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METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

Part 23—DETERMINATION OF CARBONATE CARBON IN HIGHER RANK COAL

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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 New South Wales and Queensland
Confederation of Australian Industry
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Department of Minerals and Energy, Vic.
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Electricity Supply Association of Australia
Institution of Engineers, Australia
Joint Coal Board
Royal Australian Chemical Institute
Standing Committee on Coalfield Geology, N.S.W.
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Australian Iron and Steel Pty Ltd
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PREFACE

This standard was prepared by the Association's Committee on Coal and Coke as a new method for the determination of carbonate carbon in coal and coke, to supersede the method in AS 1038, Part 6—1971, Ultimate Analysis of Coal. This method has been separated from Part 6 because the determination of carbonate carbon, while required for the correction of the total carbon in the coal, is not part of the ultimate analysis.

The carbonate carbon content of the mineral carbonates of a coal is required to correct the determined percentage of total carbon and volatile matter when calculating organic carbon and organic volatile matter respectively.

This standard specifies only the gravimetric method for the determination of carbonate carbon. The manometric and titrimetric methods have been deleted as they are no longer commonly used.

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STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard
for
METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

PART 23—DETERMINATION OF CARBONATE CARBON IN HIGHER RANK COAL

1 SCOPE. This standard sets out a method for the determination of carbonate carbon, from mineral carbonates, in the analysis sample of higher rank coal (i.e. coal with a specific energy > 27 MJ/kg on a dry ash-free basis).

NOTES:

1. The result obtained will include any carbon dioxide which has been absorbed by the coal.
2. For determinations required on low temperature oxidation residues, reference should be made to: Talanta, Vol. 22, pp 751—753. Pergamon Press (1975).

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 1152	Test Sieves.

3 PRINCIPLE. The coal is treated with hydrochloric acid and the carbon dioxide evolved from the decomposition of the carbonates is absorbed and weighed.

4 SAMPLES. The coal used shall be the analysis sample 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. The 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.

5 SPECIAL APPARATUS. Any system comprising a purification train, a reaction flask assembly and an absorption train may be used. A suitable apparatus is shown in Fig. 1 and consists of the following:

(a) *Purification train.* An absorption unit containing soda-asbestos.

(b) *Reaction flask assembly.* A 250 mL, 3-necked round bottom flask fitted with a tap funnel, a double-surface condenser and an inlet tube. The end of the inlet tube to be within 5 mm of the bottom of the flask.

A flow meter is connected to the inlet side of the reaction flask.

(c) *Absorption train.* Three vessels (Midvale, Nesbitt or U-tubes) packed as follows:

(i) Magnesium perchlorate to dry the gas.
(ii) Copper(II) phosphate granules, prepared as described in Clause 6.3.
(ii) Soda-asbestos to absorb carbon dioxide, followed by a guard of magnesium perchlorate to absorb water produced in the reaction between carbon dioxide and soda-asbestos.

(d) *Guard tube.* An absorption unit containing soda-asbestos to prevent the ingress of carbon dioxide from the atmosphere.

6 REAGENTS.

6.1 General. Unless otherwise specified, all reagents shall be of analytical reagent quality. Distilled or deionized water, free from carbon dioxide shall be used.

6.2 Hydrochloric acid (300 mL/L). Add 300 mL of hydrochloric acid (ϱ_{20} 1160 kg/m^3 to 1180 kg/m^3) to 700 mL of water.

6.3 Copper(II) phosphate granules*, specially prepared by pressing a stiff paste of the powder reagent in 1 percent starch solution, through a sheet of perforated metal (approximately 1 mm aperture), and drying at 110°.

6.4 Magnesium perchlorate anhydrous (− 1.2 + 0.7 mm).

6.5 Soda-asbestos (− 2.8 + 1.4 mm and − 1.4 + 0.8 mm).

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*Reference: P.G. Jeffery, Chemical Methods of Rock Analysis, 2nd Edition, pp 177—180. Pergamon Press (1975).