International Standard ISO 7404/3

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

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

First edition — 1984-11-15

UDC 662.66 : 552 : 620.186 **Ref. No. ISO 7404/3-1984 (E)**

Descriptors: minerals and ores, solid fuels, coal, bituminous coal, anthracite, tests, determination of content, organic matter, mineral matter, microscopical analysis.

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.

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.

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

Printed in Switzerland

Methods for the petrographic analysis of bituminous coal and anthracite — Part 3: Method of determining maceral group composition

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 Macerals are microscopically recognizable organic constituents of coal, and can be grouped together into three maceral groups: vitrinite, exinite (liptinite) and inertinite. 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. Determinations using other techniques such as fluorescence microscopy may give different results, but the techniques are not yet at a stage where standardization is feasible.

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 mineral matter cannot be satisfactorily identified under the microscope, an estimate of the total mineral matter content may be obtained from the ash.

1 Scope and field of application

This part of ISO 7404 specifies a method of determining the proportions of the maceral groups (and the mineral matter 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)].

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 References

ISO 7404, *Methods for the petrographic analysis of bituminous coal and anthracite* —

Part 1: Glossary of terms.

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

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

Part 5: Method of determining microscopically the reflectance of vitrinite.

3 Definitions

For the purpose of this part of ISO 7404 the definitions of 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 macerals are identified under an immersion medium by their relative reflectance, colour 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 — If the reflectance of the macerals is to be measured , an immersion oil as specified in ISO 7405/5 should be used.

6 Apparatus

6.1 Reflected light microscope, having an immersion objective of magnification between X 25 and X 60 and eyepiece of magnification between X 8 and X 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 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 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:

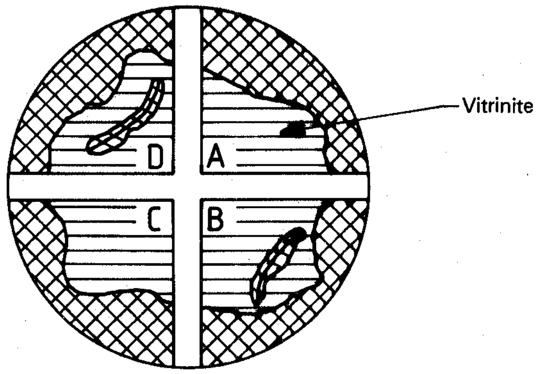
Crosslines on	Action
Vitrinite (V), exinite (E) or inertinite (I)	Operate the counter for appropriate maceral group.
Mounting medium	Ignore the point.
Mineral matter (MM)	Operate the counter for pyrite, shale, etc. or ignore the point, (see 0.2 and clause 9).
Boundary between macerals or between maceral 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 not having a boundary in it and operate counter for this material (see the figure).
Empty pore in a maceral or a void	Ignore the point.

Advance the block by one step in the left-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.

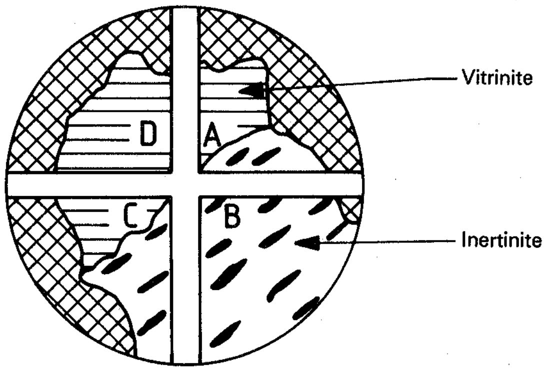
Make a total of at least 500 point counts.

1) At present at the stage of draft.

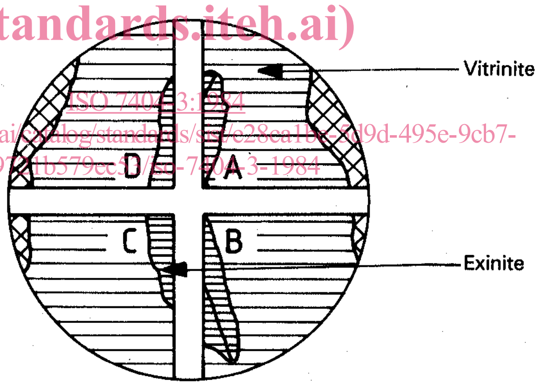
2) In preparation.



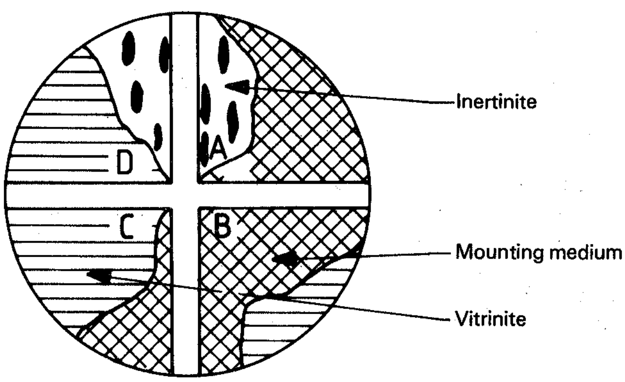
a) **Normal case — Count point A (vitrinite)**



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

8 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 nearest integer. The form of the results depends on the procedure adopted with regard to mineral matter (see 0.2) and has to be expressed on one of the following bases:

a) mineral matter ignored:
% V + % E + % I = 100

b) mineral matter counted:
% V + % E + % I + % MM = 100

c) mineral matter calculated:
% V + % E + % I + % MM = 100

In alternative c), mineral matter is ignored in the point count but its volume percentage 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 — Examples of equations to calculate the mineral matter, % MM, as a volume percentage, are as follows:

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

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

where

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

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

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

in which w_S is the sulfur, 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.

9 Precision

9.1 Repeatability

The repeatability 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 point counts 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 may be calculated from the formula

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

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 binomial distribution. Where p % of the total number of counts, 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 500 point counts for the theoretical standard deviation, coefficient of variation and repeatability calculated for a range of volume percentages of a component are given in the table.

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

Volume %, p	Standard deviation, σ_t	Coefficient of variation, $100\sigma_t/p$	Repeatability $(2\sqrt{2})\sigma_t$
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 — 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.

9.2 Reproducibility

The reproducibility 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 point counts 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.

Values of the observed standard deviation normally exceed the values for the theoretical standard deviation given in the table 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.

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 number of points counted;

d) whether mineral matter is counted, ignored or calculated, and (if calculated) the equation used;

e) the results obtained;

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