

Australian Standard™

**Methods for the analysis and testing of
lower rank coal and its chars**

**Part 6: Lower rank coal—Ultimate
analysis—Classical methods**



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This Standard was issued in draft form for comment as DR 02276.

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RECONFIRMATION

OF

AS 2434.6—2002

Methods for the analysis and testing of lower rank coal and its chars

Part 6: Lower rank coal—Ultimate analysis—Classical methods

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Approved for reconfirmation in accordance with Standards Australia procedures for reconfirmation on 15 July 2013.

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NOTES

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Originated as AS 2434.6.1—1986.
Revised and redesignated as AS 2434.6—2002.

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Published by Standards Australia International Ltd
GPO Box 5420, Sydney, NSW 2001, Australia
ISBN 0 7337 4855 4

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PREFACE

This Standard was prepared by Standards Australia Committee MN-001, Coal and Coke, to supersede AS 2434.6.1—1986, *Methods for the analysis and testing of lower rank coal and its chars*, Part 6.1: *Ultimate analysis of lower rank coal—Classical methods*.

This Standard is based on AS 1038.6.1, *Coal and coke—Analysis and testing*, Part 6.1: *Higher rank coal and coke—Ultimate analysis—Carbon and hydrogen* and AS 1038.6.2, *Coal and coke—Analysis and testing*, Part 6.2: *Higher rank coal and coke—Ultimate analysis—Nitrogen*, and has been specifically modified to meet the analytical requirements of lower rank Australian coals.

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FOREWORD

As a general rule, Standards suitable for lower rank coals can also be applied to higher rank coals, but the reverse does not apply. Some features specific to lower rank coal methods are as follows:

- (a) *Sample mass* Because of the greater heterogeneity of lower rank coals, relatively large sample masses are essential for successful analyses, and semi-micro methods are not applicable.
- (b) *Sample condition* Many lower rank coals are powerful desiccants and adsorb water even in desiccators. Preparation of lower rank coal samples for analysis should involve either air equilibration followed by a moisture determination concurrent with the analysis, or complete drying and encapsulation of the sample.
- (c) *Carbon and hydrogen* The Liebig principle is preferred for the determination of carbon and hydrogen in lower rank coals, because some high volatile coals can explode under the heating conditions of the high temperature method.

The ultimate analysis of coal comprises the determination of the elements carbon, hydrogen, nitrogen, sulphur and oxygen. This Standard describes the determination of the total amounts of the first four elements, regardless of their mode of occurrence; oxygen is estimated by difference, because simple direct methods of sufficient accuracy are not available. Hydrogen includes that present both in the moisture and in the water of hydration of constituents of the mineral matter. All nitrogen is assumed to be present in the coal substance. Sulphur is normally present in three forms: as pyrite (FeS_2) and other sulfides, as inorganic sulphates associated with the mineral matter, and as organic sulphur compounds in the coal substance.

The data obtained from use of this Standard will not lead to a useful estimate of the percentage of oxygen in lower rank coal. An accurate estimate of the oxygen content can only be obtained by expressing the carbon, hydrogen, nitrogen and sulphur contents on a dry, mineral and inorganic free (dmif) basis. This in turn requires a separate determination of coal minerals and inorganic matter*. The dmif expression of results for lower rank coals is discussed in AS 1038.16, *Coal and coke—Analysis and testing*, Part 16: *Assessment and reporting of results*.

Where the temperature for a particular operation is specified as a definite figure, it is to be understood that this figure should be attained as closely as possible and that it should be subject only to the errors inherent in accurate measurement.

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 targeted so the inherent errors in measurement do not cause a temperature outside the specified range to be used inadvertently.

* KISS, L.T., and KING, T.N. *Fuel*, 1977, 56, 340; *ibid*, 1979, 58, 437.

STANDARDS AUSTRALIA

Australian Standard

Methods for the analysis and testing of lower rank coal and its chars

Part 6: Lower rank coal—Ultimate analysis—Classical methods

1 SCOPE

This Standard sets out the method for the determination of the ultimate analysis of lower rank coals and char in terms of their carbon, hydrogen, nitrogen and sulphur contents.

2 REFERENCED DOCUMENTS

The following Standards are referred to in this Standard:

AS

- 1038 Coal and coke—Analysis and testing
- 1038.3 Part 3: Proximate analysis of higher rank coal
- 1038.8.2 Part 8.2: Coal and coke—Chlorine—High-temperature combustion method
- 1038.16 Part 16: Assessment and reporting of results
- 1038.23 Part 23: Higher rank coal—Carbonate carbon
- 1152 Specification for test sieves
- 2165 Laboratory glassware—Burettes
- 2243 Safety in laboratories (series)
- 2167 Graduated straight pipettes
- 2418 Coal and coke—Glossary of terms
- 2434 Methods for the analysis and testing of lower rank coal and its chars
- 2434.7 Part 7: Lower rank coal—Determination of moisture in the analysis sample
- 2508 Safe storage and handling information card (series)
- 4264 Coal and coke—Sampling
- 4264.3 Part 3: Lower rank coal—Sampling procedures

ISO

- 1994 Hard coal—Determination of oxygen content

BS

- 1752 Laboratory sintered or fritted filters including porosity grading

3 DEFINITIONS

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

4 SAFETY

For information on laboratory safety, reference should be made to the relevant parts of AS 2243 and AS 2508.

5 SAMPLE

5.1 General

The coal used for the ultimate analysis is the analysis sample ground to pass a 212 μm test sieve complying with AS 1152, taken and prepared in accordance with AS 4264.3. The analysis sample may be prepared to either one of two basis, air-dry or dry. If an air-dry sample is used, samples for moisture determination shall be taken and analysed concurrently with the ultimate analysis.

5.2 Preparation of air-dry coal

The sample received in the laboratory shall be brought into approximate equilibrium with the laboratory atmosphere by exposing it in a thin layer of not more than 15 mm on a tray. As many lower rank coals are prone to oxidation, the exposure time shall be kept to a minimum. The sample shall be thoroughly mixed, preferably by mechanical means, immediately before the determination.

5.3 Preparation of dry coal

5.3.1 *Sample for carbon and hydrogen*

Air-dry coal shall be used to prepare the dry sample. A small platinum or silica boat with approximate dimensions of length 25 mm, width 10 mm and height 10 mm shall be used to contain the sample.

Place a clean, dry boat in a small weighing bottle and fit a lid to it (see Figure 1). Weigh to the nearest 0.1 mg, remove the boat and fill it with 0.2 g to 0.3 g of coal. Heat the boat in a nitrogen oven at 105°C to 110°C in accordance with AS 2434.7. On removing the boat from the oven, immediately place it back in the cold weighing bottle and fit the lid. Immediately reweigh the sealed weighing bottle, containing the hot boat and coal, to the nearest 0.1 mg; remove the boat and place it in the combustion tube.

NOTE: The dry coal is extremely hygroscopic and cannot be stored in a desiccator. It is essential that the time between removing the boat (containing the dry coal) from the oven and placing it in the combustion tube be kept to less than 60 s.

Placing the hot boat in a small sealed container, such as a small weighing bottle, for reweighing serves two purposes. The small volume of the container limits the volume of air from which moisture can be absorbed. The container also eliminates buoyancy effects from the weighing of a hot sample since the container does not heat in the time taken to make the weighing.

5.3.2 *Sample for nitrogen and sulphur*

The dry coal sample for determination of nitrogen and sulphur shall be prepared as follows:

- (a) Place about 2 to 3 g of air-dry coal in a wide glass weighing bottle (lid removed).
- (b) Dry in an air oven at 110°C for 1.5 h.
- (c) Place lid on weighing bottle and cool in a desiccator.

NOTE: A very small adsorption of moisture from the atmosphere in the desiccator will not affect the result significantly. However, the dry coal sample should not be stored in the desiccator for more than 3 h.

6 DETERMINATION OF CARBON AND HYDROGEN

6.1 Introduction

Two methods are specified for the determination of the total percentages of carbon and hydrogen in coal, viz combustion at 800°C (Clause 6.2) and combustion at 1350°C (Clause 6.3).

The determined figures include the carbon present as carbonates (if any) and the hydrogen present as moisture in the coal and as water of hydration of the mineral matter.

At this stage no correction is made to the carbon figure for the presence of carbonates but the hydrogen figure is corrected for the hydrogen present as moisture in the coal.

Combustion of coal in oxygen at 800°C yields some nitric oxide which may be partially oxidized to nitrogen dioxide. The error that this causes in the hydrogen determination is, under the conditions stated, sufficiently small to be ignored. The result for carbon would, however, be about 0.2 percent high. To avoid this error, provision is made for the absorption of the oxides of nitrogen prior to the absorption of the carbon dioxide. No oxides of nitrogen from N₂ are produced in the combustion at the temperatures used.

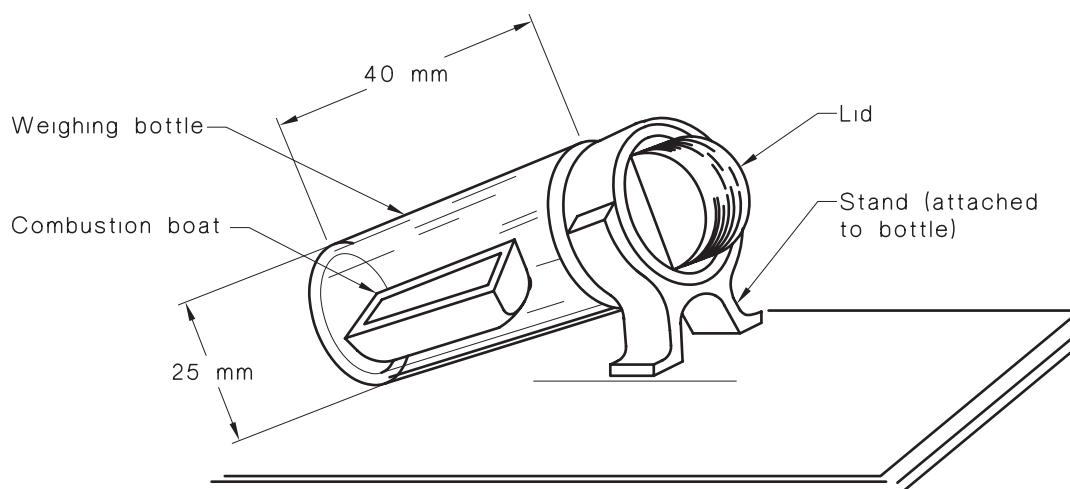


FIGURE 1 WEIGHING BOTTLE AND BOAT USED IN THE PREPARATION OF DRY COAL FOR THE ULTIMATE ANALYSIS BY THE LIEBIG PRINCIPLE

6.2 Combustion at 800°C (Liebig principle)

6.2.1 Principle

The coal is burned in a current of oxygen and the products of combustion are passed over copper oxide at 800°C. This ensures conversion of all the carbon to carbon dioxide and all the hydrogen to water. Oxides of sulphur are retained in the combustion tube by lead chromate, and chlorine by silver gauze. The water formed is absorbed by magnesium perchlorate, the nitrogen dioxide by granular manganese dioxide and the carbon dioxide by sodium hydroxide on an inert granular base, in that order.

6.2.2 Special apparatus

The following apparatus is required:

- (a) *Purification train* A purification train for removing water and carbon dioxide from oxygen. Assemble the train using the following reagents in the order stated:
 - (i) Magnesium perchlorate for removing water.
 - (ii) Sodium hydroxide on an inert granular base for removing carbon dioxide.
 - (iii) Magnesium perchlorate for removing the water produced in the reaction between carbon dioxide and sodium hydroxide.

The train should be large enough to minimize the necessity for frequent recharging.

- (b) *Control of oxygen flow rate* A reducing valve on the oxygen cylinder, together with a small needle valve before the purification train, is adequate.
- (c) *Flowmeter* A flowmeter, preferably of the variable orifice type, to measure the oxygen flow rate.
- (d) *Heating unit* A heating unit, which consists of three electrically heated furnaces. Furnace No 1, 240 mm long, heats the boat and its contents to 800°C; Furnace No 2, 470 mm long, maintains the copper oxide section of the tube at 800°C; Furnace No 3, 240 mm long, covers the lead chromate and the silver gauze and heats them to 600°C. The temperatures shall be measured by thermocouples permanently installed in the apparatus.
- (e) *Combustion tube* A tube of silica, mullite, impervious porcelain or suitable hard glass, 12 mm to 25 mm internal diameter by 1.27 m long.
- (f) *Combustion boat* A boat of platinum or silica, approximately 25 mm long by 10 mm deep by 10 mm wide is suitable.
- (g) *Absorption train* A typical absorption train is shown in Figure 2.

Suitable absorption vessels for water, carbon dioxide and oxides of nitrogen are stoppered U-tubes and Midvale tubes. A specially designed absorption tube for nitrogen dioxide is shown in Figure 3. Assemble the train using the following reagents in the order stated:

- (i) Magnesium perchlorate to absorb water produced by combustion of the coal.
NOTES:
 - 1 The spent magnesium perchlorate should be replaced daily.
 - 2 Care should be taken when disposing of exhausted magnesium perchlorate. It should be washed down the sink with a stream of water.
- (ii) Granular manganese dioxide to absorb nitrogen dioxide formed during the combustion.
- (iii) Magnesium perchlorate to absorb water evolved from the manganese dioxide.
- (iv) Sodium hydroxide on an inert granular base to absorb carbon dioxide produced by combustion of the coal.
- (v) Magnesium perchlorate to absorb water produced in the reaction between carbon dioxide and sodium hydroxide.

The train shall terminate with a flowmeter to indicate that oxygen is flowing through the apparatus.

- (h) *Duplicate results* It is recommended that duplicate results be obtained on different furnace and absorption train systems. This will show immediately when absorbers such as PbCrO_4 are exhausted. With a single apparatus, weighing the MnO_2 will also show when PbCrO_4 is exhausted.