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Methods for the petrographic analysis of coals —

Part 5: Method of determining microscopically the reflectance of vitrinite

Méthodes d'analyse pétrographique des charbons —

Partie 5: Détermination au microscope du pouvoir réflecteur de la vitrinite

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 7404-5 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This third edition cancels and replaces the second edition (ISO 7404-5:1994), which has been technically revised.

ISO 7404 consists of the following parts, under the general title *Methods for the petrographic analysis of coals*:

— *Part 1: Vocabulary*[1)]

— *Part 2: Methods of preparing coal samples*

— *Part 3: Method of determining maceral group composition*

— *Part 4: Method of determining microlithotype, carbominerite and minerite composition*[1)]

— *Part 5: Method of determining microscopically the reflectance of vitrinite*

1) Parts 1 and 4 of this International Standard will be available under the original title, *Methods for the petrographic analysis of bituminous coal and anthracite*, until the revisions of these documents have reached the stage at which they are publicly available.

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 a comprehensive handbook that is continuously updated. 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 single seam coals 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 can provide information on the distribution of coals of different rank in a coal blend. Together with a maceral group analysis, it provides information about some important chemical and technological properties of the coal and the coal blend. The reflectance of vitrinite has various other applications, such as the characterization of bulk samples and cargoes. For coal blends, the measurement of the vitrinite reflectance profile can permit the identification of the component coals and permit the estimation of the relative abundance of the component coals within the blend.

ISO 7404 (all parts) is concerned with the methods of petrographic analysis currently employed in characterizing coal in the context of their technological use.

The method of determining the reflectance of vitrinite is applicable for low-, medium- and high-rank coals [7].

The properties of a given coal are determined by the proportions and associations of the macerals and minerals present (see ISO 7404-3 [3]) and by the rank of the coal and thus its type, grade and rank. The reflectance of the vitrinite in the coal can be used as an indicator of rank, independent of the petrographic composition. Vitrinite reflectance increases progressively with rank.

The reflectances of the macerals of the vitrinite group can vary significantly in a single coal seam and therefore the value of the reflectance obtained depends also on the choice of the macerals used for measurement. Reflectance measurements are made on one or more of the macerals of vitrinite and, in reporting the results, it is necessary to specify the macerals on which the measurement were made and the proportions of the overall value contributed by each of the macerals measured. Consequently, a vital step in the measurement of vitrinite reflectance is the identification of vitrinite and its various macerals or maceral varieties. For this purpose, reference can be made to ISO 7404-1 and the ICCP [1] handbook.

For rank determination of single-seam coals, normally the reflectance of collotelinite (eu-ulminite [6] in lignites, the equivalent of low-rank B and C [6]) is determined. In cases where collotelinite (or in low-rank coals, eu-ulminite) is not present in sufficient amounts, reflectance analysis on other vitrinite macerals is performed. Reflectance analysis on various vitrinite macerals can also be applied for technological purposes and to coal blends; see 8.3.1. The reflectance value obtained also depends on whether maximum or random reflectance measurements are made, so it is necessary to specify the type of measurement. All of these analysis procedures are applicable to single-coal seams or to blends providing that adequate (see 8.3.1) reflectance measurements are made in compliance with an unbiased sampling procedure on a representative sample.

An accreditation programme for vitrinite reflectance analysis of single-seam coals (SCAP) is run regularly by the ICCP for accrediting petrologists.

NOTE As this edition of ISO 7404 covers coals of all rank, the term “vitrinite” as used in this part of ISO 7404 includes vitrinite as well as huminite. Reference can be made to ISO 7404-1 for details. The equivalent to collotelinite in lignites is ulminite B. Reflectance measurement on lignites is performed on huminite.

Methods for the petrographic analysis of coals —

Part 5: Method of determining microscopically the reflectance of vitrinite

1 Scope

This part of ISO 7404 specifies the methods for determining the reflectance of vitrinite of coals microscopically on the polished surfaces, immersed in oil. The methods are applicable to coals from single seams or coal blends covering the whole range of low-, medium- and high-rank coal.

Vitrinite reflectance measurements can be used to characterize the components within blends. Measures can be taken to correct for the vitrinite percentage within each of the components of the blend or to determine the proportion of components in a blend, particularly when the components have dissimilar vitrinite contents. This method necessitates the identification of vitrinite by the analyst.

Reflectance measurements on vitrinite obtained by interpreting the results from an automated system, are outside the scope of this part of ISO 7404.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7404-1, *Methods for the petrographic analysis of bituminous coal and anthracite — Part 1: Vocabulary*

ISO 7404-2, *Methods for the petrographic analysis of coals — Part 2: Methods of preparing coal samples*

3 Definitions

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

4 Principle

The intensity of light reflected at near-normal incidence from a specified area of well polished vitrinite under oil immersion, measured at 546 nm using a photomultiplier (or similar device), is compared with the intensity of light reflected under identical conditions from a number of standards of known reflectance. Different vitrinite particles within a single-coal seam invariably differ slightly from one another in optical properties. Therefore, an adequate number (see 8.3.1) of readings on different particles is taken to ensure that the results are representative of the coal or coal blend.

5 Reagents and materials

5.1 Immersion oil, non-drying, non-corrosive type, with a refractive index of 1,518 0 ± 0,000 4 at 23 °C and a wavelength of 546 nm.

Oil from a bottle opened more than one year ago should not be used.

In attempts to produce oils that are chemically and physically stable, toxic compounds such as polychlorinated bi-phenyls were used in some older products. Some more recent oils have been associated with allergies. The composition of the oil should, therefore, be checked to ensure that no toxic or other undesirable properties are associated with it.

5.2 Calibration standards

5.2.1 Reflectance standards, consisting of polished surfaces of materials that

a) are isotropic (or basal sections of uniaxial minerals);

b) are durable and resistant to corrosion;

c) have a reflectance that is stable over a long period;

d) are free from inclusions, grain boundaries, discontinuities, internal flaws and fractures;

e) 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 thicker 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 of an order similar to that of the coal being measured. Use at least two such standards with well spaced reflectances. If a coal with a reflectance greater than 2,0 % is being measured, use one or more additional standards with reflectance greater than 2,0 %.

Table 1 gives approximate mean values for reflectance standards or reflectance ranges as found between different standards, as calibrated against an ICCP Master Standard (see Note 2) in common use.

NOTE 1 For measuring a vitrinite reflectance of about 1,0 %, a standard with reflectance below and a standard with reflectance above 1% are used.

NOTE 2 An ICCP round robin exercise on reflectance standards demonstrated that variations between standards of nominally identical reflectances can be significant [9]; since then, the ICCP has offered the calibration of standards against ICCP master standards.

NOTE 3 It is necessary that standards be carefully cleaned to avoid scratching the polished surface. If solvents are used to remove old oil, it is necessary to take care that the evaporation of the solvent does not leave a residue on the surface of the standard. Tarnishing can 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 Calculation of reflectance standards

Some sources [4] recommend calculating the reflectance, R, of a standard as given in Equation (1):

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

where

n is the known refractive index of the standard material at a wavelength of 546 nm;

α is the known absorptance of the standard material at a wavelength of 546 nm.

NOTE The absorptance, α, is included only if it is significant.

However, the refractive index of the border phase is different from that of the interior of the standard. Consequently, solid reflectance reference materials should always be individually calibrated against a standard of known reflectance; see 5.2.1, Note 2.

Table 1 — Reflectance standards in common use

Designation	Reflectance %
Optical glasses	0,32 to 1,70
Spinel	≈ 0,42
Leucosaphire	≈ 0,59
Yttrium aluminium garnet (YAG)	0,895 to 0,916
Gadolinium gallium garnet (GGG)	1,60 to 1,80
Diamond	≈ 5,3
Silicon carbide	≈ 7,80
See also 5.2.1, Notes 1 to 3.	

5.2.3 Zero standard

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.

6 Apparatus

6.1 Reflected light microscope, with photometer (or similar device), containing the following elements (key item numbers refer to Figure 1, which shows the optical parts of a typical reflectance-measuring microscope):

NOTE 1 The component parts might not always be in the same sequence as shown in Figure 1.

a) light source (key item 1), with a stable output; a quartz halogen lamp with a rating of 100 W is recommended;

b) polarizer (key item 5), either a sheet or prism polarizer (used if maximum reflectance is determined);

c) light-controlling apertures, consisting of two variable diaphragms, one of which is focused on the back focal plane of the objective (illuminator aperture, key item 3) and the other on the surface of the specimen (field stop; key item 6); it shall be possible to centre both diaphragms on the optical axis of the microscope system;

d) vertical illuminator (key item 8), Berek prism, simple coated glass plate or Smith illuminator (a combined mirror and glass plate);

NOTE 2 Typical light paths are shown in Figure 2.

e) objective (key item 9), strain-free, designed for use with polarized light (for maximum reflectance analysis).

Magnifications higher than those achieved by the commonly used 32x to 50x objectives require numerical apertures that decrease the depth of focus to an extent that is undesirable and should, therefore, be avoided where possible;

f) eyepieces (key item 12), one or two viewing eyepieces (oculars), one of which is fitted with crosshairs that can be scaled;

NOTE 3 An additional ocular (key item 13) can be necessary in the light path leading to the photomultiplier.

g) microscope tube, with the following features:

— measuring aperture (key item 14), which restricts the light reaching the photomultiplier to that reflected from an area of the specimen (key item 10) less than 80 μm^2 and that can be aligned with the crosshairs in the viewing eyepiece (key item 12),

— means of optically isolating the viewing eyepieces from the light path to the photomultiplier if the eyepieces permit the entry of extraneous light during measurement,

— adequate blackening of the inside of the tube to absorb stray light;

NOTE 4 Subject to the above precautions, part of the light beam can be diverted to the eyepieces or to a television camera for continuous observation during reflectance measurement.

h) filter (key item 15), with a peak transmittance in the range of 546 nm ± 5 nm and a half-peak transmittance band of less than 30 nm.

The filter should be inserted into the light path immediately before the photomultiplier;

i) photomultiplier tube (key item 16), fitted in housing attached to the microscope, permitting the light passing through the measuring aperture and filter to fall onto the photomultiplier window.

The photomultiplier tube should be of a type recommended for low-light-level applications, and shall have adequate sensitivity at 546 nm with a low output of current when the front element of the photomultiplier is not illuminated (low dark-field current). It shall have a linear response over the range of the measurement and the output shall be stable over the duration of the analysis;

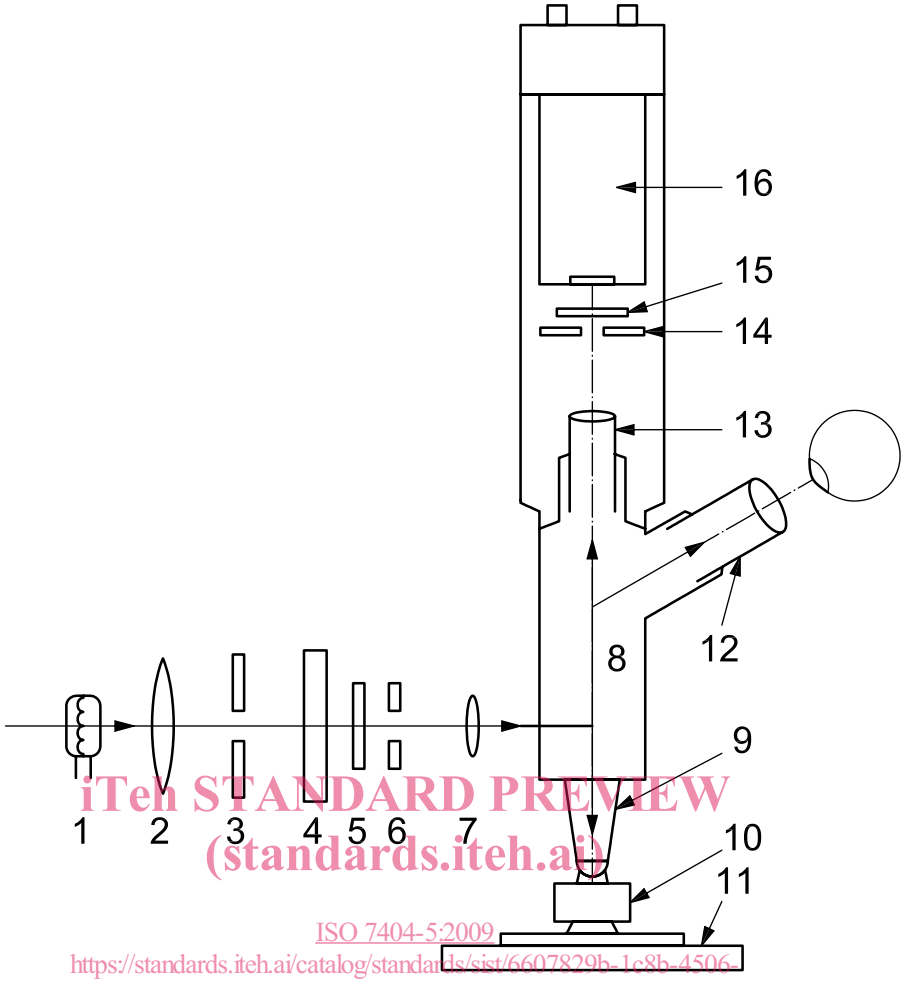
NOTE 5 Lately, semiconductor photodiodes and digital cameras of high sensitivity have been increasingly replacing photomultiplier tubes. The same requirements for precision, linearity and stability as for photomultipliers apply.

j) microscope stage (key item 11), fitted with a mechanical stage capable of advancing the specimen in the X and Y directions. For maximum reflectance, the stage shall be capable of being rotated through 360° perpendicular to the optical axis.

6.2 Power supply unit, for the light source, stabilized d.c., for which the following characteristics have been found to be satisfactory:

a) output variation of less than 0,02 % for a supply variation of 10 %;

b) ripple content at full load of less than 0,07 % peak to peak.



Key

1	lamp	9	objective
2	collector lens	10	specimen
3	illuminator aperture diaphragm	11	stage
4	heat filter	12	viewing eye-piece
5	polarizer	13	third eye-piece
6	field stop	14	measuring aperture
7	field-stop focusing lens	15	546 nm peak transmittance filter
8	vertical illuminator	16	photomultiplier tube

NOTE This set up is commonly known as Köhler Illumination [2].

Figure 1 — Optical parts of a typical reflectance measuring microscope