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**METHODS FOR THE ANALYSIS AND
TESTING OF COAL AND COKE**

Part 6.2—ULTIMATE ANALYSIS OF HIGHER RANK COAL— DETERMINATION OF NITROGEN



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Australian Coal Industry Research Laboratories Ltd
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Coal Preparation Societies of N.S.W. and Queensland
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PREFACE

This edition of this standard was prepared by the Association's Subcommittee on Coal Evaluation under the supervision of the Committee on Coal and Coke and the direction of the Minerals Standards Board. The major differences from the 1971 edition are as follows:

- (a) Division of AS 1038.6 into the following individual standards:
 - Part 1 Determination of Carbon and Hydrogen
 - Part 2 Determination of Nitrogen
 - Part 3.1 Determination of Total Sulphur (Eschka Method)
 - Part 3.2 Determination of Total Sulphur (High Temperature Combustion Method)
 - Part 3.3 Determination of Total Sulphur (Infrared Method).
- (b) Exclusion of the method for the determination of carbonate carbon which has been published as a separate method in AS 1038.23.
- (c) Modification of the method for the determination of nitrogen.

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FOREWORD

The ultimate analysis of coal comprises the determination of the elements carbon, hydrogen, nitrogen and sulphur. 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 hydration of the argillaceous constituents of the mineral matter. All nitrogen is assumed to be present in the coal substance. Sulphur is normally present in three forms: inorganic sulphides, such as iron pyrites (FeS_2), inorganic sulphates associated with the mineral matter and organic sulphur in the coal 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 sulphur 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 ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

PART 6.2—ULTIMATE ANALYSIS OF HIGHER RANK COAL—
DETERMINATION OF NITROGEN

1 SCOPE. This standard sets out a procedure for the determination of nitrogen in the analysis sample of higher rank coal by conversion to ammonium sulphate. Two procedures for the distillation of ammonia are given.

NOTE: Higher rank coals are defined as those with a gross specific energy of 27 MJ/kg or greater on a dry, ash-free basis.

2 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 2165 Burettes and Bulb Burettes
- AS 2646 Sampling of Solid Mineral Fuels
Part 6 Hard Coal—Preparation of Samples
- BS 1041 Code for Temperature Measurement.

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

4 REAGENTS.

4.1 General. All reagents shall be of analytical reagent quality unless otherwise specified. Distilled or deionized water shall be used throughout.

NOTE: Error may arise from the presence of ammonia in the water. Ammonia may be removed either by passing through a column of a suitable ion exchange resin or by protracted boiling.

4.2 Special reagents.

4.2.1 Sucrose.

4.2.2 Mixed catalyst. Consisting of:
90 parts by mass of potassium sulphate
2 parts by mass of selenium powder
5 parts by mass of vanadium pentoxide.

The catalyst mixture must be thoroughly ground prior to use.

SAFETY NOTE: The components of the mixed catalyst are dangerous to health if inhaled or if they are in occasional contact with the skin. Both selenium and vanadium compounds are serious water pollutants and should not be discarded into the sewerage or septic systems.

4.2.3 Sulphuric acid (ρ_{20} 1.8 g/mL).

4.2.4 Standard sulphuric acid solution (0.005 mol/L).

4.2.5 Boric acid solution, saturated. Dissolve 60 g of boric acid in 1 L of hot water. Allow to stand for 3 days, decant and use the clear solution.

4.2.6 Sodium hydroxide solution. Carefully dissolve 400 g of sodium hydroxide in 500 mL of water and dilute to 1 L.

4.2.7 Screened indicator solution. Prepare the screened indicator solution as follows:

- Dissolve 0.2 g of methyl red in 10 mL of ethanol solution (300 mL/L). Add 0.1 g of sodium hydroxide and heat at 40°C to complete dissolution. Cool and dilute to 100 mL with water.
- Dissolve 0.3 g of bromocresol green in 10 mL of ethanol (950 mL/L) and dilute to 100 mL with water.
- Mix equal volumes of (a) and (b) immediately before use.

NOTE: Water soluble salts of the above indicators may be used as an alternative.

5 APPARATUS. The following apparatus is required:

5.1 Digestion flasks. Kjeldahl flasks of borosilicate glass, preferably pistol shaped, of 50 mL or 100 mL capacity.

5.2 Stopper. A light, loosely fitting, blown glass stopper (or suitable alternative) for digestion flask (5.1).

5.3 Heating unit. The flask may be heated by gas, using a microburner, or by electricity.

5.4 Distillation apparatus. A distillation apparatus of borosilicate glass, fitted with a splash head to arrest the passage of any entrained sodium hydroxide solution, an upright condenser and a conical flask. Typical distillation apparatus is shown in Fig. 1 for Procedure A and Fig. 2 for Procedure B.

5.5 Burette. A 10 mL class A burette complying with AS 2165.

6 SAMPLES. Coal used shall be the analysis sample prepared in accordance with AS 2646.6 and ground to pass a 212 μm test sieve complying with AS 1152.

The sample received in the laboratory shall be brought into approximate equilibrium with the laboratory atmosphere by exposing it in a thin layer on a tray. Exposure time shall be kept to the minimum necessary, particularly with coals liable to oxidation. The sample shall be thoroughly mixed, preferably by mechanical means, immediately before the determination.