

Australian Standard™

Coal petrography

Part 2: Maceral analysis

This Australian Standard was prepared by Committee MN/1, Coal and Coke. It was approved on behalf of the Council of Standards Australia on 27 February 1998 and published on 5 May 1998.

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Australian Coal Association
Australian Coal Preparation Society
Australian Institute of Energy
Bureau of Steel Manufacturers of Australia
Coalfield Geology Council of New South Wales
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STANDARDS AUSTRALIA

RECONFIRMATION

OF

AS 2856.2—1998

Coal petrography

Part 2: Maceral analysis

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NOTES

Australian Standard™

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Part 2: Maceral analysis

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PREFACE

This Standard was prepared by the Standards Australia Committee MN/1, Coal and Coke to supersede AS 2856—1986, *Coal—Maceral analysis*. It sets out procedures for the maceral analysis of coal and classifies the different coal macerals.

This Standard provides a breakdown of maceral groups into subgroups and individual macerals, which are then classified by various properties.

The objective of this Standard is to provide a standardized procedure for the complete maceral analysis of coal.

The objective of the revision of the 1986 edition is to align with current terminology and to reflect the changes made by the International Committee for Coal and Organic Petrology (ICCP) in its 1994 handbook revision on *Vitrinite classification*.

The terms ‘normative’ and ‘informative’ have been used in this Standard to define the application of the appendix to which they apply. A ‘normative’ appendix is an integral part of a Standard, whereas an ‘informative’ appendix is only for information and guidance.

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STANDARDS AUSTRALIA

Australian Standard

Coal petrography

Part 2: Maceral analysis

1 SCOPE This Standard sets out a procedure for the maceral analysis of coal, aimed at obtaining the proportion of each maceral occurring within a coal, expressed as a volume percentage. It covers coals of all ranks.

The classification of coal macerals is set out in Appendix A,

NOTE: The ICCP subdivision of the vitrinite maceral group is given in Appendix B.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS

1038 Coal and coke—Analysis and testing

1038.22 Part 22: Higher rank coal—Mineral matter and water of constitution

2061 Preparation of coal samples for incident light microscopy

2418 Coal and coke—Glossary of terms

2486 Methods for microscopical determination of the reflectance of coal macerals

DIN

58884 Glass slides, glass cover slips and immersion oil for microscopy

3 PRINCIPLE A sample of coal is prepared as either a polished particulate block or a polished block in accordance with AS 2061. The block is examined using a reflected light microscope and the macerals are identified under an immersion medium by their relative reflectance, colour, morphology and fluorescence characteristics. The proportion of each maceral is determined by a point-count procedure.

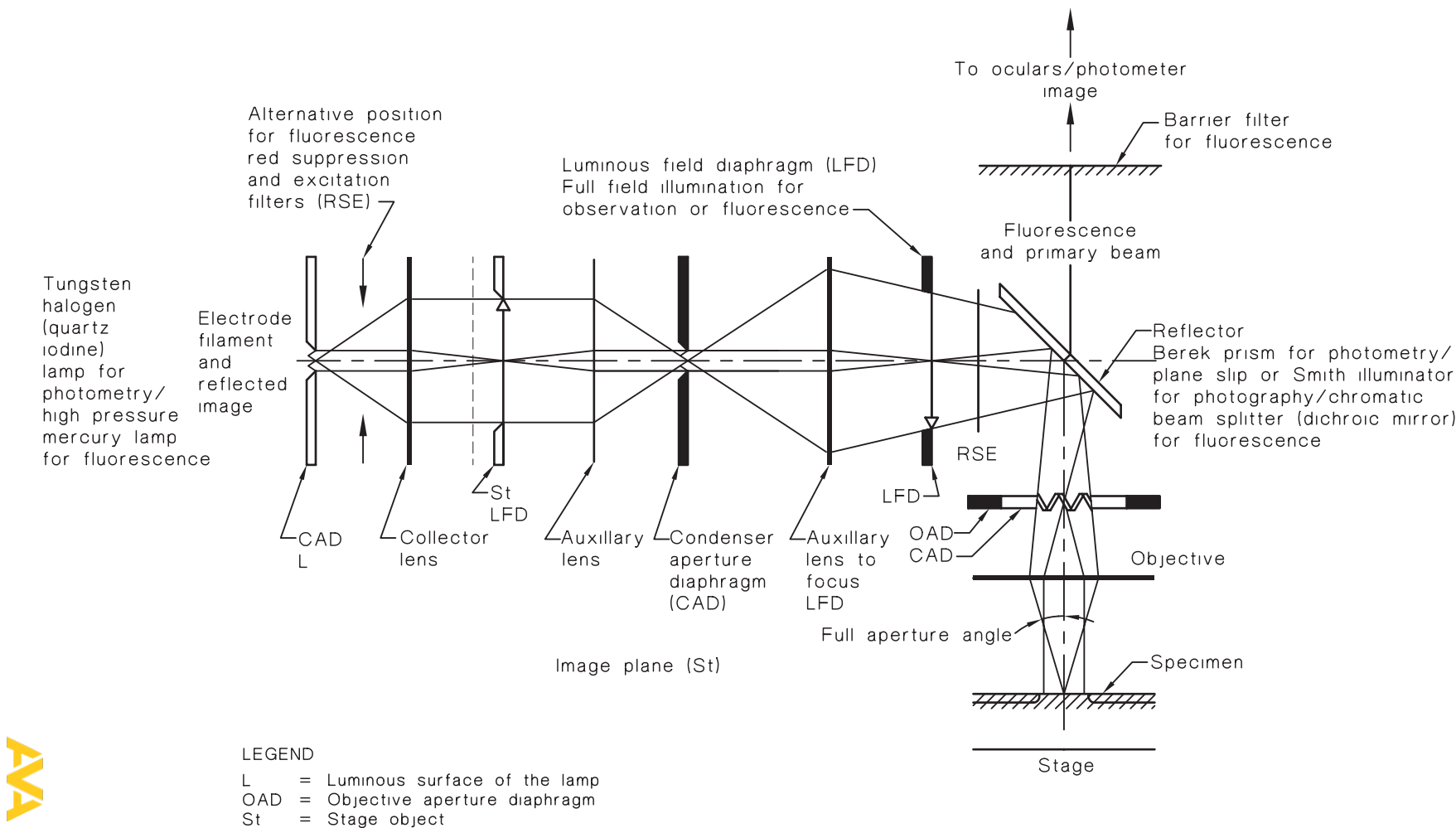
4 MATERIALS A low-fluorescing immersion medium, having a suitable refractive index which is compatible with the microscope objective, is necessary. If the reflectance of the macerals is to be measured, an immersion oil as specified in AS 2486 should be used. The oil commonly used conforms to that specified in DIN 58884.

5 APPARATUS The following apparatus is required:

- (a) *Reflected light microscope*—as described in AS 2486, having an immersion objective of magnification between 25× and 63× and eyepiece of magnification between 5× and 12.5×. It is desirable that the total magnification, including the tube magnification factor, is nominally between 400× and 600×. The eyepiece shall incorporate a suitable graticule.

The recommended optical system is shown in Figure 1. Descriptions of the light source, light-controlling apertures and vertical illuminator are given in AS 2486.

- (b) *Fluorescence microscope*—having recommended optical system for fluorescence mode as shown in Figure 1.



NOTE: Heavy and solid lines indicate location of part of the optical system, thin and open lines indicate the location of the optical system, images of the luminous field diaphragm, specimen and light source.

FIGURE 1 REFLECTED LIGHT, INCIDENT BRIGHT-FIELD ILLUMINATION (KÖHLER) WITH GLASS PLATE OR PRISM REFLECTOR

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Fluorescence colours described in this Standard are based on the use of the following:

- (i) High-pressure mercury lamp (100 W).
- (ii) Red suppression and excitation filters BG38 and BG3.
- (iii) Dichroic mirror TK 400 (chromatic beam splitter).
- (iv) Barrier filter K490.

Other filter combinations are equally acceptable but will result in slightly different fluorescence colours.

WARNING: REMOVE THE POLARIZER FROM THE VERTICAL ILLUMINATOR AND INSERT THE BARRIER FILTER INTO THE LIGHT PATH BEFORE IRRADIATING THE SAMPLE WITH ULTRA-VIOLET LIGHT. FAILURE TO DO SO WILL CAUSE DAMAGE TO THE POLARIZER AND IRREPARABLE DAMAGE TO THE EYES.

- (c) *Point-counting stage*—capable of advancing the specimen by equal steps of such length that only a negligibly small proportion of the particles examined receive more than one count on the same particle. The step length shall be equal to approximately half the maximum particle diameter and thus shall be 0.5 mm to 0.7 mm for samples having a nominal top size of 1 mm. The stage shall also permit a similar stepped advance in the perpendicular direction. The former movement is preferably actuated by the counter mechanism, whereas the perpendicular movement is satisfactorily performed manually.
- (d) *Counter*—capable of registering the counts in each category, and preferably the total of petrographic components.
- (e) *Sample mounting equipment*—comprising carrier slides, modelling clay and levelling press.

6 PROCEDURE

6.1 Sample preparation To ensure that the coal used to prepare a particulate block has a nominal maximum particle size of 1.0 mm and contains a minimum of fines, the preparation method given in AS 2061 should be followed. Any marked increase in the amount of fines or oversize particles may cause deviations in the results, and should be noted in the test report.

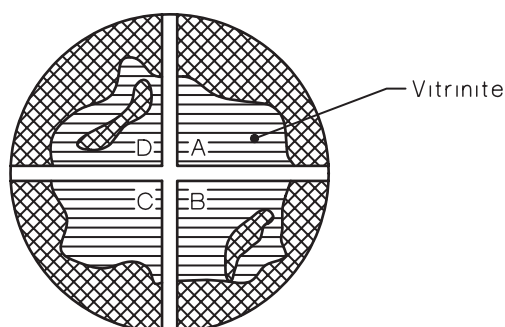
6.2 Determination The determination shall be carried out as follows:

- (a) Adjust the microscope for Köhler illumination (see Figure 1).
- (b) Set up the levelled particulate block on the stage, place the immersion medium on the surface of the block, focus and observe the image in the microscope.

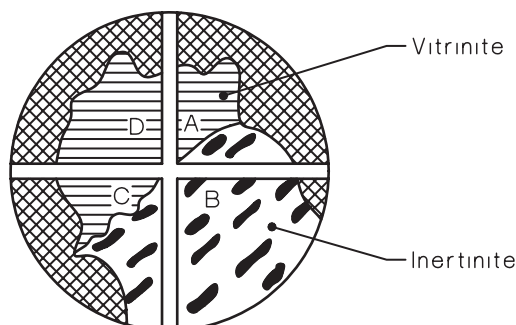
NOTE: Initial scanning using an air lens may be helpful in identifying components having refractive indices close to that of the immersion medium (e.g. quartz grains).

- (c) Identify the material lying under the intersection of the crosslines and carry out the point-count procedure as follows:

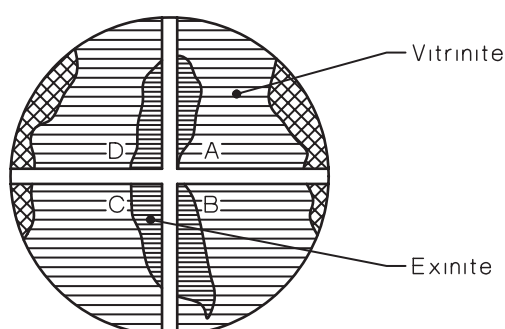
<i>Crosslines on</i>	<i>Action</i>
Maceral	Press counter button for appropriate maceral group.
Mounting medium	Move to the next point.
Minerals	Press counter button for appropriate mineral or ignore the point, if counting mineral-free.
Empty pore in a maceral or a void	Move to the next point.
Boundary cases	Refer to Figure 2



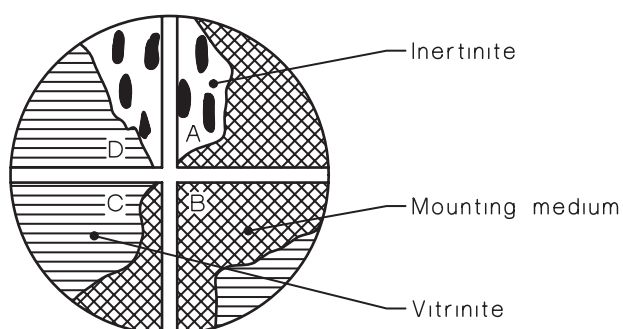
(a) Normal case—Count point A (vitrinite)



(b) Boundary case—Count point B (inertinite)



(c) Boundary case—Count point C (exinite)



(d) Boundary case—The point is not counted

NOTE: Width of crosslines exaggerated for clarity.

FIGURE 2 NORMAL AND BOUNDARY CASES BETWEEN MACERALS OR BETWEEN MACERAL AND MOUNTING MEDIUM

- (d) Advance the block by one step in a direction perpendicular to any stratification known to be present due to the sample preparation technique, and continue counting and traversing the specimen. At the end of a traverse, advance the block by a step of at least equal length in the perpendicular direction to start the next parallel traverse. The step length is chosen to ensure a uniform counting of points over the surface of the block.
- (e) The total number of counts shall be at least 500, but the whole block should be covered and the final traverse completed.

7 EXPRESSION OF RESULTS Calculate the volume percentage of each component, which is equal to the percentage number of points counted on it, expressing the results to the first decimal place. The form of the results will depend on the procedure adopted with regard to minerals and shall be expressed on one of the following bases:

- (a) Minerals free basis: $\% V + \% L + \% I = 100$.
- (b) Minerals counted: $\% V + \% L + \% I + \% M = 100$.
- (c) Minerals calculated: $\% V + \% L + \% I + \% M = 100$.

In alternative (c), minerals are included in the point count, but their volume percentages are calculated from mass percentages (see below). Mineral mass percentages can be calculated from the determined ash by means of low-temperature ashing (see AS 1038.22) or an accepted empirical relationship, e.g. minerals mass percent = $1.1 \times \text{ash}$.

The procedure adopted and the number of points counted shall be indicated in the report of the results.

NOTE: Assuming the ratio of mineral density to coal matter density = 2 (valid except in very high pyritic coal), then—

$$\begin{aligned} \text{Volume percent of minerals} &= \frac{\text{Mineral mass percent}/2}{\text{Coal matter mass percent} + (\text{Mineral mass percent})/2} \times 100 \\ &= \frac{\text{Mineral mass percent}}{2(\text{Coal matter mass percent}) + (\text{Mineral mass percent})} \times 100 \end{aligned}$$

But—

Coal matter mass percent = $100 - \text{Mineral mass percent}$

Therefore—

$$\text{Volume percent of mineral} = \frac{100 (\text{Mineral mass percent})}{200 - \text{Mineral mass percent}}$$

If a direct measure of mineral mass percent is available, then the mineral volume percent can be calculated from the above relationship.

8 EXPERIMENTAL ERROR

8.1 Repeatability The repeatability of the determination of the volume percentage of a component is that difference between two single determinations each based on the same number of point counts carried out by the same operator on the same sample, using the same apparatus, within which 95% of such differences would be expected to lie. The repeatability may be calculated from the following expression:

$$\text{Repeatability} = (2\sqrt{2})s \quad \dots (1)$$

where s is the standard deviation.

Provided that the operator makes negligible errors in classifying the macerals, the results of an analysis will be subject to standard deviations calculable on the basis of the binomial distribution. Where $p\%$ of the total number of counts (N) are registered for a given maceral group, the standard deviation of p is—

$$s = \sqrt{p(100 - p)/N} \quad \dots (2)$$

Values for the theoretical standard deviation and repeatability calculated for a range of volume percentages of a component are given in Table 1 based on 500 point counts.

TABLE 1
THEORETICAL STANDARD DEVIATION AND
REPEATABILITY OF THE PERCENTAGE OF
A COMPONENT BASED ON 500 POINT COUNTS

Volume percentage of component	Standard deviation of the volume percentage (s)	Repeatability ($2\sqrt{2}s$)
5	1.0	2.8
20	1.8	5.1
50	2.2	6.3
80	1.8	5.1
95	1.0	2.8

8.2 Reproducibility The reproducibility of the determination of the volume percentage of a component is that difference between two single determinations each based on the same number of point counts, carried out by two different operators on two different subsamples taken from the same sample, using different equipment, within which 95% of such differences would be expected to lie.

The reproducibility is determined from the following equation:

$$\text{Reproducibility} = (2\sqrt{2})s_o \quad \dots (3)$$

where s_o is the observed standard deviation.

Values of the observed standard deviation will normally exceed those of the theoretical values given in Table 1 due to the differences in identification of the macerals by different operators, and have been found to vary from approximately 1.5 times to 2.0 times the theoretical values dependent on the rank and the heterogeneity of the coal.

The observed standard deviation is determined from the following expression:

$$s_o = \sqrt{\frac{\sum_{i=1}^k (p_i - \bar{p})^2}{k - 1}} \quad \dots (4)$$

where

s_o = observed standard deviation

$$\bar{p} = \sum_{i=1}^k p_i/k$$

p_i = maceral percentage obtained by the operator

k = number of operators