INTERNATIONAL STANDARD

ISO 7404-3

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

Part 3:
Method of determining maceral group composition

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

Partie 3: Détermination de la composition en groupes de macéraux

INTERNATIONAL ISO

Reference number
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Foreword

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

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 7404-3 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels*.

This second edition cancels and replaces the first edition (ISO 7404-3:1984), 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.

Macerals are microscopically recognizable organic constituents of coal, and can be grouped together into three maceral groups: vitrinite, liptinite and inertinite.

Maceral groups and their subdivisions are listed in ISO 7404-1. The properties of a given coal are determined by the proportions and associations of the macerals and minerals present and by the rank of the coal. The method of determining maceral group composition described in this part of ISO 7404 applies only to determinations made in reflected white light. Further analysis by other techniques, such as fluorescence microscopy, can be used to assist in maceral identification.

In addition to the macerals, it is possible to identify certain minerals in coal and these may either be determined as separate categories or be ignored. As some of the minerals cannot be satisfactorily identified under the microscope, an estimate of the total mineral matter content may be obtained from the ash.

Methods for the petrographic analysis of bituminous coal and anthracite —

Part 3:
Method of determining maceral group composition

1 Scope

This part of ISO 7404 specifies a method of determining the proportions of the maceral groups (and the minerals if desired) in coals. It is concerned only with determinations made on polished particulate blocks using reflected white light. If needed, the proportions of the individual macerals may be determined by the same procedure. It is not concerned with the determination of the proportions of naturally occurring maceral associations (i.e. microlithotypes, see ISO 7404-4).

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.*

ISO 7404-4:1988, *Methods for the petrographic analysis of bituminous coal and anthracite — Part 4: Method of determining microlithotype, carbominerite and minerite composition.*

ISO 7404-5:1994, *Methods for the petrographic analysis of bituminous coal and anthracite — Part 5: Method of determining microscopically the reflectance of vitrinite.*

3 Definitions

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

4 Principle

A representative sample of coal is used to prepare a particulate block as described in ISO 7404-2. This is examined using a reflected light microscope and the maceral groups are identified under an immersion medium by their relative reflectance, colour, size and morphology. Their proportions are determined by a point count procedure.

5 Material

Immersion medium, having a suitable refractive index and compatible with the microscope objective.

NOTE 1 It is recommended that an oil with a refractive index of 1,518 0 as specified in ISO 7404-5 be used, especially if the reflectance of the macerals is to be measured.

6 Apparatus

6.1 Reflected light microscope, having an immersion objective of magnification between × 25 and × 60 and an eyepiece of magnification between × 8 and × 12. The eyepiece incorporates a fine crossline graticule.

6.2 Mechanical stage, capable of advancing the specimen laterally by equal steps of such length that only a negligibly small proportion of the particles examined receives more than one count on the same particle. The step length is equal to half the maximum particle diameter, i.e. 0,5 mm to 0,6 mm for samples with a standard top particle size of 1 mm. The stage also permits a similar stepped advance in the perpendicular direction. The lateral movement is actuated preferably by the counter mechanism, whereas the perpendicular movement may be satisfactorily performed manually.

6.3 Counter, capable of registering the counts in each category and preferably the grand total of petrographic components.

6.4 Sample mounting equipment, comprising slides, modelling clay and levelling device.

7 Preparation of coal sample

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

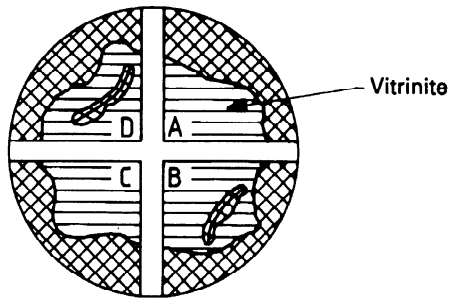
8 Procedure

Adjust the microscope (6.1) for Köhler illumination. Set up the levelled particulate block on the stage, place the immersion medium (clause 5) on the surface of the block, focus and observe the image in the microscope. Identify the material lying under the intersection of the crosslines and carry out the point count procedure as follows.

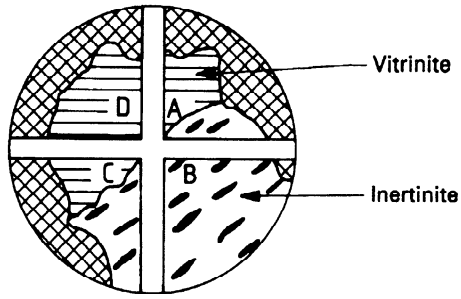
Crosslines on	Action
Vitrinite (V), liptinite (L) or inertinite (I)	Operate the counter for appropriate maceral group.
Mounting medium	Ignore the point.
Minerals (M)	Operate the counter for pyrite, shale, etc. or ignore the point (see the last three paragraphs of the introduction and clause 10).
Boundary between macerals or between maceral and mounting medium	Examine in turn the material lying immediately adjacent to crossline intersection in the top right, bottom right, bottom left and top left quadrants. Take the first of these which does not have a boundary in it, and operate the counter for this material (see figure 1).
Empty pore in a maceral or a void	Ignore the point

Advance the block by one step in the left-to-right direction, 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. Choose the step length to ensure a uniform counting of points over the surface of the block.

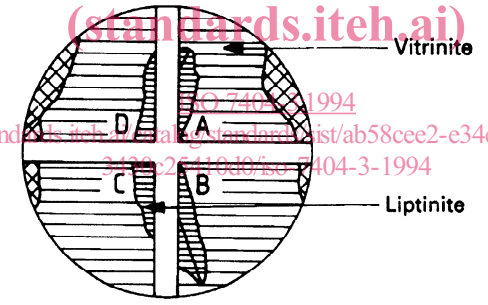
Count a total of at least 500 points.



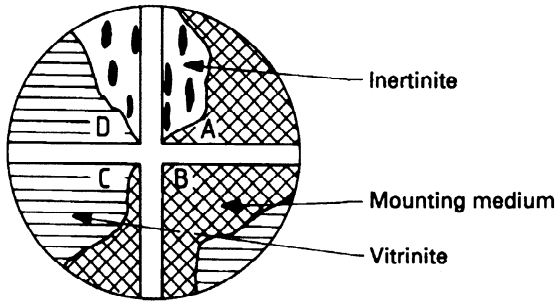
a) Normal case — count point A (vitrinite)



b) Boundary case — count point B (inertinite)



c) Boundary case — count point C (liptinite)



d) Boundary case — the point is not counted

NOTE — Width of crosslines exaggerated for clarity.

Figure 1 — Normal and boundary cases between macerals or between maceral and mounting medium

9 Expression of results

Calculate the volume percentage of each component [vitrinite (V), liptinite (L), inertinite (I)], which is equal to the percentage number of points counted on it, expressing the results to the nearest integer. The form of the results depends on the procedure adopted with regard to minerals (see the last three paragraphs of the introduction) and is expressed on one of the following bases:

a) minerals ignored:
% V + % L + % I = 100

b) minerals (M) counted:
% V + % L + % I + % M = 100

c) minerals matter (MM) calculated:
% V + % L + % I + % MM = 100

In alternative c), minerals are ignored in the point count but the volume percentage of mineral matter is calculated from the determined ash by means of an accepted empirical relationship.

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

NOTE 2 Examples of equations to calculate the mineral matter, % MM, as a volume percentage, are as follows:

$$\% \text{ MM} = 0{,}61 w_A - 0{,}21 \quad \ldots (1)$$

$$\% \text{ MM} = \frac{w_M}{2{,}07 - 0{,}011 w_M} \quad \ldots (2)$$

where

w_A is the ash content, as a mass percentage on the air-dried basis;

w_M is the mineral matter content, as a mass percentage, given by the equation

$$w_M = 1{,}08 w_A + 0{,}55 w_S$$

where w_S is the sulfur content, as a mass percentage on the air-dried basis.

Equation (2) is based on assumed relative densities of 1,35 and 2,8 for the macerals and mineral matter respectively.

These equations have been found satisfactory in certain coal basins but may not necessarily apply globally. It is essential for the user to establish suitable equations for the coals being analysed.

10 Precision

10.1 Repeatability limit

The repeatability limit of the determination of the volume percentage of a component is the value of the difference between two single determinations, each based on the same number of points counted, 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 limit may be calculated from the formula

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

where σ_t is the theoretical standard deviation.

Provided that the operator makes negligible errors in classifying the macerals, the results of an analysis are subject to standard deviations calculable on the basis of the binominal distribution.

Where p % of the total number of points counted, N, is registered for a given maceral group, the theoretical standard deviation, σ_t, of p is given by the equation

$$\sigma_t = \sqrt{\frac{p(100 - p)}{N}}$$

Values based on counts of 500 points for the theoretical standard deviation, coefficient of variation and repeatability calculated for a range of volume percentages of a component are given in table 1.

Table 1 — Theoretical standard deviation and repeatability limit of the percentage of a component, based on counts of 500 points

Volume %, p	Standard deviation, σ_t	Coefficient of variation, $100\sigma_t/p$	Repeatability limit, $\left(2\sqrt{2}\right)\sigma_t$
5	1,0	20	2,8
20	1,8	9	5,1
50	2,2	4,4	6,3
80	1,8	2,3	5,1
95	1,0	1,1	2,8

NOTE 3 For example, if the volume percentage of vitrinite in a sample is 80 %, then an operator can expect to obtain two results differing by less than 5,1 percentage points (e.g. 78 % and 83 %) in 19 cases out of 20.

10.2 Reproducibility limit

The reproducibility limit of the determination of the volume percentage of a component is that value of the difference between two single determinations, each based on the same number of points counted, 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 limit is given by the formula

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

where σ_o is the observed standard deviation.

Values of the observed standard deviation normally exceed the values for the theoretical standard deviation given in table 1 owing to the misidentification of the macerals by different operators and to variation between subsamples; they have been found to vary from approximately 1,5 to 2,0 times the theoretical values, depending on the rank and the heterogeneity of the coal.

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 number of points counted;

f) whether minerals were counted or ignored, or whether the mineral matter was calculated, and (if calculated) the equation used;

g) the results obtained;

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

NOTE 4 It may also be useful to include nominal magnification, point count stage step size and line traverse spacing.