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METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

Part 22—DIRECT DETERMINATION OF MINERAL MATTER AND WATER OF HYDRATION OF MINERALS IN HARD COAL

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Represented on the committee which was responsible for the preparation of this standard were the following:

Australian Coal Association

Australian Institute of Energy

Australasian Institute of Mining and Metallurgy

Bureau of Steel Manufacturers of Australia

Coal Preparation Societies of N.S.W. and Queensland

Confederation of Australian Industry

CSIRO, Division of Fossil Fuels

Department of Minerals and Energy, Victoria

Department of Mineral Resources, N.S.W.

Department of National Development and Energy

Electricity Supply Association of Australia

Institution of Engineers, Australia

Joint Coal Board

Queensland Coal Board

Royal Australian Chemical Institute

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PREFACE

This standard was prepared by the Association's Committee on Coal and Coke under the direction of the Minerals Standards Committee as a further part of AS 1038. It is not technically equivalent to ISO 602, Coal—Determination of Mineral Matter.

The organic material of coal is associated with minerals which have been deposited or formed within the coal. These minerals provide the ash obtained on combustion of the coal; however the mineral matter to ash ratios are quite variable between and within seams. Accordingly, the mineral matter content of coal must be known if coal analyses are to be converted to the dry, mineral-matter-free basis (i.e. organic coal basis) necessary for classification.

Methods of calculating the mineral matter of coal from the percentage ash and other analyses have been specified for British coals in BS 1016, Methods for the Analysis and Testing of Coal and Coke, Part 16—Reporting of Results. These formulas are derived from the mineral compositions and associations found in British coals and are not always appropriate for Australian coals.[1,2]

ISO 602 specifies a method for the direct determination of mineral matter using an acid demineralization procedure.

The stated repeatability of the ISO method is relatively poor and figures for reproducibility are not given. Inherent in this determination is the necessity to make corrections for ash, insoluble pyrite and absorbed hydrochloric acid; furthermore, the method may yield anomalous results on certain low rank bituminous and lower rank coals.[3]

The direct determination of mineral matter and water of hydration in Australian coals has been investigated by Brown *et al* who described the following procedures for the determination of mineral matter:

(a) Low temperature (370°C) oxidation of the coal substance.

(b) Acid demineralization similar to that specified in ISO 602.

(c) A combined procedure in which a hydrochloric acid extraction of carbonate, sulphate and phosphate minerals is followed by oxidation at 370°C.

Other methods for the determination of mineral matter are as follows:

(i) Low temperature (370°C) oxidation, with direct gravimetric determination of combined water.[4]

(ii) Low temperature (approximately 150°C) oxidation of the coal substance with a radio-frequency excited oxygen plasma.[3]

An outstanding advantage of the low temperature oxidation procedures over other procedures is that a direct determination of the water of hydration of the coal can be carried out on the separated mineral matter. The mineral matter residues are also suitable for X-ray diffraction examination or chemical analysis.

The committee organized an inter-laboratory test program to obtain information on the repeatability and reproducibility of the methods. The following laboratories participated in the test program to provide the data given in Tables B1 and B2:

Australian Coal Industry Research Laboratories Ltd
Australian Iron and Steel, Port Kembla
Broken Hill Proprietary Company Ltd, Central Research Laboratories
Joint Coal Board
SGS Australia Pty Limited
State Electricity Commission of Victoria, Herman Research Laboratory

1. H.R. Brown, R.A. Durie, and H.N.S. Schafer, *Fuel*, 1959, 38, 295 and *Fuel*, 1960, 39, 50.
2. N.A. Brown, T.G. Callcott, and N.Y. Kirov, 'Notes on Mineral Matter in Australian Coals', Paper A6, Symposium on Australian Fuels and Their Utilization, Newcastle, N.S.W., August 1959. Published by the Australian Membership of the Institute of Fuel.
3. F.W. Frazer and C.B. Belcher, *Fuel*, 1973, 52, 41.
4. N.A. Brown, C.B. Belcher, and T.G. Callcott, *J. Inst. Fuel*, 1965, 38, 198.

STANDARDS ASSOCIATION OF AUSTRALIA

Australian Standard

METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

PART 22—DIRECT DETERMINATION OF MINERAL MATTER AND WATER OF HYDRATION OF MINERALS IN HARD COAL

1 SCOPE. This standard sets out two methods for the determination of the amounts of mineral matter and water of hydration of minerals in hard coal, viz:

(a) Air oxidation (370°C).

(b) Radio-frequency oxygen plasma oxidation.

Both methods are known as low temperature oxidation techniques. The radio-frequency method yields a residue which contains essentially the original unchanged mineral matter of a coal. Predictable alteration of some minerals occurs in the air oxidation process, and appropriate corrections are made.

NOTES:

1. See Appendix A for the applicability of this method to low rank coals.
2. It is advisable to use the radio-frequency (RF) oxygen plasma oxidation method for coals of high ash (and for coals with high gypsum levels), due to significant irreversible loss of water of hydration in the air oxidation method.

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 6—Ultimate Analysis of Coal Part 11—Forms of Sulphur in Coal Part 16—Reporting of Results
AS 1152	Test Sieves
AS 1676	Methods for the Sampling of Hard Coal
AS 2243	Safety in Laboratories Part 5—Non-ionizing Radiations

3 DEFINITIONS. For the purpose of this standard, the following definitions apply:

3.1 Hard coal—mature coal having a gross specific energy of, or in excess of, 27 MJ/kg on a dry ash-free (daf) basis, according to the classification system for Australian hard coals.

3.2 Coal substance—a complex organic material consisting of essentially all the organic carbon, hydrogen, nitrogen, oxygen, and sulphur of coal but excluding moisture, mineral matter and, in the case of lower rank coals, inorganic matter.

The calculation of the composition of this organic matter on a mineral-matter-free basis requires the knowledge of the amount and composition of the mineral matter present in the sample.

3.3 Mineral matter in coal (*MM*)—the minerals and other inorganic material in, and associated with coal. The mineral component includes water of hydration and the carbon dioxide of mineral carbonates.

3.4 Mineral matter in low rank coal—the mineral matter in low rank coal can be subdivided as follows:

(a) Minerals—the discrete inorganic material in coal.

(b) Inorganic matter—elements chemically bound to coal organic matter and water soluble salts in coal.

3.5 Ash (*A*)—the inorganic residue after the incineration of coal or coke to constant mass under standard conditions. In general it differs in mass and composition from the original mineral matter.

3.6 Low temperature oxidation residue (*R*)—the product of the low temperature oxidation process after this product has been equilibrated with the laboratory atmosphere. The nature of the product will depend upon the oxidation technique, but is more closely related to the original mineral matter than is ash.

3.7 Moisture (*M*)—water in coal quantitatively determined by standard methods.

3.7.1 ***Moisture in air-dried coal (M_{ad})***—the moisture retained by the coal sample after it has attained equilibrium with the air to which it is exposed.