International Standard 7404/5

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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

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Methods for the petrographic analysis of bituminous coal and anthracite — Part 5 : Method of determining microscopically the reflectance of vitrinite

0 Introduction

0.1 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 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 International Standard 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 mineral matter 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.

This International Standard 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 : Glossary of terms.

Part 2 : Method of preparing coal samples.[1)]

Part 3 : Method of determining maceral group composition.

Part 4 : Method of determining microlithotype composition.[2)]

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* published by the ICCP.[3)]

0.2 The properties of a given coal are determined by the proportions and associations of the macerals and minerals present (see ISO 7404/3), 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 reflectance 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. 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 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 and field of application

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 the characterization of either coals from single seams or coal blends covering the whole rank range of bituminous coal and anthracite.

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.

1) At present at the stage of draft.

2) In preparation.

3) The second edition (1963), together with the supplement issued in 1971 may be obtained from Professor D. G. Murchison, Organic Geochemistry Unit, Department of Geology, University of Newcastle, Newcastle-upon-Tyne, NE1 7RU, United Kingdom. The supplement issued in 1973 may be obtained from Centre national de la recherche scientifique, 15 quai Anatole-France, F-75007 Paris, France.

2 Reference

ISO 7404/1, *Methods for the petrographic analysis of bituminous coal and anthracite — Part 1 : Glossary of terms.*

3 Definitions

For the purpose of this part of ISO 7404, the definitions of 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, a wavelength of 546 nm and with a temperature coefficient $-dn/dt$ 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; and

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 2.)

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 — 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

Table 1 gives mean values of refractive index and of reflectance for reflectance standards in common use. Determine exactly the reflectance of each 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

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

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 3).

NOTES

1 A recommended method of mounting a wedge-shaped standard is shown in figure 1.

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

3 Standards need careful cleaning to avoid scratching the polished surface. Tarnishing may also occur. 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 : A non-reflecting standard consisting 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.

6 Apparatus

6.1 Binocular polarizing reflected light microscope with photometer

NOTE — The reference letters refer to figure 2.

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): 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 optic axis of the microscope system. The optical parts of a typical reflectance measuring microscope are shown in figure 2.

NOTE — 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 3.

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 X 25 and X 60 and a low value for the parasitic reflectance. See 7.2.3.

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 X 250 and X 750.

NOTE — 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 — 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 ± 5 nm and a half-peak transmittance band of less than 30 nm.

NOTE — 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 on to the photomultiplier window.

NOTES

1 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.

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

6.1.10 Microscope stage, capable of being rotated through 360° perpendicular to the optical axis, and which can be centred by adjusting either the stage or the objective. The rotating stage is fitted with a mechanical stage capable of advancing the specimen by 0,5 mm steps in the X and Y directions and is provided with a means for making small adjusting movements of up to 10 µm in either direction.

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}.

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 — 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 0,005 % reflectance. A facility for backing-off the small positive voltage due to glare and photomultiplier dark current shall be provided.

NOTES

1 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.

2 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 Procedure

7.1 Setting up the apparatus

(In 7.1.3 and 7.1.4 letters in parentheses relate to the key in figure 2.)

7.1.1 Starting procedure

Ensure that the room temperature is stable within 23 ± 3 °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.

7.1.2 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.

7.1.3 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.

7.1.4 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.

7.2 Checking the reliability and calibration of the apparatus

7.2.1 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 0,02 % within a period of 15 min.

7.2.2 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.

7.2.3 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 7.2.1 and the zero standard in turn until no further adjustment is necessary.

7.2.4 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 maximum reading attained matches the calculated value. If the reading for any standard differs from its calculated reflectance value by more than 0,02 %, clean the standard and repeat the standardizing process. Repolish any standard still displaying a reflectance differing by more than 0,02 % from its calulated value.

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 non-linear, replace the photomultiplier tube and carry out further checks as necessary to achieve linearity of the signal.

7.2.5 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 7.2.1 to 7.2.4. The reflectance of each standard shown on the display apparatus shall not differ by more than 0,02 % from its calculated value.

7.3 Measurement of the reflectance of vitrinite

7.3.1 General

The procedure for measuring maximum reflectance is specified in 7.3.2 and that for random reflectance is specified in 7.3.3. In these subclauses the term vitrinite refers to one or more of the submacerals in the vitrinite group.

As explained in 0.2, the choice of the submacerals on which the measurements are made affects the results and consequently it is important to decide on which submacerals to measure reflectance and to identify them when reporting the results.

7.3.2 Procedure for the measurement of the maximum reflectance of vitrinite in oil

Ensure that the polarizer is fitted to the microscope as specified in 7.1.2 and that the appropriate checks on the apparatus have been made as specified in 7.1 and 7.2.

Immediately after calibrating the apparatus, place a levelled polished block prepared from the sample to be tested on the mechanical stage to allow measurements to be made starting at one corner of the area to be traversed, apply immersion oil to the surface and bring the specimen into focus.

Move the specimen slightly using the mechanical stage until the crosslines are focused on a suitable area of vitrinite. Ensure that the measuring area contains no cracks, polishing defects, mineral inclusions or relief effects, and is away from maceral boundaries.

Allow the light to pass to the photomultiplier and rotate the stage through 360° at a rotational frequency not exceeding 10 min^{-1}. Record the highest reflectance reading obtained during the stage rotation.

NOTE — During rotation of the block through 360°, ideally two identical maximum readings should be obtained. If the two readings differ significantly, the reason should be sought and the fault corrected. Occasionally air bubbles in the oil pass into the measuring area causing erratic readings. If air bubbles are seen or suspected, ignore the reading and remove the air bubbles by racking down the stage, wiping the front element of the objective lens with a lens tissue, adding a drop of oil to the surface and refocusing the specimen.

Traverse the specimen in the X direction stepwise using a steplength of 0,5 mm and make a measurement when the crosslines fall on a suitable area of vitrinite. To ensure that the measurement is made on a suitable area of vitrinite, the specimen may be moved up to 10 µm using the mechanical stage. At the end of a traverse, move the specimen to the beginning of the next traverse keeping to an interline distance of at least 0,5 mm. Choose the precise interline distance to ensure that the measurements are evenly distributed over the surface of the block. Continue making reflectance measurements using this sampling procedure.

At 15 min intervals (or not more than 50 counts), re-check the calibration of the apparatus using the standard nearest in reflectance to that of the highest reflecting vitrinite in the specimen (see 7.2.5). If the reflectance of the standard differs by more than 0,02 % from its theoretical value, discard the last set of readings and repeat them after recalibrating the apparatus using the full range of standards.

Make reflectance measurements on the vitrinite until the required number has been recorded. If, for 98 % of the measurements, the dispersion of reflectance values is

a) less than 0,35 %, calculate the mean reflectance on the basis of 100 measurements;

b) between 0,35 % and 0,70 %, calculate the mean reflectance on the basis of at least 500 measurements;

c) greater than 0,70 %, calculate the mean reflectance on the basis of at least 1 000 measurements.

7.3.3 Procedure for the measurement of the random reflectance of vitrinite in oil

Adopt the same procedure as specified in 7.3.2, but make the measurements without the polarizer and without rotation of the sample block. Calibrate the apparatus as specified in 7.2.5.

Make reflectance measurements on the vitrinite until the required number has been recorded. If, for 98 % of the measurements, the dispersion of reflectance values is

a) less than 0,40 %, calculate the mean reflectance on the basis of 100 measurements;

b) between 0,40 % and 0,80 %, calculate the mean reflectance on the basis of at least 500 measurements;

c) greater than 0,80 %, calculate the mean reflectance on the basis of at least 1 000 measurements.

8 Expression of results

The results may be reported as individual values or as numbers of measurements in intervals of 0,05 % reflectance (½ V-step) or in intervals of 0,10 % reflectance (V-step). Calculate the mean reflectance and the standard deviation of the distribution as follows :

If individual readings are reported, calculate the mean reflectance and the standard deviation from equations (1) and (2) respectively.

$$\bar{R} = \frac{\Sigma R_i}{n} \qquad \dots (1)$$

$$\sigma = \sqrt{\frac{n\Sigma R_i^2 - (\Sigma R_i)^2}{n\,(n-1)}} \qquad \dots (2)$$

where

$\bar{R}$ is the mean maximum or mean random reflectance percentage;

R_i is the ith individual reading of reflectance;

n is the number of measurements;

σ is the standard deviation.

If the results are reported as the number of measurements in ½ V-steps or V-steps, the corresponding equations are given as follows :

$$R = \frac{\Sigma R_l\, x_l}{n}$$

$$\sigma = \sqrt{\frac{\Sigma(R_l - x_l)^2 - n\,\bar{R}^2}{n-1}}$$

where

R_l is the mid-value of the ½ V-step or V-step;

x_l is the number of reflectance measurements in the ½ V-step or V-step.

Record the submacerals of vitrinite to which the value of $\bar{R}$ refers, whether maximum or random reflectance measurements were made and the number of points measured. The percentage of the vitrinite in each ½ V-step or V-step may be plotted as a histogram. An example of a suitable method of expressing results is shown in table 2 and the corresponding histogram is shown in figure 4.

NOTE — A V-step has a range of 0,1 % reflectance and a ½ V-step a range of 0,05 % reflectance. In order to avoid overlap of reflectance values expressed to two places of decimals, the ranges of values belonging to selected V-steps and ½ V-steps are, for example, as follows :

V-step : 0,60 to 0,69; 0,70 to 0,79; etc. (inclusive)

½ V-step : 0,60 to 0,64; 0,65 to 0,69; etc. (inclusive)

The mid-point of range 0,60 to 0,69 is 0,645.

The mid-point of range 0,60 to 0,64 is 0,62.

Table 2 — An example of a method of expressing the results

Sample No. 1

Reflectance measured : Random

Vitrinite submacerals : Telocollinite and desmocollinite

Reflectance[1)]	Number of observations	Percentage observations
0,40 to 0,44		
0,45 to 0,49		
0,50 to 0,54		
0,55 to 0,59		
0,60 to 0,64		
0,65 to 0,69		
0,70 to 0,74		
0,75 to 0,79		
0,80 to 0,84		
0,85 to 0,89	2	—
0,90 to 0,94	12	2
0,95 to 0,99	12	2
1,00 to 1,04	15	3
1,05 to 1,09	14	3
1,10 to 1,14	39	8
1,15 to 1,19	78	15
1,20 to 1,24	47	9
1,25 to 1,29	39	8
1,30 to 1,34	18	4
1,35 to 1,39	20	4
1,40 to 1,44	23	5
1,45 to 1,49	29	6
1,50 to 1,54	66	13
1,55 to 1,59	65	13
1,60 to 1,64	13	3
1,65 to 1,69	8	2
1,70 to 1,74		
1,75 to 1,79		
1,80 to 1,84		
1,85 to 1,89		
1,90 to 1,94		
1,95 to 1,99		

Total number of measurements, n : 500

Mean reflectance, $\bar{R}$: 1,32 %

Standard deviation of the distribution, σ : 0,20 %

1) Upper and lower limits can be changed as appropriate.

9 Precision

9.1 Repeatability

The repeatability of the determination of the mean maximum or mean random reflectance is that value of the difference bet-

ween two single determinations each based on the same number of measurements carried out by the same operator on the same block using the same apparatus, below which 95 % of such differences are expected to lie. The repeatability is given by the formula

$$(2\sqrt{2})\sigma_t$$

where σ_t is the theoretical standard deviation.

The repeatability depends on a number of factors including

a) the limited accuracy in setting the calibration by means of the reflectance standards (see 7.2.5);

b) the permissible drift in the calibration during the measurements (see 7.3.2);

c) the number of measurements made and the range of reflectance occurring within the vitrinite even in a single coal seam.

The combined effect of these factors can be expressed as a standard deviation of the mean reflectance of up to 0,02 % for a single seam coal. This corresponds to a repeatability of up to 0,06 %.

9.2 Reproducibility

The reproducibility of the determination of the mean maximum or mean random reflectance is that value of the difference between two single determinations each based on the same number of measurements carried out by two different operators on two different subsamples taken from the same sample, using different equipment, below which 95 % of such differences are expected to lie. The reproducibility is given by the formula

$$(2\sqrt{2})\sigma_o$$

where σ_o is the observed standard deviation.

Provided that operators are adequately trained in the identification of vitrinite or the appropriate submacerals and that the reflectances of the standards used are reliably known, determinations of the mean reflectance by different operators in different laboratories show standard deviations of the order of 0,03 %. The reproducibility is thus of the order of 0,08 %.

10 Test report

The test report shall include the following information :

a) reference to this part of ISO 7404;

b) all details necessary for identification of the sample;

c) the total number of measurements;

d) type of measurement made, i.e. maximum or random;

e) type of vitrinite submacerals used in the determination;

f) the results obtained;

g) any other characteristics of the sample observed in the analysis that may be relevant to the use of the results.