SLOVENSKI STANDARD
SIST ISO 7404-4:1998

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Metode petrografskih analiz bituminoznega premoga in antracita - 4. del: Določ evanje mikrolitotipov, karbamineritov in mineritov

Methods for the petrographic analysis of bituminous coal and anthracite -- Part 4: Method of determining microlithotype, carbominerite and minerite composition

Méthodes d'analyse pétrographique des charbons bitumineux et de l'anthracite -- Partie 4: Détermination de la composition en microlithotypes, carbominérites et minérites

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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Methods for the petrographic analysis of bituminous coal and anthracite —

Part 4:
Method of determining microlithotype, carbominerite and minerite composition

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

Partie 4: Détermination de la composition en microlithotypes, carbominérites et minérites

Reference number
ISO 7404-4 : 1988 (E)

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 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 7404-4 was prepared by Technical Committee ISO/TC 27, *Solid mineral fuels.*

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Methods for the petrographic analysis of bituminous coal and anthracite —

Part 4:
Method of determining microlithotype, carbominerite and minerite 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.

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* published by the ICCP[1].

0.2 Microlithotypes are the naturally occurring associations of macerals which characterize the visibly different types of coal. By convention, the identity of a microlithotype is determined by the maceral group or groups occurring within an area of at least 50 µm × 50 µm and which are present in amounts equal to or exceeding 5 % by volume. Hence they can comprise a single maceral or maceral group if it exceeds these dimensions. Microlithotypes may include up to 20 % by volume of minerals such as clay, quartz and carbonates or up to 5 % by volume of sulfide minerals. If the content of mineral matter exceeds these amounts, the material is designated as minerite or carbominerite depending on the proportions of coal and mineral matter.

Carbominerites can be subdivided according to the type of mineral matter.

Microlithotypes contribute information on the genesis of coal seams and can assist in solving problems of seam correlation. Because they determine, together with rank and mineral matter, the hardness and density of the bulk coal substance, microlithotypes affect the behaviour of coal in mining and coal preparation processes. The different microlithotypes determine, under given geological conditions, the distribution of micro-cracks and to some extent the cleat in the coal. The results of maceral analyses can be interpreted more meaningfully from a knowledge of microlithotype composition. Such information can assist in explaining the behaviour of coal in commercial and experimental utilization processes where the association of macerals is known to be important.

1) 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.

NOTE — The percentage volume of carbonate, clay and quartz minerals on the one hand and sulfide minerals on the other, which define the carbominerites and minerites, correspond to the densities which separate coal from middlings and middlings from discard in coal preparation.

1 Scope and field of application

This part of ISO 7404 specifies a method, using a graticule with 20 crossline intersections, for determining the proportions of microlithotypes, carbominerite and minerite in coals. It applies only to determinations made on polished particulate blocks using reflected white light.

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.*

— *Part 3: Method of determining maceral group composition.*

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

3 Definitions

For the purposes of this part of ISO 7404, the definitions of ISO 7404-1, together with the following, apply.

point: An observation of a portion of the particulate block through the eyepiece graticule made during the analysis.

NOTE — The allocation of a point to a particular microlithotype, or to carbominerite or minerite, is determined by the macerals and/or mineral matter present at the 20 intersections of the graticule. A point is only counted if at least 10 intersections fall on a particle.

4 Principle

Examination by using a reflected light microscope and point count procedure of a representative sample of coal prepared as a particulate block as described in ISO 7404-2. Identification under an immersion medium of the microlithotypes from their maceral composition. The proportions of the macerals are determined using a graticule having a grid with 20 points of intersection spaced to define a distance of 50 µm on the specimen between extreme intersections in the x (abscissa) and y (ordinate) directions respectively. The use of such a graticule allows the analyst to comply with the 5 % minimum content and 50 µm minimum size stipulation.

5 Material

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

NOTE — If reflectance measurements are required on the same particulate block an immersion oil as specified in ISO 7404-5 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 shall have the facility for inserting a graticule.

6.2 Graticule, inscribed with a grid having 20 crossline intersections according to the pattern shown in figure 1. The effective distance between extreme intersections in the x and y directions respectively is 50 µm.

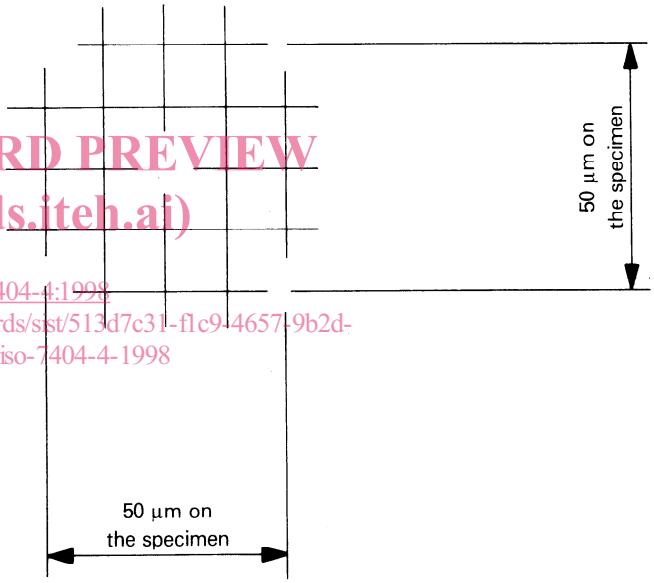

Figure 1 — Design of grid for microlithotype analysis

NOTES

1 With a total number of 20 crossline intersections a maceral appearing beneath a single intersection can be assumed to occupy 5 % by volume of the area covered by the grid.

2 The graticule is designed for use with a particular combination of objective and eyepiece. Change in either objective or eyepiece will necessitate the use of a grid of different dimensions.

6.3 Mechanical stage, capable of advancing the specimen in the x-direction 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 y-direction. The stage movement may be actuated manually or by the counter mechanism.

6.4 Counter, capable of registering the points in each category and preferably the grand total of points counted.

6.5 Sample mounting equipment, comprising slides, modelling clay and levelling press.

7 Procedure

Insert the graticule (6.2) into the eyepiece of the microscope (6.1).

Adjust the microscope for Köhler illumination. Place the levelled particulate block, prepared in accordance with ISO 7404-2, on the stage. Add the immersion medium to the surface of the block, focus and observe the image in the microscope.

Assess the number of crossline intersections lying on the particle in order to decide whether the point should be recorded in the counting procedure and, if so, whether it should be assigned to the category of a microlithotype, carbominerite or minerite. If the number of intersections on one particle is 10 or more the point shall be accepted for analysis. If there are no intersections on coal or mineral matter, the point is ignored (i.e. it is not recorded). If the number is less than 10 it shall be recorded as a rejected category and the stage shall be advanced by one step. The number of such rejected points shall not exceed 10 % of the total accepted and rejected points. If the proportion exceeds 10 %, excessive fines may have been produced during preparation and a fresh sample should be prepared if material is available. If this is not possible the fact shall be recorded in the test report. For identifying the material under the individual crosslines, apply the procedure described in ISO 7404-3. The criteria for determining accepted and rejected points are shown in figure 2.

If the particle is accepted for analysis, assess the number of intersections lying on mineral matter. If the number of crossline intersections on mineral matter exceeds the number shown in table 1 for a given number of intersections lying within the particle, the material is either carbominerite or minerite.

Table 1 — Maximum permissible number of intersections falling on mineral matter for the point to be classified as a microlithotype

No. of intersections lying within the particle	Number of intersections on carbonate, clay, quartz	Number of intersections on sulfide
16 to 20	3	0
11 to 15	2	0
10	1	0

If the particle is a microlithotype, it is identified according to the criteria shown in table 2 ignoring any intersections on carbonate, clay or quartz.

Table 2 — Delimitation of microlithotypes

Microlithotype	Location of the crossline intersections lying in the coal
Vitrite	All intersections in the vitrinite
Liptite	All intersections in the exinite (liptinite)
Inertite	All intersections in the inertinite
Clarite	All intersections in the vitrinite and exinite, at least one intersection in each of the two maceral groups
Durite	All intersections in the inertinite and exinite, at least one intersection in each of the two maceral groups
Vitrinertite	All intersections in the vitrinite and inertinite, at least one intersection in each of the two maceral groups
Trimacerite	At least one intersection in each of the three maceral groups

These criteria are applicable to microlithotypes containing mineral matter not exceeding the limits given in table 1.

If the intersections on mineral matter exceed the limits given in table 1, determine whether the particle is minerite or a carbominerite according to the criteria given in table 3 and table 4, if necessary. In using table 3 to identify carbominerite or

Table 3 — Delimitation of carbominerite (excluding carbopolyminerite with sulfide) and minerite

No. of intersections lying within particle	Intersections falling on specified minerals			
	Carbominerite		Minerite	
	Carbonate, clay, quartz	Sulfide	Carbonate, clay, quartz	Sulfide
19 or 20	4 to 11	1 to 3	> 11	> 3
17 or 18	4 to 10	1 to 3	> 10	> 3
16	4 to 9	1 to 3	> 9	> 3
14 or 15	3 to 8	1 or 2	> 8	> 2
12 or 13	3 to 7	1 or 2	> 7	> 2
11	3 to 6	1 or 2	> 6	> 2
10	2 to 5	1	> 5	> 1

minerite, the minerals appearing under the intersections shall be either sulfide or other minerals, but not both. The carbominerite may be named carbankerite, carbargilite, carbosilicite, carbopyrite or carbopolyminerite, according to the mineral matter appearing under the intersections.

If both sulfide and other minerals occur under intersections of the grid, within the limits given in table 4, the carbominerite is named carbopolyminerite, otherwise the particle is again assigned to minerite.

Table 4 — Delimitation of carbopolyminerite with sulfide

No. of intersections lying within the particle	Carbopolyminerite	
	Intersection falling on specified minerals	
	Carbonate, clay, quartz	Sulfide
16 to 20	1 to 3	1 to 3
11 to 15	1 or 2	1 or 2
10	1	1

The maceral group or groups associated with the mineral matter and appearing under the residual intersections of the grid may be recorded to further characterize the carbominerite.

Having identified the point, advance the particulate block by one step along the x-axis, and continue counting and traversing the specimen. At the end of a traverse, advance the block by a step of at least equal length along the y-axis to start the next parallel traverse. The step length shall be chosen to ensure a uniform counting of points over the surface of the particulate block.

The total number of accepted points shall be at least 500.

NOTE — For the assignment of a microlithotype, only the maceral group or groups appearing under the crossline intersections are considered. If a natural boundary between the two different microlithotypes lies under the grid, the assessment is made as if there were no boundary.

8 Expression of results

Report the number of accepted points per microlithotype, per carbominerite and per minerite as the percentage of total number of accepted points. Express the values obtained as percentages by volume to the nearest integer.

The number of accepted points and the percentage of rejected points shall be indicated in the test report. An example of a suitable method of expressing results is shown in table 5.

The nature of coal associated with carbominerite and minerite, if determined, expressed as

a) general qualitative observations; or

b) quantitative record of maceral groups under residual intersections

may be recorded as shown in table 6.

Table 5 — An example of a method of expressing the results

Sample No.	Date:	
Microlithotype	**Number of accepted points**	**Volume %**
Vitrite	101	20
Liptite		
Inertite	64	13
Clarite	57	11
Durite	115	23
Vitrinertite		
Trimacerite	141	28
	Total microlithotypes	95
Carbominerite	**Number of accepted points**	**Volume %**
Carbargilite	13	3
Carbankerite		
Carbosilicite		
Carbopyrite	5	1
Carbopolyminerite		
	Total carbominerite	4
	Number of accepted points	**Volume %**
Minerite	4	1
	Grand total	100
Total number of accepted points	500	
Percentage of rejected points	7	

Table 6 — Nature of coal associated with carbominerite and minerite (example)

Carbominerite/minerite (specify)	Associated maceral group(s) observed[1)]	Number of accepted points	Accepted points as a percentage of carbominerite/minerite
Carbargilite	V	3	20
	V + E	12	80
Carbopyrite	V	1	20
	V + E	3	60
	V + E + I	1	20
Minerite	V + I	5	100

1) E = exinite
I = inertinite
V = vitrinite

9 Precision

9.1 Repeatability

The repeatability of the determination of the volume percentage of a component is the 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, below which 95 % of such differences would be 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 microlithotypes, 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, is registered for a given microlithotype, the standard deviation, σ_t, of p is given by the equation

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

Values for the theoretical standard deviation and repeatability calculated for a range of volume percentages of a component are given in table 7 based on 500 point counts.

Table 7 — Theoretical standard deviation and repeatability of the percentage of a component based on 500 point counts

Volume percentage, p	Standard deviation, σ_t, of the volume percentage	Repeatability, $(2\sqrt{2})\,\sigma_t$
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

9.2 Reproducibility

The reproducibility of the determination of the volume percentage of a component is the difference between two single determinations each based on the same number of point counts, carried out by two different operators on two separate sub-samples taken from the same sample, using different equipment, below which 95 % of such differences would be 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 those of the theoretical values given in table 7 due to misidentification of the microlithotypes or minerals by different operators and to variation between sub-samples. There is, at present, insufficient evidence from collaborative analyses to estimate the effect of misidentification.

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) number of accepted points analysed and the percentage of rejected points;

d) the results obtained;

e) the nature of coal associated with the carbominerite and minerite if determined.