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METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

Part 6.3.1—ULTIMATE ANALYSIS OF HIGHER RANK COAL— DETERMINATION OF TOTAL SULPHUR (ESCHKA METHOD)

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Australasian Institute of Mining and Metallurgy
Australian Coal Association
Australian Coal Industry Research Laboratories Ltd
Australian Institute of Energy
Bureau of Steel Manufacturers of Australia
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 to the Eschka method for the determination of sulphur.

CONTENTS

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 the 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 6.3.1—ULTIMATE ANALYSIS OF HIGHER RANK COAL—DETERMINATION OF TOTAL SULPHUR (ESCHKA METHOD)

1 SCOPE. This standard sets out procedures for the determination of total sulphur in the analysis sample of higher rank coal by the Eschka method.

NOTES:

1. Where the temperature for a particular operation is specified as a definite figure, this figure should be attained as closely as possible and it should be subject only to errors inherent in accurate measurement, as defined in BS 1041.
2. Where a temperature range is given, the temperature may be anywhere in the range without detriment to the result. However, the mean of the range should be aimed at in order that errors inherent in measurement do not cause a temperature outside the specified range to be used inadvertently.
3. 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 2167	Straight Pipettes
AS 2646	Sampling of Solid Mineral Fuels Part 6 Hard Coal—Preparation of Samples
BS 1041	Code for Temperature Measurement
ASTM D3177	Total Sulphur in the Analysis Sample of Coal and Coke.

3 PRINCIPLE. A known mass of the sample is ignited in intimate contact with Eschka mixture in an oxidizing atmosphere at 800 °C to decompose organic material and to convert all sulphur to sulphate. The sulphate is then dissolved in dilute hydrochloric acid and determined gravimetrically by precipitation with barium chloride.

4 REAGENTS.

4.1 General. Unless otherwise specified, all reagents shall be of analytical reagent quality. Distilled or deionized water shall be used throughout.

4.2 Special reagents.

4.2.1 *Eschka mixture,* (light calcined magnesium oxide (MgO) and anhydrous sodium carbonate (Na_2CO_3) in the ratio of 2:1 by mass).

4.2.2 *Hydrochloric acid,* (ϱ_{20} 1.18 g/mL).

4.2.3 *Ammonia solution,* (ϱ_{20} 0.88 g/mL).

4.2.4 *Barium chloride solution* (100 g/L). Dissolve 100 g of crystalline barium chloride dihydrate ($BaCl_2.2H_2O$) in water and dilute to 1 L.

4.2.5 *Potassium sulphate solution* (2 g/L). Weigh about 2 g (to the nearest 0.1 mg) of potassium sulphate (K_2SO_4), previously dried at 110 °C for 2 h. Dissolve in water and dilute to 1 L in a volumetric flask.

4.2.6 *Silver nitrate solution* (16 g/L). Dissolve 16 g of silver nitrate ($AgNO_3$) in water and dilute to 1 L.

4.2.7 *Methyl red indicator.* Dissolve 0.02 g of methyl red in 60 mL of ethanol (950 mL/L) and dilute to 100 mL with water.

NOTE: A water soluble salt of the above indicator may be used as an alternative.

5 APPARATUS. The following apparatus is required:

5.1 Muffle furnace. An electrically heated muffle furnace capable of maintaining a zone within the range of 800 ±25 °C. The ventilation shall be such as to give at least 4 air changes per minute at 800 °C.

NOTE: This is the type of furnace specified for the determination of ash in AS 1038.3, but sulphur and ash determinations should not be carried out simultaneously in the same furnace as the Eschka mixture absorbs oxides of sulphur.

5.2 Crucibles. Shallow crucibles of glazed porcelain or platinum, approximately 25 mL capacity.

5.3 Silica plate. A silica plate, 6 mm thick, that is an easy sliding fit in the muffle furnace.

5.4 Silica crucibles. Crucibles of approximately 25 mL volume.

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.

7 PROCEDURE.

7.1 Determination of sulphur in the sample. The procedure shall be as follows:

(a) Weigh about 1 g of the sample to the nearest 0.1 mg and mix intimately with 3 g of the Eschka mixture (4.2.1) in the crucible (5.2). Level the contents of the crucible and cover with a layer of 1 g of the Eschka mixture. Concurrently with weighing the sample for analysis, weigh another sample for determining the moisture in the coal by one of the methods specified in AS 1038.3.

NOTE: For coals containing more than 5 percent sulphur, reduce the mass of coal taken to 0.5 g.

(b) Place the charged crucible into the cold muffle furnace. Raise the temperature of the muffle furnace