

Australian Standard™

Coal and coke—Analysis and testing

**Part 14.3: Higher rank coal ash and coke
ash—Major and minor elements—
Wavelength dispersive X-ray
fluorescence spectrometric method**

This Australian Standard was prepared by Committee MN/1, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 5 October 1999 and published on 5 November 1999.

The following interests are represented on Committee MN/1:

Australasian Institute of Mining and Metallurgy
Australian Coal Association
Australian Coal Preparation Society
Australian Institute of Energy
Bureau of Steel Manufacturers of Australia
Coalfield Geology Council of N.S.W.
CSIRO, Division of Energy Technology
Department of Mines & Energy, Qld
Electricity Supply Association of Australia
Institute of Engineers, Australia
Minerals Council of Australia
University of Newcastle
University of New South Wales
University of Queensland

Keeping Standards up-to-date

Standards are living documents which reflect progress in science, technology and systems. To maintain their currency, all Standards are periodically reviewed, and new editions are published. Between editions, amendments may be issued. Standards may also be withdrawn. It is important that readers assure themselves they are using a current Standard, which should include any amendments which may have been published since the Standard was purchased.

Detailed information about Standards can be found by visiting the Standards Australia web site at www.standards.com.au and looking up the relevant Standard in the on-line catalogue.

Alternatively, the printed Catalogue provides information current at 1 January each year, and the monthly magazine, *The Australian Standard*, has a full listing of revisions and amendments published each month.

We also welcome suggestions for the improvement in our Standards, and especially encourage readers to notify us immediately of any apparent inaccuracies or ambiguities. Contact us via email at mail@standards.com.au, or write to the Chief Executive, Standards Australia International Ltd. PO Box 1055, Strathfield., NSW 2135.

This Standard was issued in draft form for comment as DR 99018.

STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 1038.14.3—1999

Coal and coke—Analysis and testing

Part 14.3: Higher rank coal ash and coke ash—Major and minor elements—
Wavelength dispersive X-ray fluorescence spectrometric method

RECONFIRMATION NOTICE

Technical Committee MN-001 has reviewed the content of this publication and in accordance with Standards Australia procedures for reconfirmation, it has been determined that the publication is still valid and does not require change.

Certain documents referenced in the publication may have been amended since the original date of publication. Users are advised to ensure that they are using the latest versions of such documents as appropriate, unless advised otherwise in this Reconfirmation Notice.

Approved for reconfirmation in accordance with Standards Australia procedures for reconfirmation on 15 July 2013.

The following are represented on Technical Committee MN-001:

Australasian Institute of Mining and Metallurgy
Australian Coal Association
Australian Coal Industry Reference Samples
Australian Coal Preparation Society
Bureau of Steel Manufacturers of Australia
Coalfield Geology Council of NSW
CSIRO Energy Technology
National Association of Testing Authorities Australia
National Generators Forum
The University of New South Wales
The University of Queensland
University of Newcastle

NOTES

Australian Standard™

Coal and coke—Analysis and testing

Part 14.3: Higher rank coal ash and coke ash—Major and minor elements— Wavelength dispersive X-ray fluorescence spectrometric method

First published as AS 1038.14.3—1999

COPYRIGHT

© Standards Australia International

All rights are reserved. No part of this work may be reproduced or copied in any form or by any means, electronic or mechanical, including photocopying, without the written permission of the publisher.

Published by Standards Australia International Ltd
PO Box 1055, Strathfield, NSW 2135, Australia
ISBN 0 7337 3008 6

PREFACE

This Standard was prepared by the Standards Australia Committee MN/1, Coal and Coke, as an additional method in the series of standard methods for the analysis and testing of coal and coke.

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the appendix to which they apply. A ‘normative’ appendix is an integral part of a Standard, whereas an ‘informative’ appendix is only for information and guidance.

CONTENTS

	<i>Page</i>
1 SCOPE	3
2 REFERENCED DOCUMENTS	4
3 DEFINITIONS	4
4 PRINCIPLE	4
5 SAFETY	4
6 REAGENTS	4
7 APPARATUS	5
8 SAMPLE	5
9 PROCEDURE	6
10 CALCULATION	9
11 REPORTING OF RESULTS	9
12 PRECISION	10
13 TEST REPORT	10

APPENDICES

A ACCEPTANCE OF ANALYTICAL RESULTS FOR LABORATORY METHOD	11
B REAGENTS SUITABLE FOR THE PREPARATION OF SYNTHETIC CALIBRATION STANDARDS	13
C STANDARD DEVIATION OF GLASS DISC PREPARATION	14
D PRECISION DATA FOR ELEMENTAL ANALYSIS OF COAL ASH AND COKE ASH	16

STANDARDS AUSTRALIA

Australian Standard

Coal and coke—Analysis and testing

Part 14.3: Higher rank coal ash and coke ash—Major and minor elements—Wavelength dispersive X-ray fluorescence spectrometric method

1 SCOPE This Standard sets out a wavelength dispersive X-ray fluorescence procedure for the determination of silicon, aluminium, iron, calcium, magnesium, sodium, potassium, titanium, manganese, phosphorus and sulfur.

The method is applicable to coal ashes, coke ashes and boiler ashes having components of concentration ranges specified in Table 1.

TABLE 1
RANGES OF APPLICATION OF
THE METHOD

Oxide	Concentration range, %		
SiO ₂	5	to	100
Al ₂ O ₃	5	to	80
Fe ₂ O ₃	0.1	to	25
CaO	0.05	to	25
MgO	0.05	to	25
Na ₂ O	0.05	to	5
K ₂ O	0.05	to	5
TiO ₂	0.05	to	5
Mn ₃ O ₄	0.005	to	5
P ₂ O ₅	0.01	to	5
SO ₃	0.05	to	10

NOTES:

- 1 Additional analytes may be included in the method provided appropriate validation using Certified Reference Materials (CRM) is carried out.
- 2 The precision statistics reported in Appendix D are only robust for the concentration ranges included in the test program. Outside of these ranges precision should be determined using suitable Certified Reference Materials.
- 3 The method has been tested for the following additional analytes; BaO (0.01 to 1%); SrO (0.01 to 1%) and ZnO (0.005 to 1%).

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

AS

- 1038 Coal and coke—Analysis and testing
- 1038.3 Part 3: Proximate analysis of higher rank coal
- 1038.4 Part 4: Coke—Proximate analysis
- 1038.16 Part 16: Assessment and reporting of results
- 2243 Safety in laboratories (series)
- 2418 Coal and coke—Glossary of terms
- 2508 Safe storage and handling information card (series)
- 2563 Wavelength dispersive X-ray fluorescence spectrometers—Determination of precision
- 2706 Numerical values—Rounding and interpretation of limiting values
- 4264 Coal and coke—Sampling
- 4264.1 Part 1: Higher rank coal—Sampling procedures
- 4264.2 Part 2: Coke—Sampling procedures

ISO

- 5725 Accuracy (trueness and precision) of measurement methods and results (series)

3 DEFINITIONS For the purpose of this Standard, the definitions given in AS 2418 apply.

4 PRINCIPLE The specimen on which X-ray fluorescence measurements are made is prepared by incorporating the test portion of the sample, via fusion, into a borate glass disc using a casting or press quenching procedure. By using such a specimen, particle size effects are eliminated.

Calibration is carried out using pure chemicals or analysed standards or both, and making matrix corrections for inter-element effects.

5 SAFETY For information on laboratory safety, reference should be made to the relevant parts of AS 2243 and AS 2508.

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

6 REAGENTS

6.1 General Unless otherwise specified, all reagents shall be of analytical reagent grade and only distilled water or water of equivalent purity shall be used.

6.2 Flux A number of different borate fluxes have been found suitable for the preparation of glass discs from coal/coke ash samples. These fluxes are based on mixtures of one or more of lithium tetraborate, lithium metaborate and lanthanum oxide. Flux may be purchased commercially or prepared by fusion of a mixture of individual reagents.

NOTE: The levels of contamination in the flux should be checked. Because levels of contamination may vary from batch to batch, the same batch of flux should be used for all specimens (synthetic standards, external standards and unknowns). When using a fresh batch of flux, CRM specimens should be prepared to determine whether adjustments to the calibration are required.

7 APPARATUS

7.1 General All apparatus shall be constructed from materials that are thermally stable and chemically inert under the conditions of the procedure.

7.2 Crucible—shall be made from a non-wetting platinum alloy. The crucible shall have sufficient capacity to hold the flux and sample required for fusion. Normally, crucibles of 15 mL capacity are adequate for 32 mm diameter discs, and 25 mL capacity crucibles for 40 mm diameter discs.

NOTES:

- 1 Either platinum/gold or platinum/gold/rhodium alloys are suitable.
- 2 Because the crucible and lid are to be used for fusion work, the normal precautions associated with the care of hot platinum ware should be observed. It is necessary, therefore, to have suitable tongs and crucible-resting surface. Stainless steel or titanium tongs are a suitable alternative to platinum-tipped tongs. The hot crucible can be rested on a refractory surface which has to be kept very clean.
- 3 Although the crucible is fabricated from an alloy that is not wetted by the glass, for the most precise work the crucible should be cleaned between each fusion. The use of 20% w/v citric acid or dilute (10–50% v/v) hydrochloric acid has proven to be suitable for crucible cleaning. The use of an ultrasonic bath will accelerate this process. An alternative method of cleaning is to fuse several grams of flux in the crucible, moving the melt around so as to clean the entire inner surface. The molten flux is poured from the crucible. If a droplet adheres to the crucible, this can easily be flaked off when the crucible is cold.

7.3 Mould—shall be made from non-wetting material, for example platinum alloy is commonly used for casting discs and aluminium or graphite is suitable for press quenching.

In the casting technique, the bottom of the disc is the analytical surface. The mould should therefore be flat and sufficiently thick that it is not easily deformed. Casting moulds should be regularly checked for flatness and should be polished regularly to ensure that the disc releases from the mould. Platens for press quenching are constructed so that the depth gradually increases from the perimeter to the centre. The analytical surface for press quenched discs is that surface which contacts the plunger head. Caution should be exercised not to choose the platen side for intensity measurements.

7.4 Crucible tongs—platinum tipped or stainless steel or titanium.

7.5 Desiccator—containing freshly regenerated, self-indicating silica gel.

7.6 Sample holders—for specimen presentation, matched in accordance with AS 2563.

7.7 X-ray fluorescence spectrometer—any conventional wavelength dispersive (sequential, simultaneous or combination simultaneous/sequential), vacuum path X-ray fluorescence spectrometer may be used, provided that it conforms to precision requirements of AS 2563 at the 0.1% (10^6 counts) precision level.

8 SAMPLE

8.1 Coal and coke ash prepared in the laboratory The coal or coke sample shall be the analysis sample, prepared to a nominal top size of $-212\ \mu\text{m}$. Sample preparation procedures are described in AS 4264, Parts 1 and 2.

The sample shall then be ashed by the procedure specified in AS 1038.3 (coal) or AS 1038.4 (coke) as appropriate.

8.2 Coal and coke ash Coal or coke ash received in the laboratory shall be heated at 815°C for 15 min, and cooled in a desiccator over freshly regenerated, self-indicating silica gel immediately prior to weighing for analysis.

NOTE: Boiler ash received in the laboratory should be ground to a nominal top size of $-63\ \mu\text{m}$ and dried at 105°C for 1 h then stored in a desiccator over freshly regenerated, self-indicating silica gel.