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METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

Part 14.2—ANALYSIS OF HIGHER RANK COAL ASH AND COKE ASH (ACID DIGESTION—FLAME ATOMIC ABSORPTION SPECTROMETRIC METHOD)

STANDARDS ASSOCIATION OF AUSTRALIA
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The following interests are represented on Committee MN/1:

Australasian Institute of Mining and Metallurgy

Australian Coal Association

Australian Coal Industry Research Laboratories Ltd

Australian Institute of Energy

Bureau of Steel Manufacturers of Australia

Coal Preparation Societies of New South Wales and Queensland

Confederation of Australian Industry

CSIRO, Division of Fossil Fuels

Department of Mineral Resources, N.S.W.

Department of Mines, Qld

Department of National Development

Electricity Supply Association of Australia

Institution of Engineers, Australia

Joint Coal Board

Queensland Coal Board

Royal Australian Chemical Institute

Standing Committee on Coalfield Geology, N.S.W.

State Chemical Laboratory, Vic.

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AUSTRALIAN STANDARD

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PREFACE

This standard was prepared by the Association's Committee on Coal and Coke, as a companion standard to AS 1038, Part 14.1, Analysis of Coal Ash, Coke Ash and Mineral Matter (Borate Fusion—Flame Atomic Absorption Spectrometric Method). Whereas Part 14.1 specifies decomposition of the ash by borate fusion, this standard sets out a bomb digestion procedure using a hydrochloric acid/hydrofluoric acid mixture for the decomposition of higher rank coal ash and coke ash, followed by flame atomic absorption spectrometry for the determination of various elements in the ash.

By convention, both phosphorus and sulphur are included in the normal ash constituent analysis.

Phosphorus is determined spectrophotometrically in the solution prepared from the ash, while sulphur can be determined by the high temperature or gravimetric procedures.

The Committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the method. The following laboratories participated in the test program:

Australian Coal Industry Research Laboratories Ltd
Cargo Superintendents Co. (A/Asia) Pty Ltd
Newcastle Wallsend Coal Co. Pty Ltd
R.W. Miller and Company Pty Limited
SGS Australia Pty Ltd

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1 SCOPE. This standard sets out methods for the determination of aluminium, calcium, iron, magnesium, manganese, potassium, silicon, sodium and titanium in higher rank coal ash and coke ash by flame atomic absorption spectrometry, and for the determination of phosphorus in higher rank coal ash and coke ash by spectrophotometry. Higher rank coal is defined as coal having a gross specific energy of 27 MJ/kg or greater on a dry, ash-free basis.

NOTE: Although a volumetric approach has been specified for solution preparation, a gravimetric alternative may be used.

2 APPLICATION. This method is applicable to the determination of the elements listed in Clause 1 (expressed as oxides), in the concentration ranges specified in Table 1.

Table 1 has been devised to accommodate any type of ash which is likely to arise from Australian higher rank coals or cokes. The results in Appendix A have been obtained from test work carried out on the available samples. Although testing was not carried out with a comprehensive range of likely coal compositions, the method is expected to apply over the wider ranges covered by the calibration standards and indicated in Table 1.

TABLE 1

CONCENTRATION RANGES

Element	Reported as oxide	Range, percent
Silicon	SiO_2	20 to 85
Aluminium	Al_2O_3	10 to 50
Iron	Fe_2O_3	0.1 to 15
Calcium	CaO	0.05 to 25
Magnesium	MgO	0.05 to 25
Sodium	Na_2O	0.05 to 12.5
Potassium	K_2O	0.05 to 12.5
Titanium	TiO_2	0.1 to 5
Manganese	Mn_3O_4	0.005 to 0.6
Phosphorus	P_2O_5	0.01 to 5

3 REFERENCED DOCUMENTS. The following standards are referred to in this standard:

AS 1038 Methods for the Analysis and Testing of Coal and Coke
Part 3 —Proximate Analysis of Hard Coal
Part 16—Reporting of Results*
AS 1152 Test Sieves
AS 2134 Chemical Analysis of Materials by Flame Atomic Absorption Spectroscopy
AS CK19 Code of Recommended Practice for the Chemical Analysis of Materials by Ultraviolet/Visible Spectrophotometry
ISO 5725 Precision of Test Methods—Determination of Repeatability and Reproducibility by Inter-laboratory Tests

4 PRINCIPLE. The sample is decomposed by digestion with hydrofluoric acid/hydrochloric acid mixture in a PTFE-lined bomb or polypropylene bottle. Boric acid is added to the digestion mixture to complex excess fluoride and the resulting solution is aspirated into an atomic absorption spectrometer after the appropriate dilutions have been made.

Phosphorus is determined spectrophotometrically on an aliquot of the diluted solution.

5 APPARATUS. The following apparatus is required:

5.1 Glassware. Grade A volumetric glassware shall be used throughout.

5.2 Atomic absorption spectrometer. The instrument and its usage shall comply with AS 2134. The instrument shall have burners suitable for use with air/acetylene and nitrous oxide/acetylene flames. The suggested flames and wavelengths are given in Table 2. Other parameters shall be adjusted in accordance with the manufacturer's instructions.

* In course of revision.