

Australian Standard[®]

Coal and coke—Analysis and testing

Part 6.2: Higher rank coal and coke—Ultimate analysis—Nitrogen

This Australian Standard was prepared by Committee MN/1, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 17 January 1997 and published on 5 April 1997.

The following interests are represented on Committee MN/1:

ACIRL

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 New South Wales

CSIRO, Division of Coal and Energy Technology

Department of Mines and Energy, Queensland

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Originated as part of AS CK2.3—1949.
Previous edition AS 1038.6.2—1986 and part of AS 1038.7—1981.
Revised, amalgamated and redesignated in part as AS 1038.6.2—1997.

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 of AS 1038.6.2—1986, *Methods for the analysis and testing of coal and coke*, Part 6.2: *Ultimate analysis of higher rank coal—Determination of nitrogen*, and (in part) AS 1038.7, *Methods for the analysis and testing of coal and coke*, Part 7: *Ultimate analysis of coke*. Major differences from the previous edition are as follows:

- (a) Modification to incorporate the determination of nitrogen in coke.
- (b) Inclusion of safety aspects.

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FOREWORD

The ultimate analysis of coal and coke comprises the determination of the elements carbon, hydrogen, nitrogen and sulfur. Determination of the total amounts of these elements, regardless of their origin, is described. Carbon includes that which is present in the mineral carbonates and hydrogen includes that which is present both in moisture (for which a correction is made in the calculation) and in water of constitution in the mineral matter. All nitrogen is assumed to be present in the coal and coke substance. Sulfur is normally present in three forms: inorganic sulfides such as iron pyrites (FeS_2), inorganic sulfates associated with the mineral matter and organic sulfur in the coal and coke substance.

An estimate of the percentage of oxygen on an air-dry basis can be obtained by subtracting the sum of the determined percentages of moisture, ash, carbon, hydrogen, nitrogen and sulfur from 100. The value thus obtained should be termed 'oxygen by difference' (see AS 1038.16). A more satisfactory value for oxygen by difference is obtained when the ultimate analysis is expressed on a dry, mineral matter free basis after making all appropriate corrections.

STANDARDS AUSTRALIA

Australian Standard

Coal and coke—Analysis of testing

Part 6.2: Higher rank coal and coke—Ultimate analysis—Nitrogen

1 SCOPE This Standard sets out a method for the determination of nitrogen in the analysis sample of higher rank coal or coke by conversion to ammonium sulfate. Two methods for the distillation of ammonia are given.

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
- 2165 Burettes and bulb burettes
- 2243 Safety in laboratories
- 2418 Coal and coke—Glossary of terms
- 2508 Safe storage and handling information cards for hazardous materials
- 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

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

4 PRINCIPLE A known mass of sample is heated with concentrated sulfuric acid in the presence of a catalyst to destroy organic material and to convert nitrogen to ammonium sulfate. Ammonia liberated by the addition of excess sodium hydroxide is steam distilled, absorbed in boric acid solution and titrated directly with sulfuric acid solution.

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 Sucrose**6.3 Mixed catalyst**

6.3.1 Catalyst 1 Consisting of—

- (a) 90 parts by mass of potassium sulfate;
- (b) 2 parts by mass of selenium powder; and
- (c) 5 parts by mass of vanadium pentoxide.