Australian Standard®

METHODS FOR THE ANALYSIS AND TESTING OF BROWN COAL AND BROWN COAL CHAR

Part 5—DETERMINATION OF MOISTURE IN BULK SAMPLES AND IN ANALYSIS SAMPLES OF CHAR FROM LOWER RANK COAL

This Australian standard was prepared by Committee MN/1, Coal and Coke. It was approved on behalf of the Council of the Standards Association of Australia on 4 July 1984 and published on 5 October 1984.

The following interests are represented on Committee MN/1:

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 New South Wales and Queensland

Confederation of Australian Industry

CSIRO, Division of Fossil Fuels

Department of Mineral Resources, N.S.W.

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Electricity Supply Association of Australia

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Joint Coal Board

Queensland Coal Board

Royal Australian Chemical Institute

Standing Committee on Coalfield Geology, N.S.W.

State Chemical Laboratory, Vic.

Universities

First published 1984

This standard was issued in draft form for comment as DR 83100.

ISBN 0 7262 3501 6

PREFACE

This standard was prepared by the Association's Committee on Coal and Coke under the direction of the Minerals Standards Board. Procedures for the determination of moisture in coal and coke as set out in AS 1038, Methods for the Analysis and Testing of Coal and Coke, are not applicable to char from lower rank coal because some of the moisture is strongly adsorbed on to the char and is not completely removed at 110°C. Therefore the moisture content determined by the methods in AS 1038 tends to be lower than the amount of moisture actually present in the char.

Char from lower rank coal readily absorbs moisture from the air, to an equilibrium content of about 12 percent. If the moisture content of the char sample is much different from this equilibrium content, the moisture content will change during the sample preparation and analysis procedures specified for coal and coke, and so special procedures are necessary.

Char from lower rank coal is utilized often as a premium grade carbon and the moisture is a diluent of the carbon content. It may also be a source of undesirable hydrogen. Therefore it is important to know the moisture content of the bulk char sample.

The accurate determination of moisture content is also essential to the volatile matter determination which is the most frequently used commercial criterion of char quality. If the moisture content is erroneously low, the volatile matter result will be erroneously high by the same amount. The same consideration applies to the elemental hydrogen content.

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PART 5—DETERMINATION OF MOISTURE IN BULK SAMPLES AND IN ANALYSIS SAMPLES OF CHAR FROM LOWER RANK COAL

1 SCOPE. This standard sets out procedures for the determination of the moisture content of bulk samples and analysis samples of char from lower rank coal.

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 16—Reporting of Results

AS 2409 Interchangeable Conical Ground Glass Joints

AS R25 Dean and Stark Apparatus

3 DETERMINATION OF THE MOISTURE CONTENT OF THE BULK SAMPLE.

3.1 Principle. Azeotropic distillation of the bulk char sample with toluene followed by measurement of the volume of water collected.

3.2 Sample preparation. A 1 kg subsample of the bulk sample is crushed quickly in a jaw crusher to a particle size of minus 5.6 mm.

NOTE: The sample must be taken and prepared, and the determination carried out, within 5 days of the bulk sample having been taken because, even when the char is stored in a closed container, its moisture content can change with prolonged storage.

3.3 Apparatus.

3.3.1 *Dean and Stark apparatus.* The apparatus specified in AS R25, with a 10 mL receiver shall be used.

NOTES:

1. It is convenient to use a larger distillation flask, with a larger mouth e.g. a 1 L round-bottom flask with a B55 mouth complying with AS 2409, and a B24-29/B55 adaptor to fit the standard receiver.
2. Boiling aids may be added to reduce bumping.

3.3.2 *Balance.* Capable of weighing to the nearest 0.01 g.

3.4 Toluene. Toluene which distils in the range 109.5°C to 112.0°C. Not more than 5 percent shall distil below 109.5°C and not more than 5 percent shall distil above 112.0°C.

WARNING: Toluene is flammable and toxic by inhalation, ingestion or skin absorption.

3.5 Procedure. The procedure shall be as follows:

(a) Weigh, to the nearest 0.01 g, approximately 100 g of char (m) prepared in accordance with Clause 3.2 and transfer to the distillation flask.

NOTE: There is a risk of glass breakage during the distillation. This could result in a toluene fire. It is recommended that the apparatus be installed in a suitably fire-proof area where any burning toluene can be contained and extinguished.

(b) Add 250 mL of toluene to the flask and fit the flask to the condenser and receiver.

(c) Bring the toluene to the boil (10 min to 15 min) and keep it boiling actively for 1 h. Collect the water in the receiver.

(d) Run the water carefully into a dry weighed weighing bottle (m_1) and weigh the bottle plus water (m_2) to the nearest 0.01 g.

NOTE: Care must be taken to ensure that all water runs into the weighing bottle and that it is not lost as droplets adhering to the walls of the apparatus.

3.6 Calculation. Calculate the moisture content of the bulk char sample from the following formula:

$$M_{as} = \frac{m_2 - m_1}{m} \times 100$$

where

M_{as} = moisture content of the bulk char sample, as a percentage

m_1 = mass of dry, empty weighing bottle, in grams

m_2 = mass of weighing bottle plus water, in grams

m = mass of char sample, in grams.

4 DETERMINATION OF MOISTURE IN THE ANALYSIS SAMPLE.

4.1 Principle. The char sample is allowed to attain equilibrium with the atmosphere. It is then ground to pass a 212 μm sieve. The moisture in the sample is determined by heating the sample at 240°C in a stream of nitrogen and collecting the evolved water by absorption in anhydrous magnesium perchlorate.

4.2 Sample preparation. Allow the char sample to attain equilibrium with the atmosphere then crush the char to pass a 212 μm test sieve. Store the crushed sample in an air-tight container.

NOTE: The container should be well-filled to minimize the volume of air in contact with the sample.

4.3 Apparatus.

4.3.1 *Aluminium heating block.* An aluminium heating block capable of maintaining temperature between 235°C and 245°C over a distance of 100 mm is used to accommodate the glass drying tube/tubes. Depending upon the number of samples to be analysed daily, up to 6 tubes may be accommodated in parallel in the one heating block. A suitable heating block is shown in Fig. 1.

4.3.2 *Borosilicate glass tubes.* Borosilicate glass tubes, each 25 mm internal diameter, which pass right through the heating block. A suitable tube is shown in Fig. 2.

4.3.3 *Borosilicate glass boats.* Borosilicate glass boats which fit into the glass tubes and slide freely within the tubes. A convenient size for the boats is 50 mm × 10 mm × 10 mm.

NOTE: When the boats are not in use, they should be kept in a desiccator over anhydrous magnesium perchlorate.