Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

Indian Standard

METHODS FOR THE PETROGRAPHIC ANALYSIS OF BITUMINOUS COAL AND ANTHRACITE

PART 5 METHOD OF DETERMINING MICROSCOPICALLY THE REFLECTANCE OF VITRINITE

( First Revision )
NATIONAL FOREWORD

This Indian Standard (Part 5) (First Revision) which 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’ issued by the International Organization for Standardization (ISO) was adopted by the Bureau of Indian Standards on the recommendations of the Solid Mineral Fuels Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1987 which was largely based on ISO/DIS 7404-5. ISO 7404-5 was first published in 1984 and then revised in 1994 as ISO 7404-5:1994. The Committee, therefore, decided to revise this Indian Standard to completely align it with ISO 7404-5:1994 and publish as dual number standard. Consequently, the title has been modified.

The text of ISO Standard has been approved as suitable for publication as an Indian Standard without deviations. Certain conventions are, however, not identical to those used in Indian Standards. Attention is particularly drawn to the following:

a) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

b) Comma (, ) has been used as a decimal marker while in Indian Standards, the current practice is to use a point (.) as the decimal marker.

CROSS REFERENCES

In this adopted standard reference appears to certain International Standards for which Indian Standards also exist. The corresponding Indian Standards which are to be substituted in their place are listed below along with their degree of equivalence for the editions indicated.

<table>
<thead>
<tr>
<th>International Standard</th>
<th>Corresponding Indian Standard</th>
<th>Degree of Equivalence</th>
</tr>
</thead>
</table>

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2:1960 ‘Rules for rounding off numerical values (revised)’.
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.


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 $-\frac{dn}{dr}$ 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 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 0.5%, 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 $x \times 25$ and $x \times 60$ 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 $x \times 250$ and $x \times 750$.

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 $80 \mu m^2$ 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 $\pm$ 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$^{-1}$. 

IS 9127 (Part 5) : 2004
ISO 7404-5 : 1994
Figure 1 — Optical parts of a typical reflectance measuring microscope
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
vitrinite refers to one or more of the submacerals in the vitrinite group.

As explained in the last four paragraphs of the introduction, 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.

8.3.2 Measurement of the maximum reflectance of vitrinite in oil

Ensure that the polarizer is fitted to the microscope as specified in 8.1.2 and that the appropriate checks on the apparatus have been made as specified in 8.1 and 8.2.

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 18 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 step length of 0.5 mm. If the intersection of the crosslines covers a coal particle, use the mechanical stage to move the specimen, if necessary, so that the measuring area coincides with a suitable area of vitrinite and make a measurement. Return the mechanical stage to its original position and continue to make measurements at intervals of 0.5 mm. 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), recheck the calibration of the apparatus using the standard nearest in reflectance to that of the highest reflecting vitrinite in the specimen (see 8.2.5). If the reflectance of the standard differs from its theoretical value by more than 2% relative to the 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 range 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 1000 measurements.

8.3.3 Measurement of the random reflectance of vitrinite in oil

Adopt the same procedures as specified in 8.3.2, but make the measurements without the polarizer and without rotation of the sample block. Calibrate the apparatus as specified in 8.2.5.

Make reflectance measurements on the vitrinite until the required number has been recorded. If, for 98% of the measurements, the range 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 1000 measurements.

9 Expression of results

The results may be reported as individual values or as numbers of measurements in intervals of 0.05%
reflectance (1/2 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 maximum or mean random reflectance percentage, \( \bar{R} \), and the standard deviation, \( \sigma \), from equations (1) and (2) respectively.

\[
\bar{R} = \frac{\sum R_i}{n} \quad \ldots \text{(1)}
\]

\[
\sigma = \sqrt{\frac{n \sum R_i^2 - (\sum R_i)^2}{n(n-1)}} \quad \ldots \text{(2)}
\]

where

\( R_i \) is the \( i \)th individual reading of reflectance;

\( n \) is the number of measurements.

If the results are reported as the number of measurements in 1/2 V-steps or V-steps, the corresponding equations are given as follows:

\[
R = \frac{\sum R_i x_i}{n} \quad \ldots \text{(3)}
\]

\[
\sigma = \sqrt{\frac{\sum (R_i x_i)^2 - (\sum R_i)^2}{n - 1}} \quad \ldots \text{(4)}
\]

where

\( R_i \) is the mid-value of the 1/2 V-step or V-step;

\( x_i \) is the number of reflectance measurements in the 1/2 V-step or V-step.

Record the submacerals of vitrinite to which the value of \( R \) refers, whether maximum or random reflectance measurements were made and the number of points measured. The percentage of the vitrinite in each 1/2 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 3.

**NOTE 19** A V-step has a range of 0,1 % reflectance and a 1/2 V-step a range of 0,05 % reflectance. In order to avoid overlap of reflectance values expressed to two decimal places, the ranges of values belonging to selected V-steps and 1/2 V-steps are, for example, as follows:

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

1/2 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

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reflectance measured:</td>
<td>Random</td>
</tr>
<tr>
<td>Vitrinite submacerals (proportion of measurements):</td>
<td>Telocollinite (60 %) and desmocollinite (40 %)</td>
</tr>
<tr>
<td>Reflectance 1)</td>
<td>Number of observations</td>
</tr>
<tr>
<td>----------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>0,40 to 0,44</td>
<td>2</td>
</tr>
<tr>
<td>0,45 to 0,49</td>
<td>12</td>
</tr>
<tr>
<td>0,50 to 0,54</td>
<td>12</td>
</tr>
<tr>
<td>0,55 to 0,59</td>
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<tr>
<td>0,60 to 0,64</td>
<td>14</td>
</tr>
<tr>
<td>0,65 to 0,69</td>
<td>39</td>
</tr>
<tr>
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<td>39</td>
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<tr>
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<td>78</td>
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<tr>
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<td>1,75 to 1,79</td>
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<td>1,80 to 1,84</td>
<td>15</td>
</tr>
<tr>
<td>1,85 to 1,89</td>
<td>15</td>
</tr>
<tr>
<td>1,90 to 1,94</td>
<td>15</td>
</tr>
<tr>
<td>1,95 to 1,99</td>
<td>8</td>
</tr>
</tbody>
</table>

Total number of measurements, \( n \): 500

Mean reflectance, \( R \): 1,32 %

Standard deviation of the distribution, \( \sigma \): 0,20 %

1) Upper and lower limits can be changed as appropriate.
<table>
<thead>
<tr>
<th>Sample number</th>
<th>1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Date</td>
<td>78-11-03</td>
</tr>
<tr>
<td>Mean maximum reflectance, %</td>
<td>—</td>
</tr>
<tr>
<td>Mean random reflectance, %</td>
<td>1.32</td>
</tr>
<tr>
<td>Standard deviation</td>
<td>0.20</td>
</tr>
<tr>
<td>Vitrinite submacerals (proportion)</td>
<td>Telocollinite (60%) and desmocollinite (40%)</td>
</tr>
<tr>
<td>Number of measurements</td>
<td>500</td>
</tr>
</tbody>
</table>

1) Delete as appropriate.

Figure 3 — Histogram plotted from the results given in table 2

10 Precision

10.1 Repeatability

The repeatability 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 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 $\sigma_t$ 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 8.2.5);

b) the permissible drift in the calibration during the measurements (see 8.3.2);

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 %.

10.2 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

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

where $\sigma_o$ 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 in the order of 0.03 %. The reproducibility is thus in the order of 0.08 %.

11 Test report

The test report shall include the following information:

a) a reference to this part of ISO 7404;
b) all details necessary for identification of the sample;
c) the name and address of the testing laboratory;
d) date of test;
e) the total number of measurements;
f) type of measurements made, i.e. maximum or random;
g) type and proportion of vitrinite submacerals used in the determination;
h) the results obtained;
i) any other characteristics of the sample observed during the analysis that may be relevant to the use of the results.
Annex A
(informative)

Bibliography


NOTE 20 The second edition (1963), together with the first supplement (1971, corrected and revised in 1985), second supplement (1975) and third supplement (1993) may be obtained from Professor D.G. Murchison, Fossil Fuels and Environmental Geochemistry, Drummond Building, University of Newcastle, Newcastle-upon-Tyne, NE1 7RU, United Kingdom.

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