

Methods for

Analysis and testing of coal and coke —

Part 106: Ultimate analysis of coal and
coke —

Section 106.1 Determination of carbon
and hydrogen content —

Subsection 106.1.2 Liebig method

ICS 75.160.10

Committees responsible for this British Standard

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- British Cement Association
- British Coal Corporation
- British Iron and Steel Producers' Association
- Electricity Association
- GAMBICA (BEAMA Ltd.)
- Power Generation Contractors' Association (PGCA (BEAMA Ltd.))

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National foreword

This British Standard has been prepared by Technical Committee SFI/3 and is identical to ISO 625:1996 *Solid mineral fuels — Determination of carbon and hydrogen — Liebig method*, incorporating Corrigendum 1:1996, published by the International Organization for Standardization (ISO) and in the preparation of which the UK played a full part.

BS 1016-106 will form a revision of Parts 6 to 11 of BS 1016. This method is an alternative to Subsection 106.1.1 *High temperature combustion method*, together with which it supersedes the following:

- in BS 1016-6:1977, clauses 5 and 6 and that part of clause 11 relating to carbon and hydrogen;
- in BS 1016-7:1977, clauses 5 and 6 and that part of clause 10 relating to carbon and hydrogen.

BS 1016-106 is part of a rationalized and restructured BS 1016. The Parts numbered from 1 to 21 are gradually being withdrawn and replaced by Parts in the new series. The full list of Parts in the new series, together with corresponding numbering of the old series and related International Standards is given in BS 1016 *Analysis and testing of coal and coke* Part 100:1994 *General introduction and methods for reporting results*.

Cross-references.

The Technical Committee has reviewed the provisions of ISO 1015:1992, ISO 5068:1983 and ISO 5069-2:1983 to which normative reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard.

British Standards related to ISO 331:1983, ISO 687:1974, ISO 925:1980, ISO 1170:1977, ISO 1988:1975 and ISO 2309:1980 are BS 1016: *Methods for analysis and testing of coal and coke* Part 104: *Proximate analysis* Section 104.1:1991 *Determination of the moisture content of the general analysis sample of coal*, Section 104.2:1991 *Determination of the moisture content of the general analysis sample of coke*, Part 6:1991 *Ultimate analysis of coal*, Part 100:1994 *General introduction and methods for reporting results*, BS 1017: *Sampling of coal and coke* Part 1:1989 *Methods for sampling of coal* and Part 2:1994 *Methods for sampling of coke*. ISO 1988 and ISO 2309 are being revised and will be published in eight Parts, and it is intended to implement these Parts as identical British Standards superseding BS 1017.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

Introduction

An alternative method to that specified in this International Standard is given in ISO 609:1996, *Solid mineral fuels — Determination of carbon and hydrogen — High temperature combustion method*.

1 Scope

This International Standard specifies a method of determining the total carbon and the total hydrogen in hard coal, brown coal and lignite, and coke, by the Liebig method.

The results include the carbon in the carbonates and the hydrogen combined in the moisture and in the water of constitution of silicates. A determination of moisture is carried out at the same time, and an appropriate correction is applied to the hydrogen value obtained by combustion. A determination of carbon dioxide may also be made and the total carbon value corrected for the presence of mineral carbonates.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 331:1983, *Coal — Determination of moisture in the analysis sample — Direct gravimetric method*.

ISO 687:1974, *Coke — Determination of moisture in the analysis sample*.

ISO 925:1980, *Solid mineral fuels — Determination of carbon dioxide content — Gravimetric method*.

ISO 1015:1992, *Brown coals and lignites — Determination of moisture content — Direct volumetric method*.

ISO 1170:1977, *Coal and coke — Calculation of analyses to different bases*.

ISO 1988:1975, *Hard coal — Sampling*.

ISO 2309:1980, *Coke — Sampling*.

ISO 5068:1983, *Brown coals and lignites — Determination of moisture content — Indirect gravimetric method*.

ISO 5069-2:1983, *Brown coals and lignites — Principles of sampling — Part 2: Sample preparation for determination of moisture content and for general analysis*.

3 Principle

A known mass of coal or coke is burnt in a current of oxygen in a tube impervious to gases, the products of the incomplete combustion being further burnt over copper oxide; all the hydrogen is converted to water and all the carbon to carbon dioxide. These products are absorbed by suitable reagents and determined gravimetrically. Oxides of sulfur are retained by lead chromate, chlorine by a silver gauze roll and oxides of nitrogen by granular manganese dioxide.

4 Reagents and materials

WARNING — Care should be exercised when handling reagents, many of which are toxic.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Magnesium perchlorate, anhydrous, less than 1,2 mm in size and preferably within the size range 1,2 mm to 0,7 mm.

WARNING — Due regard must be taken of local regulations when disposing of exhausted magnesium perchlorate. Regeneration of magnesium perchlorate must not be attempted, owing to the risk of explosion.

4.2 Sodium hydroxide on an inert base, preferably of a coarse grading, for example 3,0 mm to 1,5 mm, but not finer than the grading 1,2 mm to 0,7 mm, and preferably of the self-indicating type.

4.3 Manganese dioxide, granular, 1,2 mm to 0,7 mm.

Manganese dioxide in the granular form and the size required can be prepared as follows.

Dissolve manganese sulfate in water and boil the solution. Make the solution alkaline with ammonium hydroxide and add solid ammonium persulfate, in small portions, to the boiling solution until precipitation is complete. Filter through a hardened fast-filter paper, wash with water by decantation, then with dilute sulfuric acid and finally with water until acid-free. Transfer the moist precipitate to a mortar and place in an oven until most of the water has evaporated, but the powder is still damp. Press the mass into a cake with a pestle, using firm pressure. Complete the drying, break up the cake cautiously and sieve to separate the 1,2 mm to 0,7 mm size.

4.4 Copper gauze, of mesh approximately 1 mm and 10 mm wide.

4.5 Copper oxide, wire form, chopped to particles approximately 3 mm long with a diameter of approximately 0,2 mm.