AS 1038.9.1—1992

Australian Standard®

Coal and coke—Analysis and testing

Part 9.1: Coal and coke—Phosphorus—Ash digestion/molybdenum blue method

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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

Australian Coal Preparation Society

Australian Institute of Energy

Bureau of Steel Manufacturers of Australia

Confederation of Australian Industry

CSIRO, Division of Coal and Energy Technology

Department of Resource Industries, Queensland

Electricity Supply Association of Australia

Institution of Engineers, Australia

Joint Coal Board

National Association of Testing Authorities, Australia

Queensland Coal Board

Royal Australian Chemical Institute

Standing Committee on Coalfield Geology of New South Wales

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PREFACE

This Standard was prepared by the Standards Australia Subcommittee on Coal Evaluation, under the supervision of the Committee on Coal and Coke, as a revision in part of AS 1038.9–1977, *Methods for the analysis and testing of coal and coke*, Part 9: *Phosphorus in coal and coke*. Major differences from the previous edition are as follows:

(a) Division of AS 1038.9 into the following individual parts:

Part 9.1: Ash digestion/molybdenum blue method
Part 9.2: Coal extraction method
Part 9.3: Ash digestion method

Part 9.3 is a reproduction of the method currently contained in AS 1038.14.2, *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)*

(b) The addition of a pH adjustment prior to colour development in the dry oxidation procedure.

(c) Deletion of the wet oxidation procedure.

This Standard is not technically equivalent to ISO 622:1981, *Solid mineral fuels – Determination of phosphorus content – Reduced molybdophosphate photometric method*.

CONTENTS

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Part 9.1: Coal and coke – Phosphorus – Ash digestion/molybdenum blue method

1 SCOPE This Standard sets out a method for the spectrophotometric determination of phosphorus in coal and coke, as molybdenum blue.

This method is applicable also to the determination of phosphorus as phosphorus pentoxide in coal ash and coke ash.

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

AS	
1038	Methods for the analysis and testing of coal and coke
1038.3	Part 3: Proximate analysis of higher rank coal
1038.4	Part 4: Proximate analysis of coke
1038.16	Part 16: Acceptance and reporting of results
2096	Classification and coding systems for Australian coals
2243	Safety in laboratories
2508	Safe storage and handling information cards for hazardous materials
2646	Sampling of solid mineral fuels
2646.6	Part 6: Hard coal – Preparation of samples
2646.7	Part 7: Coke – Preparation of samples
2706	Numerical values – Rounding and interpretation of limiting values
3753	Recommended practice for chemical analysis by ultraviolet/visible spectrophotometry
SAA	
ASCRM-009	Certified reference coal sample
ASCRM-010	Certified reference coal ash sample

3 DEFINITIONS For the purpose of this Standard, the definitions below apply.

3.1 Higher rank coal (as defined in AS 2096) – coal having a gross specific energy of 21 MJ/kg or greater on an ash-free, moist basis *and* a gross specific energy of 27 MJ/kg or greater on a dry, ash-free basis.

3.2 Coke – the agglomerated product of coal carbonization, generally at a temperature in excess of 900°C.

3.3 Ash – the inorganic matter remaining after the coal or coke has been incinerated to constant mass under standard conditions.

4 PRINCIPLE The carbonaceous matter of the coal or coke is removed by ashing and the phosphorus extracted from the ash by treatment with sulfuric and hydrofluoric acid, silicon being volatilized as silicon tetrafluoride. The addition of a reagent solution, containing ammonium molybdate and ascorbic acid as a reducing agent, to a measured quantity of the extracted solution produces a molybdenum blue colouration, the absorbance of which is measured. The amount of phosphorus in the solution is obtained by reference to a standard solution.

Errors due to the presence of arsenic in the amounts normally found in coal ash or coke ash are insignificant.

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

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 Solutions

6.2.1 *Hydrofluoric acid,* (ρ_{20} 1.15 g/mL), 40 percent m/m.

WARNING: HARMFUL TO SKIN AND EYES.

6.2.2 *Sulfuric acid solution (5 mol/L)* Add 28 mL of sulfuric acid (ρ_{20} 1.84 g/mL) to 50 mL of water. Cool, dilute to 100 mL and mix.