

Australian Standard<sup>®</sup>

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**Coal and coke—Analysis and  
testing**

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

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This Australian Standard was prepared by Committee MN/1, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 3 February 1992 and published on 15 June 1992.

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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  
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Royal Australian Chemical Institute  
Standing Committee on Coalfield Geology of New South Wales  
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First published as AS K152.9—1965 (endorsement of  
BS 1016.9—1960 with amendment).  
Revised and redesignated AS 1038.9—1977.  
Revised and redesignated AS 1038.9.1—1992.

## 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*.

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Australian Standard

**Coal and coke – Analysis and testing**

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

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**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**, ( $\rho_{20}$  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 ( $\rho_{20}$  1.84 g/mL) to 50 mL of water. Cool, dilute to 100 mL and mix.