

Petrographic analysis of bituminous coal and anthracite —

Part 5: Method of determining microscopically the reflectance of vitrinite

Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee SFI/4, Coal petrography, upon which the following bodies were represented:

- British Coal Corporation
- British Gas plc
- British Steel Industry
- Combustion Engineering Association
- International Committee for Coal and Organic Petrology
- Power Generation Contractors' Association (PGCA) (BEAMA Ltd.)

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on 15 April 1995

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First published August 1981
Second edition April 1995

The following BSI references relate to the work on this standard:
Committee reference SFI/4
Draft for comment 90/53086 DC

ISBN 0 580 23873 3

Amendments issued since publication

Amd. No.	Date	Comments

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National foreword

This British Standard has been prepared under the direction of Technical Committee SFI/4, and is identical with ISO 7404-5:1994 *Methods for the petrographic analysis of bituminous coal and anthracite — Part 5: Method of determining microscopically the reflectance of vitrinite* published by the International Organization for Standardization (ISO), in the preparation of which the United Kingdom played a full part.

This British Standard is a revision of BS 6127-5:1981, which is withdrawn.

Cross-references

International Standard	Corresponding British Standard
	BS 6127 <i>Petrographic analysis of bituminous coal and anthracite</i>
ISO 7401-1:1994	Part 1:1995 <i>Glossary of terms relating to the petrographic analysis of bituminous coal and anthracite</i> (Identical)
ISO 7404-2:1985	Part 2:1982 <i>Method of preparing coal samples for petrographic analysis</i> (Technically equivalent)

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 12, 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

Petrographic analyses have been recognized internationally as important in the context of the genesis, vertical and lateral variation, continuity, metamorphism and usage of coal. The International Committee for Coal and Organic Petrology (ICCP) has made recommendations concerning nomenclature and analytical methods and has published an extensive handbook describing in detail the characteristics of a wide range of coals. The text of this part of ISO 7404 agrees substantially with the text of the handbook and incorporates many useful comments made by members of the ICCP and by member bodies of ISO/TC 27, *Solid mineral fuels*.

Petrographic analyses of a single coal provide information about the rank, the maceral and microlithotype compositions and the distribution of minerals in the coal. The reflectance of vitrinite is a useful measure of coal rank and the distribution of the reflectance of vitrinite in a coal blend, together with a maceral group analysis, can provide information about some important chemical and technological properties of the blend.

ISO 7404 is concerned with the methods of petrographic analysis currently employed in characterizing bituminous coal and anthracite in the context of their technological use. It establishes a system for petrographic analysis and comprises five parts, as follows:

- *Part 1: Vocabulary;*
- *Part 2: Method of preparing coal samples;*
- *Part 3: Method of determining maceral group composition;*
- *Part 4: Method of determining microlithotype, carbominerite and minerite composition;*
- *Part 5: Method of determining microscopically the reflectance of vitrinite.*

For information on the nomenclature and analysis of brown coals and lignites, reference should be made to the *International Handbook of Coal Petrography*[1] published by the ICCP.

The properties of a given coal are determined by the proportions and associations of the macerals and minerals present (see ISO 7404-3[2]) and by the rank of the coal. One property that normally can be used as an indicator of rank, independent of the petrographic composition, is the reflectance of the vitrinite of the coal which increases progressively with increasing degree of coalification.

The reflectances of the submacerals of vitrinite differ even in a single coal seam and therefore the value of the reflectance obtained depends on the choice of the submacerals used for measurement. Reflectance measurements are made on one or more of the submacerals of vitrinite, and in reporting the results it is necessary to specify on which submacerals the measurements were made and in what proportion. Consequently, a vital step in the measurement of vitrinite reflectance is the identification of vitrinite and its various submacerals. For this purpose, ISO 7404-1 and the ICCP handbook should be consulted.

The reflectance value obtained also depends on whether maximum or random reflectance measurements are made so that the type of measurement has to be specified.

All of these analysis procedures are applicable to single coal seams or to blends providing that enough measurements are made in compliance with an unbiased sampling procedure on a representative sample.

1 Scope

This part of ISO 7404 specifies the method for determining microscopically the maximum and random reflectance in oil of polished surfaces of the vitrinite component of coals. The method is applicable to either coals from single seams or coal blends covering the whole range of bituminous coal and anthracite. By itself, this method is unsuitable for determining the proportion of components in a blend, particularly when the components have dissimilar measurable vitrinite contents.

The method necessitates the identification of vitrinite by the operator. Reflectance measurements on vitrinite obtained by interpreting the results of a computerized automated system of microscopic analysis are outside the scope of this part of ISO 7404.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 7404. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 7404 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 7404-1:1994, *Methods for the petrographic analysis of bituminous coal and anthracite* — *Part 1: Vocabulary*.

ISO 7404-2:1985, *Methods for the petrographic analysis of bituminous coal and anthracite — Part 2: Method of preparing coal samples.*

3 Definitions

For the purposes of this part of ISO 7404, the definitions given in ISO 7404-1 apply.

4 Principle

Light, with a wavelength of 546 nm, reflected at near normal incidence from a specified area of well-polished vitrinite, measured under oil immersion using a photomultiplier (or similar device), is compared with light reflected under identical conditions from a number of standards of known reflectance. Because different vitrinite particles within a single coal seam invariably differ slightly from one another in optical properties, enough readings on different particles are taken to ensure that the results are representative.

5 Materials

5.1 Immersion oil, of a non-drying, non-corrosive type, with a refractive index of $1,518\ 0 \pm 0,000\ 4$ at 23 °C and a wavelength of 546 nm and with a temperature coefficient $-\frac{dn}{dt}$ of less

than $0,000\ 5\ K^{-1}$.

NOTE 1 Oil from a bottle which was first opened more than a year previously should not be used unless the refractive index has been checked.

NOTE 2 The oil should not contain polychlorinated biphenyls or other toxic components.

5.2 Calibration standards

5.2.1 Reflectance standards, consisting of polished surfaces of materials that

- are isotropic (or basal sections of uniaxial minerals);
- are durable and resistant to corrosion;
- have constant reflectance over a long period;
- are free from inclusions, grain boundaries, discontinuities, internal flaws and fractures;
- have negligibly low absorptance.

To avoid significant amounts of light other than that reflected from the top surface returning to the objective, the body of the standard shall be either deeper than 5 mm or wedge-shaped. The lower surface shall be matt if it makes an angle of less than 10° with the upper polished surface.

The sides shall be shielded from external light.

The reflectance of the standards shall be in the region of the reflectance of the coal to be measured. Use at least three such standards with well-spaced reflectances (see note 3).

If a coal with a reflectance greater than 2,0 % is to be measured, use one or more additional standards with reflectance greater than 2,0 %.

Table 1 gives mean values of refractive index and of reflectance for reflectance standards in common use. Determine exactly the reflectance of any optical glass standard by using a comparable standard or by means of a determination of the refractive index.

Where the refractive index, n , and (if significant) the absorptance, α , of the standard material are known for a wavelength of 546 nm, calculate the reflectance, R , as a percentage using the equation

$$R = \frac{(n - 1,518)^2 + n^2 \alpha^2}{(n + 1,518)^2 + n^2 \alpha^2} \times 100$$

Table 1 — Reflectance standards in common use

Designation	Refractive index	Reflectance in immersion oil of 1,518 at 546 nm (see 5.1) %
Optical glasses	1,70 to 1,97	0,32 to 1,66
Spinel	1,73	0,42
Leucosapphire	1,77	0,59
Yttrium aluminium garnet (YAG)	1,84	0,92
Gadolinium gallium garnet (3G)	1,98	1,73
Diamond	2,42	5,28
Silicon carbide	2,66	7,50

Where the refractive index is not known, or where it is suspected that the surface properties may not exactly match the nominal bulk properties, determine the reflectance by careful comparison with a standard of known reflectance (see note 4).

NOTE 3 For measuring a coal reflectance of about 1,0 %, standards with reflectances of approximately 0,6 %, 1,0 % and 1,6 % should be used.

NOTE 4 Standards need careful cleaning to avoid scratching the polished surface. Tarnishing may also occur with some standard materials, particularly glasses. When the surface becomes scratched, or when comparison with the other standards shows that the reflectance value has changed, polishing is necessary.

5.2.2 Zero standard

NOTE 5 A suitable non-reflecting standard consists of a coal or opaque resin block with a hole about 5 mm in diameter and 5 mm deep, drilled in its upper surface and filled with immersion oil. Alternatively, optical glasses of refractive index lower than that of the immersion oil may be used.