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Australian Standard 1038.11—1982

METHODS FOR THE ANALYSIS AND TESTING OF COAL AND COKE

Part 11—FORMS OF SULPHUR IN COAL

PUBLISHED BY THE STANDARDS ASSOCIATION OF AUSTRALIA
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THE FOLLOWING INDUSTRIAL, SCIENTIFIC AND GOVERNMENTAL ORGANIZATIONS and departments were officially represented on the committee entrusted with the preparation of this standard:

Australian Coal Association

Australian Institute of Energy

Australasian Institute of Mining and Metallurgy

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 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

Universities

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PREFACE

This edition of this standard was prepared by the Association's Committee on Coal and Coke, under the direction of the Minerals Standards Committee, to supersede AS 1038, Part 11-1973. The new edition was prepared to correct some minor technical details and to update the references to other standards.

For many purposes a knowledge of the total sulphur in a coal is sufficient but for certain work it is also necessary to know how the sulphur is distributed between the coal substance and the mineral matter. In particular, knowledge of the forms of sulphur may be required in connection with coal classification and coal cleaning.

Sulphate sulphur is determined directly as sulphate in a hydrochloric acid extract of the sample. Pyritic sulphur is calculated from the determination of iron soluble in nitric acid following removal of non-pyritic iron by hydrochloric acid. The organic sulphur is obtained by subtracting the sum of pyritic sulphur and the sulphate sulphur from the total sulphur, determined by one of the methods given in AS 1038, Part 6.

This standard allows the use of a colorimetric method for the determination of iron. An atomic absorption method has been added for the determination of pyritic sulphur and the 'indirect' method has been deleted in favour of the 'direct' method, which has now been found suitable for all coals.

This standard requires reference to the following standards:

AS 1038	Methods for the Analysis and Testing of Coal and Coke Part 1—Total Moisture in Hard Coal Part 3—Proximate Analysis of Hard Coal Part 6—Ultimate Analysis of Coal Part 16—Reporting of Results
AS 1152	Test Sieves
AS 1676	Methods for the Sampling of Hard Coal
AS 2134	Code of Practice for the Chemical Analysis of Materials by Flame Atomic Absorption Spectroscopy
AS 2164	One-mark Volumetric Flasks
AS 2165	Burettes and Bulb Burettes
AS 2166	One-mark Pipettes
AS 2167	Straight Pipettes
AS 2418	Glossary of Terms Relating to Solid Mineral Fuels
AS CK19	Code of Recommended Practice for the Chemical Analysis of Materials by Ultraviolet/Visible Spectrophotometry.

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STANDARDS ASSOCIATION OF AUSTRALIA

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

PART 11—FORMS OF SULPHUR IN COAL

1 SCOPE. This standard sets out methods for determining the amount of sulphate sulphur, pyritic sulphur and organic sulphur in coal, brown coal briquettes and brown coal char.

NOTE: Non-pyritic sulphide sulphur may occasionally occur in some coals, predominantly associated with sphalerite. An indication of a significant concentration of non-pyritic sulphides is available through the release of H_2S gas (objectionable odour) when hydrochloric acid is added. A nitric acid extraction of the coal sample, in the manner of the pyritic sulphur determination, yields a result which is the sum of sulphate, pyritic and sulphide sulphur; the extracted residue following the sulphate sulphur determination, processed by the pyritic sulphur method, will yield pyritic sulphur only.

2 DEFINITIONS. For the purposes of this standard, the following definitions, and the definitions given in AS 2418, apply:

Forms of sulphur—the pyritic sulphur, the sulphate sulphur and the organic sulphur in the coal.

Pyritic sulphur—that part of the sulphur which is present in the coal in the form of pyrite or marcasite.

Sulphate sulphur—that part of the sulphur which is present in the coal as sulphate.

Organic sulphur—the difference between the total sulphur and the sum of the pyritic sulphur and sulphate sulphur.

3 COAL SAMPLE.

3.1 General. The coal used for the determination of sulphate and pyritic sulphur is the analysis sample of coal ground to pass a 212 μm test sieve complying with AS 1152, taken and prepared in accordance with AS 1676.

3.2 Equilibration of the Sample. The sample received in the laboratory shall be brought into equilibrium with the laboratory atmosphere by exposure 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 analysis. Portions for duplicate determinations shall be weighed out consecutively.

4 DETERMINATION OF SULPHATE SULPHUR.

4.1 Principle. The coal sample is extracted with boiling dilute hydrochloric acid for 30 min to bring the sulphate sulphur into solution. After filtration, the sulphate sulphur is precipitated from the filtrate as barium sulphate and determined gravimetrically.

4.2 Apparatus.

4.2.1 *Condenser.* A 'cold-finger' condenser (e.g. an Arnold bubbler) which will fit loosely into the neck of a 250 mL conical flask.

4.2.2 *Muffle furnace.* An electrically heated muffle furnace capable of maintaining a zone within the range 800 ± 25°C. The ventilation shall be such as to give at least four air changes per minute at 800°C (see AS 1038, Part 3).

4.2.3 *Plate.* A silica plate, 6 mm thick, which is an easy sliding fit in the muffle furnace.

4.2.4 *Volumetric glassware.* Class A volumetric glassware complying with the relevant Australian standards shall be used.

4.3 Reagents.

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

4.3.2 *Solutions.*

4.3.2.1 *Hydrochloric acid* (ρ_{20} 1160 kg/m³ to 1180 kg/m³).

4.3.2.2 *Hydrochloric acid solution* (ρ_{20} 1160 kg/m³ to 1180 kg/m³), diluted 1 + 1.

4.3.2.3 *Ammonia solution* (ρ_{20} 880 kg/m³), diluted 1 + 2.

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

4.3.2.5 *Potassium sulphate solution* (2 g/L). Dissolve 2 g of potassium sulphate in water and dilute to 1 L.

4.3.2.6 *Methyl red indicator solution* (0.2 g/L). Dissolve 0.02 g of methyl red in 60 mL of ethanol (95 percent) and dilute to 100 mL with water.