INTERNATIONAL STANDARD

ISO 7404-5

Second edition
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Methods for the petrographic analysis of bituminous coal and anthracite —

Part 5:

Method of determining microscopically the reflectance of vitrinite

Méthodes d'analyse pétrographique des charbons bitumineux et de l'anthracite —

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

Reference number
ISO 7404-5:1994(E)

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.

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.

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

This second edition cancels and replaces the first edition (ISO 7404-5:1984), which has been technically revised.

ISO 7404 consists of the following parts, under the general title *Methods for the petrographic analysis of bituminous coal and anthracite*:

— *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*

Annex A of this part of ISO 7404 is for information only.

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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.

Methods for the petrographic analysis of bituminous coal and anthracite —

Part 5:
Method of determining microscopically the reflectance of vitrinite

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 ± 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}.

NOTES

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.

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

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

b) are durable and resistant to corrosion;

c) have constant reflectance 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 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).

NOTES

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.

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.

6 Apparatus

6.1 Binocular reflected light microscope with photometer.

NOTE 6 The reference letters refer to figure 1.

6.1.1 Light source (A).

Any light source with a stable output may be used; a quartz halogen lamp with a rating of 100 W is recommended.

6.1.2 Polarizer (E) (optional): A sheet or prism polarizer.

6.1.3 Light-controlling apertures, consisting of two variable diaphragms, one of which is focused on the back focal plane of the objective [illuminator aperture (C)] and the other on the surface of the specimen [field stop (F)]. It shall be possible to centre both diaphragms on the optical axis of the microscope system. The optical parts of a typical reflectance measuring microscope are shown in figure 1.

NOTE 7 The component parts may not always be in the same sequence.

6.1.4 Vertical illuminator, Berek prism, simple coated glass plate or Smith illuminator (a combined mirror and glass plate). Typical light paths are shown in figure 2.

6.1.5 Objective (I), Strain-free objective, designed for use with polarized light and an immersion oil of refractive index 1,518, with a magnification of between × 25 and × 60 and a low value for the parasitic reflection (see 8.2.3).

NOTE 8 The diameter of the objective should be as large as possible. A larger objective gives increased light intensity, thereby reducing the signal amplification and hence the electronic noise; it also makes it possible to work with a smaller measuring aperture.

6.1.6 Eyepieces (L), Two viewing eyepieces, one of which is fitted with crosslines which may be scaled, such that the total magnification given by the objective, eyepiece and tube factor (if any) is between × 250 and × 750.

NOTE 9 A third eyepiece (M) may be necessary in the light path leading to the photomultiplier.

6.1.7 Microscope tube, with the following features:

a) measuring aperture (N) which restricts the light reaching the photomultiplier to that reflected from an area of the specimen (J) less than 80 μm^2 and which can be aligned with the crosslines in the viewing eyepiece;

b) a means of optically isolating the viewing eyepieces if they permit the entry of extraneous light during measurement;

c) adequate blackening to absorb stray light.

NOTE 10 Subject to the above precautions, part of the light beam may be diverted to the eyepieces or to a television camera for continuous observation during reflectance measurement.

6.1.8 Filter(s) (O), with a peak transmittance in the range of 546 nm ± 5 nm and a half-peak transmittance band of less than 30 nm.

NOTE 11 The filters should be inserted into the light path immediately before the photomultiplier.

6.1.9 Photomultiplier tube (P), fitted in a housing attached to the microscope, permitting the light passing through the measuring aperture and filter to fall onto the photomultiplier window.

NOTES

12 The photomultiplier tube should be of a type recommended for low light-level applications, and should have adequate sensitivity at 546 nm with low dark current. It should have a linear response over the range of the measurement and the output should be stable over periods of up to 1 h.

13 A 50 mm diameter straight tube with an end window and containing 11 dynodes is frequently used.

6.1.10 Microscope stage (K), fitted with a mechanical stage capable of advancing the specimen by 0,5 mm steps in the X and Y directions and provided with a means for making small adjusting movements. For maximum reflectance, the stage shall be capable of being rotated through 360° perpendicular to the optical axis, and being centred by adjusting either the stage or the objective.

6.2 Stabilized d.c. power supply unit for the light source.

The following characteristics have been found to be satisfactory:

a) a lamp output of between 90 % and 95 % of rated output;

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

c) a ripple content at full load of less than 0,07 % peak to peak;

d) a temperature coefficient of less than 0,05 % K^{-1}.

A Lamp
B Collector lens
C Illuminator aperture
D Heat filter
E Polarizer
F Field stop
G Field-stop focusing lens
H Vertical illuminator
I Objective
J Specimen
K Stage
L Viewing eye-pieces
M Third eye-piece
N Measuring aperture
O 546 nm interference filter
P Photomultiplier tube

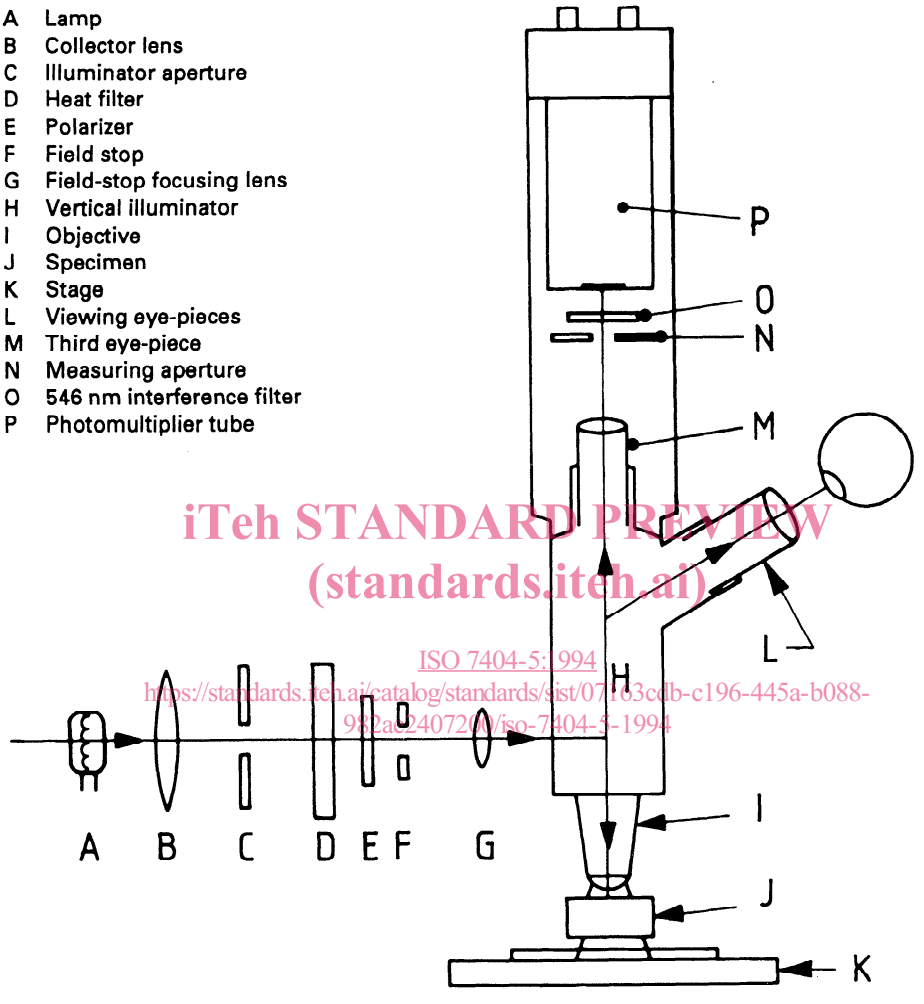
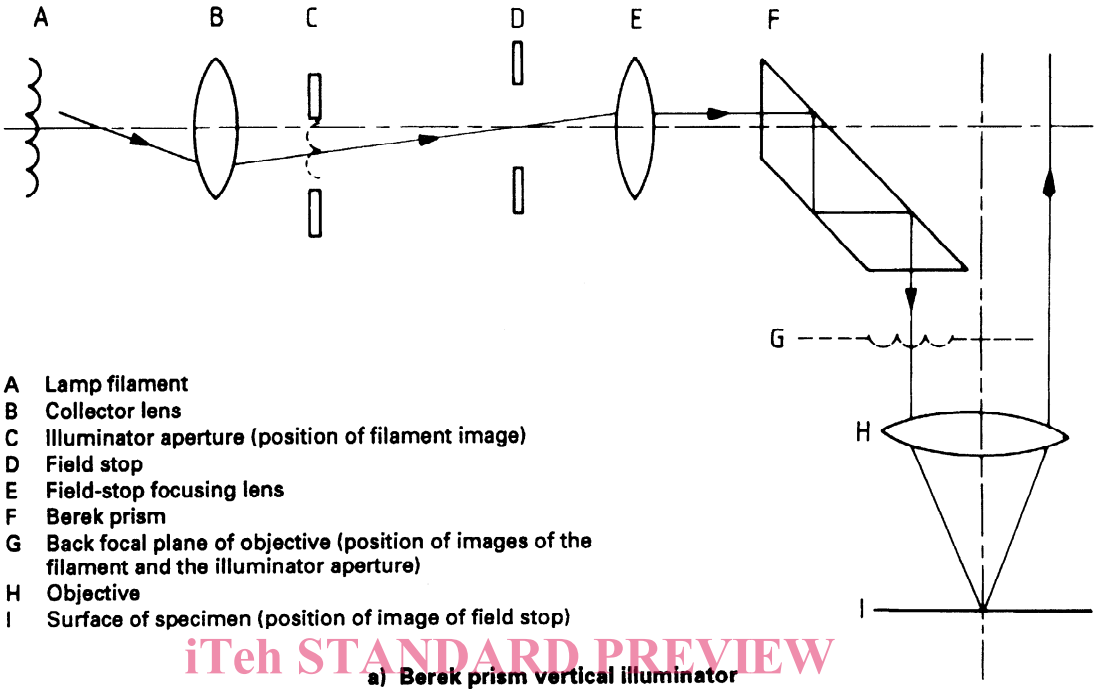
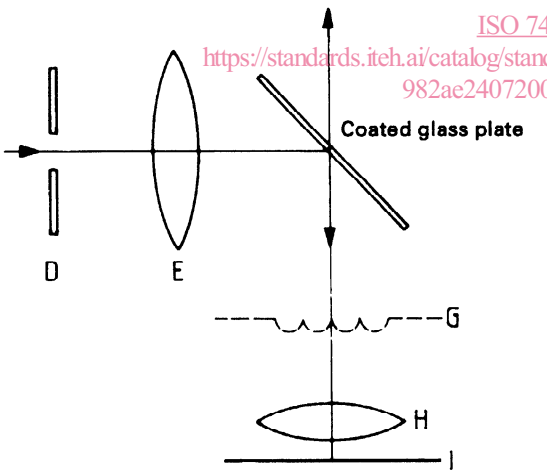


Figure 1 — Optical parts of a typical reflectance measuring microscope

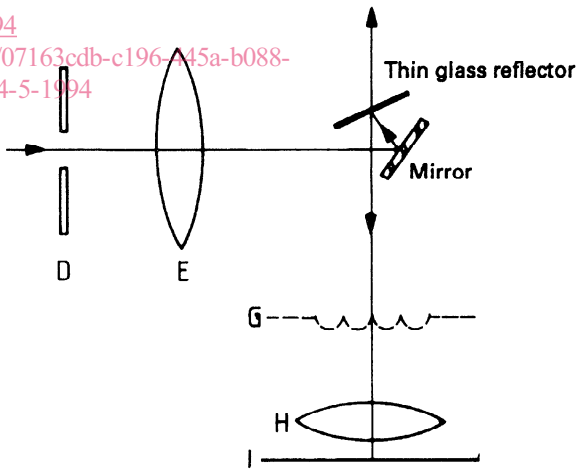


A Lamp filament
B Collector lens
C Illuminator aperture (position of filament image)
D Field stop
E Field-stop focusing lens
F Berek prism
G Back focal plane of objective (position of images of the filament and the illuminator aperture)
H Objective
I Surface of specimen (position of image of field stop)

a) Berek prism vertical illuminator



b) Glass plate illuminator



c) Smith illuminator

Figure 2 — Types of vertical illuminators

6.3 Stabilized d.c. voltage power supply unit for the photomultiplier tube.

The following characteristics have been found to be satisfactory:

a) an output variation of less than 0,05 % for a 10 % variation in supply voltage;

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

c) a temperature coefficient of less than 0,05 % K^{-1};

d) a change in load from zero to full rated load that causes less than 0,1 % variation in output voltage.

NOTE 14 If the mains voltage at peak periods is expected to fall below 90 % of the normal rated value, a suitably rated stabilizing autotransformer should be connected between the mains power point and the two stabilized power supplies (see 6.2 and 6.3).

6.4 Display, comprising one of the following devices:

a) galvanometer with a minimum sensitivity of 10^{-10} A/mm;

b) a chart recorder;

c) a digital voltmeter or digital indicator.

The device used shall be so adjusted that its response time for full-scale deflection is less than 1 s, and shall be capable of resolution of 0,01 % reflectance. A facility for backing-off the small positive voltage due to glare and photomultiplier dark current shall be provided.

NOTES

15 It is an advantage to have a maximum seeking facility incorporated with the digital voltmeter or digital indicator to enable values for the maximum reflectance to be indicated when the specimen on the stage is rotated. Individual reflectance values may also be stored electronically or magnetically for later processing.

16 A low noise variable gain amplifier may be used if necessary to amplify the signal from the photomultiplier before it is passed to the display.

6.5 Sample mounting apparatus, comprising slides, modelling clay and levelling device.

7 Preparation of coal sample

Prepare and polish a particulate block as described in ISO 7404-2.

8 Procedure

8.1 Setting up the apparatus

NOTE 17 In 8.1.3 and 8.1.4 letters in parentheses relate to the key in figure 1.

8.1.1 Starting procedure

Ensure that the room temperature remains within the range 18 °C to 28 °C. Switch on the lamp, power supplies and other electrical parts of the apparatus. Set the power supply to the photomultiplier to within the voltage range recommended by the manufacturer of the particular photomultiplier tube. Allow about 30 min to elapse for stability of the apparatus to be attained prior to making any measurements.

8.1.2 Adjusting the microscope for random or maximum measurements

If random reflectance measurements are to be made, remove the polarizer. If maximum reflectance measurements are to be made, set the polarizer to zero if using a glass plate or Smith illuminator, or to 45° if using a Berek prism. If a sheet polarizer is used, check and replace it if it shows significant discoloration. If the microscope has an analyser, remove this from the light path.

8.1.3 Illumination

Apply immersion oil to the polished surface of a particulate coal block, mounted and levelled on a glass slide, and place the specimen on the stage.

Check that the microscope has been correctly adjusted for Köhler illumination. Adjust the illuminated field by means of the field stop (F) so that its diameter is about one-third of the diameter of the full field. Adjust the illuminator aperture (C) to reduce glare but without reducing the light intensity excessively. Once adjusted, do not alter the size of the apertures.

8.1.4 Alignment

Centre and focus the image of the field stop, centre the objective (I) with respect to the axis of rotation of the stage and adjust the centre of the measuring aperture (N) to be coincident with either the crosslines or a known datum point in the viewing system.

If it is not possible to view the image of the measuring aperture superimposed on the specimen, select a field of view that contains a small bright inclusion, such as a crystal of pyrite, and align this directly under the crossline. Adjust the centring of the measuring aperture (N) until the photomultiplier reading is at its highest value.

8.2 Checking the reliability and calibration of the apparatus

8.2.1 Stability of apparatus

Place the standard with the highest reflectance value under the microscope and focus under oil immersion.

Adjust the amplifier or the voltage to the photomultiplier until the reading on the display has the same numerical value as the reflectance of the standard (for example, 173 mV might correspond to a reflectance of 1,73 %). Ensure that the signal is stable by checking that the reading changes by less than 2 % relative to the first reading within a period of 15 min.

8.2.2 Variation in reading on rotating a reflectance standard on the stage

Place a standard with a reflectance in oil between 1,6 % and 2,0 % on the stage and focus under oil immersion. Slowly rotate the stage and verify that the maximum variation in the reading is less than 2 % relative to the reflectance of the standard being used. If this value is exceeded, check the levelling of the standard, and ensure that the stage is perpendicular to the optical axis and that it rotates in a fixed plane. If these checks do not reduce the variation to less than 2 %, the mechanical stability of the stage and the geometry of the microscope have to be checked by the manufacturer.

8.2.3 Correction for parasitic reflections and photomultiplier dark current

Place the zero standard on the stage and note the reading that represents the sum of the photomultiplier dark current and the parasitic reflections. If the photomultiplier dark current and parasitic reflections exceed 0,04 % reflectance in total, determine their relative proportions by interrupting the light reaching the photomultiplier so that any residual signal is then due to the photomultiplier dark current. Check the setting of the illuminator aperture and change the photomultiplier tube and/or the objective, as appropriate, so that the total signal is below 0,04 % reflectance. When the total signal is below 0,04 % reflectance, adjust the display to zero using the backing-off control (see 6.4). Continue making adjustments using the highest standard as in 8.2.1 and the zero standard in turn until no further adjustment is necessary.

8.2.4 Linearity of the signal from the photomultiplier

Measure the reflectance of the other standards whilst maintaining the constant settings of the voltage supplies and light-controlling apertures in order to check that the measuring system has a linear response in the range to be measured and that the standards match their calculated value. Rotate each standard to ensure that the mean reading attained matches the calculated value. If the reading for any standard differs from its calculated reflectance value by more than 2 % relative to the calculated value, clean the standard and repeat the standardizing process. Repolish any standard still displaying a reflectance differing from its calculated value by more than 2 %.

If the reflectances of the standards still do not give a linear plot, check the linearity of the photomultiplier signal using standards from other sources. If these fail to give a linear plot, check the linearity of the signal by means of several calibrated neutral density filters to reduce the luminous flux by known amounts. If the signal from the photomultiplier is confirmed to be nonlinear, reduce the photomultiplier voltage by 50 V and recheck. If the signal is still nonlinear, check the size of the measuring aperture and if necessary reduce it. If rechecking still shows the signal to be nonlinear, replace the photomultiplier tube and carry out further checks as necessary to achieve linearity of the signal.

8.2.5 Calibration of the apparatus

Having established the reliability of the apparatus, ensure that the display gives the correct readings for the zero standard and the three reflectance standards in the region of the reflectance of the coal to be measured, by carrying out the procedures specified in 8.2.1 and 8.2.4. The reflectance of each standard shown on the display apparatus shall not differ from its calculated value by more than 2 % relative to the calculated value.

8.3 Measurement of the reflectance of vitrinite

8.3.1 General

The procedure for measuring maximum reflectance is specified in 8.3.2 and that for random reflectance is specified in 8.3.3. In these subclauses the term