
SECTION 7. DETERMINATION OF SODIUM, POTASSIUM AND LITHIUM OXIDES	
7.1 SCOPE OF SECTION	17
7.2 REAGENTS	17
7.3 APPARATUS	17
7.4 PROCEDURE	17
SECTION 8 TEST REPORT	18
APPENDICES	
A STANDARDIZATION OF CDTA AND EGTA SOLUTIONS	19
B STANDARDIZATION OF AMMONIUM IRON(II) SULFATE SOLUTION	21
C SCHEME OF ANALYSIS FOR CHROME-BEARING MATERIALS	22

STANDARDS AUSTRALIA

Australian Standard

Refractories and refractory materials—Chemical analysis

Part 5: Chrome-bearing materials

SECTION 1. SCOPE AND GENERAL

1.1 SCOPE. This Standard sets out methods for the sampling, sample preparation, and analysis of chrome-bearing refractories and refractory materials. Procedures are described for determining—

- the loss on ignition*;
- the chemical composition*, viz the amount of silicon, chromium, titanium, iron, aluminium, manganese, calcium, magnesium, sodium, potassium and lithium present in chrome-bearing materials, expressed as the oxides of these elements.

NOTE: Appendix C provides an overview of these test procedures in flowchart form.

This Standard applies to chrome-bearing materials represented by those defined in Clause 1.5, whose composition is typified by Table 1.1. It may be applicable to materials which fall outside these ranges, but if used for such cases, it is necessary to exercise caution when interpreting the results as it cannot be assumed the limits cited in Clause 1.4 apply.

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

AS	
2162	Code of practice for the use of volumetric glassware
2243	Safety in laboratories
2243.2	Part 2: Chemical
2497	Sampling procedures for acceptance testing of shaped refractory products
2508	Safe storage and handling information cards for hazardous materials
2508.8.002	Hydrochloric acid (muriatic acid, spirits of salts)
2508.8.004	Acetic acid (containing 80% acid or more, by mass, in water)

2508.8.006	Sodium hydroxide (caustic soda) (solid and solution)
2508.8.007	Nitric acid
2508.8.010	Sulfuric acid
2508.8.013	Hydrofluoric acid (aqueous)
2780	Refractories and refractory materials—Glossary of terms
CK19	Code of recommended practice for the chemical analysis of materials by ultra-violet visible spectrophotometry

1.3 PRINCIPLE. The principles of the methods of the various analyses set out in this Standard are as follows:

- Loss on ignition.* A sample is heated to constant mass at 1000°C.
- Determination of silicon.* A sample is fused with potassium carbonate/sodium carbonate and boric acid, and the product dissolved in sulfuric acid. After dilution and the addition of ammonium molybdate, the pH is adjusted and silicomolybdate precipitated by quinoline. The silicon content is determined gravimetrically by drying the filtered precipitate to constant mass.
- Determination of chromium.* After fusion of a sample with potassium carbonate/sodium carbonate and boric acid, and dissolution in sulfuric acid, any chromate present is oxidized with potassium permanganate. Residual permanganate is removed and excess iron(II) ammonium sulfate added, followed by phosphoric acid. The solution is then back-titrated with standard dichromate solution.

* The original paper on which these methods are based was written by BENNETT, H. and REED, R.A., *The Analyst*, Vol. 97 (1972), pp. 794-819.

TABLE 1.1
APPLICABILITY OF TEST METHODS TO
CHROME-BEARING REFRACTORY MATERIALS

Elemental constituent	Expressed as	Limit for accurate determination percent (m/m)	Typical composition percent (m/m)
Magnesium	MgO	0.1	≤ 85
Chromium	Cr ₂ O ₃	0.05	≥ 3, ≤ 55
Aluminium	Al ₂ O ₃	0.1	≤ 35
Iron	Fe ₂ O ₃	0.3	≤ 20
Silicon	SiO ₂	0.02	≤ 25
Calcium	CaO	0.05	≤ 20
Manganese	MnO	0.05	≤ 5
Titanium	TiO ₂	0.2	≤ 2.5
Sodium	Na ₂ O	0.05	≤ 0.5
Potassium	K ₂ O	0.05	≤ 0.5
Lithium	Li ₂ O	0.05	≤ 0.5

NOTE: The values given in Column 3 represent the lowest concentration of constituent for which the test methods described in this Standard are considered to produce accurate results.