AS 1038.9.3—1991

Australian Standard®

Coal and coke–Analysis and testing

Part 9.3: Coal and coke–Phosphorus–Ash digestion 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 20 September 1990 and published on 11 February 1991.

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 Minerals and Energy, N.S.W.

Department of Resource Industries, Qld

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

University of New South Wales

University of Queensland

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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 and the direction of the Minerals Standards Board as a part of the subdivision of AS 1038.9–1977, *Methods for the analysis and testing of coal and coke*, Part 9: *Phosphorus in coal and coke*, into three separate Standards, as follows:

Part 9.1: 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*).

CONTENTS

STANDARDS AUSTRALIA

Australian Standard
Coal and coke–Analysis and testing

Part 9.3: Coal and coke–Phosphorus–Ash digestion method

1 SCOPE This Standard describes a method for the spectrophotometric determination of phosphorus in coal and coke, as phosphomolybdovanadate. Two alternative methods of sample decomposition are included.

This method is also applicable to the determination of phosphorus 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

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 coal or coke has been incinerated to constant mass under standard conditions.

4 PRINCIPLE The carbonaceous matter of coal or coke is removed by ashing and phosphorus is extracted from the ash by decomposition with hydrochloric and hydrofluoric acids in a sealed vessel. After complexing fluoride with boric acid, molybdovanadate reagent is added to an aliquot of the solution. The absorbance of the yellow phosphomolybdovanadate formed is measured. The amount of phosphorus in the solution is obtained from a standard calibration.

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 Boric acid (H_3BO_3)

6.3 Solutions

6.3.1 *Hydrochloric acid* (ρ_{20} 1.16 g/mL to 1.19 g/mL)

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

WARNING: HYDROFLUORIC ACID IS HARMFUL TO SKIN AND EYES.

6.3.3 *Nitric acid* (ρ 1.42 g/mL), diluted 1 + 1