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MOSCOU*

<p>Edition 1975</p>	<p>International Commission for Coal Petrology Nomenclature Subcommittee Analysis Subcommittee</p>	
	<p>Introduction to the 2nd supplement to the 2nd edition</p>	

The 2nd supplement to the 2nd edition of the International Handbook of Coal Petrography contains the results obtained by the lignite group (director: M. TEICHMÜLLER) and the analysis subcommittee (director: H. HAGEMANN) during the years 1971 to 1974 under the presidency of R. NOEL.

The 1st supplement to the 2nd edition (1971) contained for the first time definitions of lignite macerals of which only the most important were finished. Within the 2nd supplement the remaining lignite macerals (alginite, suberinite, bituminite, semifusinite, fusinite and macrinite) have been defined. Most numbers for the literature references refer to the "References related to lignite nomenclature" which were published in 1971 within the 1st supplement. Additional references are given in a supplementary reference-list ("References related to lignite nomenclature, 2nd supplement to the 2nd edition, sheets 13-16) presented here. For the time being this completes the work on the definition of lignite macerals.

The contribution of the analysis subcommittee concerns the relatively young and at the present time especially topical domain of qualitative and quantitative fluorescence microscopy of lignites and bituminous coals. The methods described on 22 pages may be of special interest not only for coal petrologists, but also for palynologists, geologists and geochemists who are concerned with oil exploration.

Responsible for editing the 2nd supplement to the 2nd edition are: B. ALPERN, N. BOSTICK, R. NOEL, W. SPACKMAN and M. TEICHMÜLLER.

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1975 Edition	International Commission for Coal Petrology Nomenclature Subcommittee	Lignite Reflected Light Transmitted Light
	ALGINITE * (Stopes-Heerlen System)	

I - ORIGIN OF THE TERM

The term alginite was first used by A. DRATH** in the sense used here. Since 1963, the International Commission for Coal Petrology has designated alginite as a maceral of the Liptinite (Exinite) Group, it being derived mostly from algae.

Etymology: alga (Latin) = sea grass, seaweed
Algae - a lower plant group containing many types and forms (Thallophyta)

Synonyms and analogous terms: Algal remains, algite (134)
alginite-tallomite (120)
Tallomo-alginite (135)

II - DESCRIPTION

A. Morphography

Alginite quite often displays the original structure of the algal colonies from which it is derived. There are three types:

1. Pila type: oval compact bodies that may consist of several hundred cells (= individuals) arranged like funnels;
- 2) Reinschia type: colonies constructed in the manner of the Recent Volvox spheres. Since these bodies are hollow, they are usually compressed during fossilization. They are easily mistaken for sporinite when viewed in sections prepared at right angles to the bedding. Note the small, cell openings which are always directed to the outside.
3. Cladicothallus type: Radiating colonies consisting of tubular, forked cells about 4-5 microns in length.

* Original language: German

** DRATH, A., Badania petrograficzne boghedu z kopalni Radzionkow, G'orny Slask. (Petrographic studies of bogheads from the Radzionkow Mine, Upper Silesia). Serv. Geol. de Pologne, Inst. Géol. de Pologne, Bull. 21, 1-82. Warszawa, 1939.

Alginite 2

The size of alginite masses varies between 60 and 600 microns (in their greatest dimension).

Oval liptinitic bodies without any structure are not classed as alginite.

B. Physical Properties

Color: Transmitted Light: yellowish-white, light yellow with a greenish tint, gold-yellow or orange-yellow. Color is dependent on degree of coalification.

Reflected light, bright field, oil immersion: black to dark, blue-gray, usually darker than the sporinite of the same coal. Primarily oxidized algae have yellow edges.

Reflected light, bright field, methylene iodide immersion: dark brown to black-brown.

Reflected light, fluorescence: white-yellow, green-yellow, lemon yellow, rarely yellow-brown to brown. The fluorescing colors of alginite are dependent on the degree of coalification.

Refractive Index: Alginite from the Moscow Basin: $n = 1.55$ (128).

Anisotropy: Alginite displays weak double refraction and is optically positive, therefore different in this regard from most liptinite macerals, which are optically negative (112).

Reflectivity: $R_0 = 0.03 - 0.13$ (150)

Hardness: $7.8 - 9.5 \text{ kg/mm}^2$ (150). Compared with huminite macerals, alginite displays a greater polishing resistance (grinding-hardness) and therefore a positive relief.

C. Chemical Composition and Chemical Properties

The oils of recent algae consist mainly of esters of glycerol with higher saturated fatty acids (usually palmitic and stearic acid) and unsaturated oleic acids. They are mixtures of triglycerides. The porphyrin systems of chlorophyll and the lipids and proteins of chloroplasts also play a role. In the Glanzbraunkohle stage and in the high volatile bituminous stage, these substances are converted into n-paraffins and isoprenes through polymerization.

(Lignite)

Alginite 3

Work of Millais and Murchison (146) with alginite concentrates (more than 90 vol. percent) from bogheads of Mooifontein (South Africa), Torbane Hill (Scotland) and Autun (France) have led to the following results: alginite spectra are characterized by strong absorption that is caused by non-aromatic hydrogen groups. There is only little evidence of aromatic compounds. Peaks at 750, 812 and 870 cm^{-1} suggest the presence of polynuclear condensed structures or simple benzene structure. An absorption of medium intensity between 1695 and 1725 cm^{-1} must be caused by carbonyl groups.

Compared with huminite macerals, alginite is rich in hydrogen.

	Boghead (146)	Autun (169)	Balkaschite (Recent) (170)	Coorongite (Recent) (165)
A. Example of a Proximate Analysis				
Water	0.5%	0.4%	1.8%	1.6%
Volatile Matter	67.7%	56.9%	94.2%	90.1%
Fixed Carbon	4.9%	7.7%		
Ash	26.9%	35.0%	4.0%	5.7%
B. Example of an Ultimate Analysis				
Carbon, %, daf*	84.3	84.1	73.8	79.7
Hydrogen, %, daf*	11.5	10.2	10.9	1.20
Sulfur, %, daf*	3.1	1.8	1.0	0.1
Oxygen, %, daf*		2.8	13.7	7.5
Nitrogen, %, daf*	1.1	1.1	0.6	0.7
Alginite concentration	> 90.0%	ca.70.0%	?	?

*Percentages based on dry, ash-free substance.

(Lignite)

Alginite 4

III - BOTANICAL AFFINITIES AND GENESIS

Alginite consists of colonies of lipid-containing algae, mainly recent genera of the Chlorophyceae (Botryococcaceae and related ones). The different forms have been described under the following names:

Ordovician - Gloeocapsomorpha prisca ZALESSKY

Lower Carboniferous - Pila karpinskii RENAULT

Cladiscothallus keppeni BERTRAND

Carboniferous/
Permian

- Pila bibractensis BERTRAND and RENAULT

- Reinschia australis BERTRAND and RENAULT

Permian - Tasmanites NEWTON

Jurassic - Pila liassica

A complete list of synonyms of the forms that are related to Botryococcus braunii KUTZING has been published by HARRIS (137).

Tasmanites deviates in structure from all other listed forms. It is similar to the recent alga Pachysphaera pelagica OSTENFELD.

IV - ALTERATION DURING COALIFICATION

During the Braunkohle stage, changes in the optical properties are mainly the color in transmitted light (from yellowish-white to orange-yellow) and the fluorescing color (a shift in the fluorescence spectrum to a longer wavelength, towards red). Reflecting capacity probably does not change (difficult to measure). In the bituminous coal stage, (30 - 20% volatile matter) the reflecting capacity gradually becomes similar to that of vitrinite. At the same time the positive relief disappears.

V - OCCURRENCE

Alginite can be found from the Ordovician to the present. Alginite is the characterizing component of gyttja and sapropelic rocks, occurring therefore in boghead coal and oil shale. It may be present in concentrations up to 80-90% by volume. However, alginite can also be found in humic coals, but in smaller amounts.

Alginite derived from algae of the Pila type and the Reinschia type is particularly prevalent in limnic swamp environments. Gloeocapsomorpha and Tasmanites occur in marine environments.

(Lignite)

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Alginite is known from many occurrences. It is particularly prevalent in the following rocks, where it is in the lignite, or peat, stage of coalification.

Kuckersite (Ordovician)

Marahunite (Tertiary)

Balkaschite (Recent)

Coorongite (Recent)

VI - PRACTICAL IMPORTANCE

A. Low-temperature carbonization

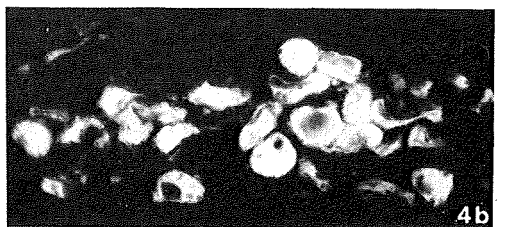
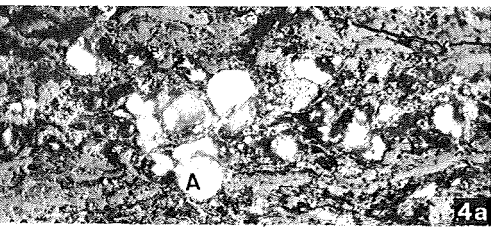
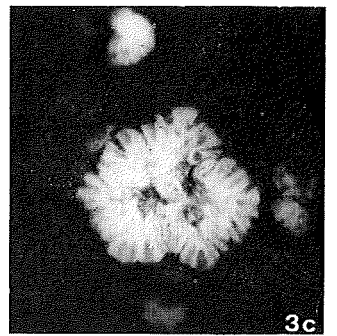
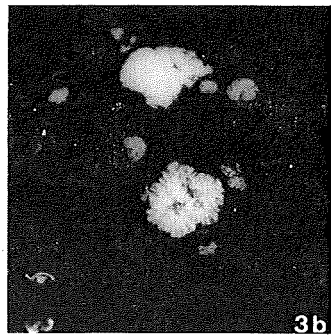
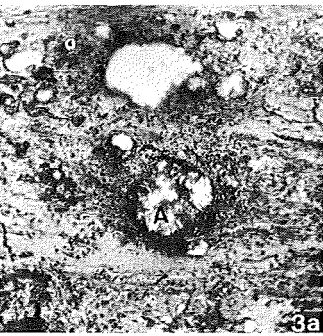
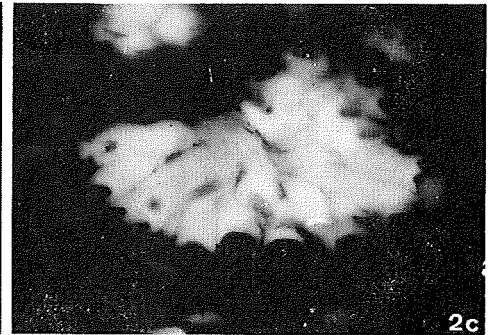
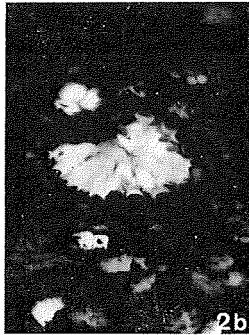
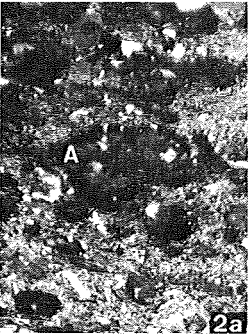
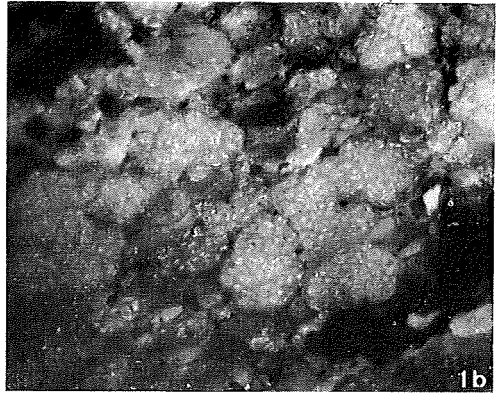
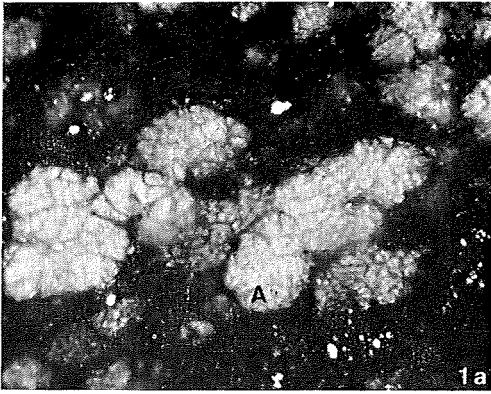
Alginite supplies high yields of tar and gas.

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- Fig. 1 Alginite (Gloeocapsomorpha) in Ordovician "carbonaceous shale" "Kuckersite" from Estonia (UdSSR).
1a. Reflected light-bright field, oil immersion, 180X
1b. Fluorescence (blue light excitation), reflected light, oil immersion, 180X
- Fig. 2 Alginite (Pila) in Lower Carboniferous lignite of the Moscow Basin (UdSSR).
2a. Reflected light-bright field, oil immersion, 180X
2b. Fluorescence (blue light excitation), reflected light, oil immersion, 180X
2c. Fluorescence (blue light excitation), reflected light, oil immersion, 400X
- Fig. 3 Alginite (Botryococcus) in Eocene lignite from Helmstedt, Lower Saxony (Western Germany).
3a. Reflected light-bright field, oil immersion, 200X
3b. Fluorescence (blue light excitation), reflected light, oil immersion, 200X
3c. Fluorescence (blue light excitation) reflected light, oil immersion, 400X
- Fig. 4 Alginite (of unknown botanical affinity) in Miocene lignite from the Lower Rhine Bay (Western Germany).
4a. Reflected light-bright field, oil immersion, 360X
4b. Fluorescence (blue light excitation), reflected light, oil immersion, 360X

A = Alginite

ALGINITE — ALGINIT



1975 Edition	International Commission for Coal Petrology Nomenclature Subcommittee	Lignite Reflected Light Transmitted Light
	BITUMINITE * (Stopes-Heerlen System)	

I - ORIGIN OF THE TERM

According to H. POTONIE (152), bituminite is "a general designation for a particular bitumen-containing rock, i.e. fossil sapropelite". SZADÉCZKY-KARDOSS (111) and MALAN (145) have used the term as a synonym for the maceral group liptinite. HAMRLA (136) called bituminite an amorphous mass that is found in the cracks and crevices of Tertiary lignite near RASA, Istria, Yugoslavia. M. TEICHMULLER (163) named the amorphous, hydrogen-rich, kerobitumen of bituminous rocks "bituminite". She later (164) extended the term to the amorphous liptinitic substances in coal (excluding exudates) (164). In 1975, the term bituminite was introduced by the International Commission of Coal Petrology for a maceral of the liptinite group that is distinct by virtue of its amorphous character. In contrast to the equally formless "exudatinite" (164), bituminite is autochthonous in origin, i.e. it has not migrated.

Etymology: Bitumen (Latin) = mineral pitch
asphalt

Synonyms and analogous terms:

Amorphous bitumen (in part) (113); fine detrital
polymerized bitumen (in part) (29, 35).

II - DESCRIPTION

A. Morphology

Bituminite has no specific form. It differs from the equally formless liptinite maceral "exudatinite" by virtue of its layer-bound autochthonous occurrence. In the bright lithotypes of low rank lignites, bituminite forms a fine-grained ground mass which

* Original Language: German

Bituminite 2

appears very dark in reflected light. It is also found as streaks of varying size and shape in other lignitic lithotypes. The fine granular appearance is particularly evident under the fluorescence microscope with blue light illumination. With high magnification, it can be seen that the granules are round to oval and have a diffuse, indistinctly delimited margin. Often they merge into one another. Their size varies from 2 to 4 microns.

B. Physical Properties

Color: Transmitted light: yellow-white

Reflected light, bright field, oil immersion: in low rank lignites, dark red-brown with red inner-reflection.

Reflected light, fluorescence: in low rank lignites, relatively weak pale brown, in part pale green fluorescence. For the same level of alteration, it displays distinctly less fluorescence than sporinite. Upon irradiation, the intensity of fluorescence increases more rapidly and to a greater degree than in the case of other liptinite macerals. Moreover, the fluorescence colors are displaced into the yellow range. All together, bituminite contrasts with the other liptinite macerals by displaying a greater degree of alteration.

Reflectivity: Very weak and for that reason not measurable. The light transmittance capacity is greater than most other liptinite macerals in lignites.

Remission: Bituminite-rich coals yield high remission values of 10% and more (up to 20%) (35, 106), (air dry sample, measured relative to $\text{BaSO}_4 = 100$, wave length 600 nm.)

Abrasive Hardness: Bituminite is distinctly softer than sporinite (see Fig. 2a). It is very difficult to polish since it smears in the polishing process.

Density: Probably very low, since bituminite-rich coals have an especially low specific gravity (153).

C. Chemical Composition and Chemical Properties

Pure bituminite was not investigated. From the behavior of lignite lithotypes extremely rich in bituminite, it can be concluded

that bituminite contains high percentages of hydrogen and carbon (106, 153) and will yield large amounts of volatile matter, extracted bitumens (benzol/alcohol) and tar.

The following average values are given by Preuss and Soos (153) for the bright layers of lignite from Lower Lausitz:

% Bitumen (benzol/alcohol) (d)*	11 - 19%	
% Tar (daf)**	12 - 36%	Ave. 26%
% C (daf)	69.5 - 71.6%	Ave. 70.8%
% H (daf)	5.9 - 7.4%	Ave. 6.6%
% O (daf)	20.8 - 25.4%	Ave. 22.3%

* dry

** dry, ash free

For bright coal used for low temperature carbonization from Upper Lausitz, JACOB (29) reports a bitumen content of 23%. Of this, 53% is Montan Wax and 47% Montan Resin. As reported by TEICHMULLER and THOMPSON (116) and JACOB (35), the huminite content of coal used for low temperature carbonization is extremely low and the exinite and resinite are not affected by the extraction process. Therefore, it can be assumed that the bituminite ground mass provides the major portion of the bitumen extract during the production of Montan Wax.

Work by PREUSS and SOOS (153) resulted in infra-red spectra indicating high contents of saturated hydrocarbons and COOH groups in bituminite-rich bright layers of the lignites of Lower Lausitz.

The moisture content of bituminite-rich bright lignite layers is always lower than that of the dark layers from the same seam.

III - BOTANICAL AFFINITIES AND GENESIS

Bituminite is a degradation product whose botanical origin can no longer be detected. According to JACOB (35), bituminite probably consists of a mixture of "humus" and bitumen that can no longer be differentiated microscopically; bitumen is believed to be derived from the metabolic and degradation products of microorganisms.

IV - ALTERATION DURING COALIFICATION

The behavior of bituminite during coalification has been very little investigated. According to TEICHMULLER (163, 164), petroleum-like products and micrinite are formed from bituminite during coalification through disproportionation.

V - OCCURRENCE

Bituminite is heavily concentrated in the bright lithotypes (bitumen-coal, bituminous-like normal-coal) of low rank lignites, especially in the deposits of Eocene "Weichbraunkohlen" used for low temperature carbonization in Germany. It is extremely concentrated in pyropissite. It is usually accompanied by relatively high proportions of liptodetrinite, sporinite, and resinite, for which it forms the groundmass in the bright lignite strata. There it is also found as the contents of root casings. Furthermore, bituminite is the characteristic maceral of sapropel coal, in which it also exists as the basic groundmass for other liptinite macerals (especially alginite) and the macerals of the inertinite group (e.g. in the boghead deposits of Lower Carboniferous lignite in the Moscow Basin). In the Tertiary coals of the Ukraine (132) and in the Jurassic coals of Tkibulskoie, Transkaukasien (131), bituminite is widely distributed.

VI - PRACTICAL IMPORTANCE

The technological properties of pure bituminite (which is practically impossible to isolate) have not been investigated. However, it is known from the behavior of bituminite-rich lithotypes that bituminite supplies large yields of extract (Montan Wax) in Montan Wax production and much tar during low temperature carbonization (see also II- C). During briquetting in an extrusion press, bituminite-rich lignite layers tend to flow (70).

Fig. 1 Fine-grained bituminite, seen in reflected light, to form a very dark ground mass for macerals displaying cell structure (sclerotinite, fusinite, corpohuminite, resinite, inertodetrinite, and liptodetrinite), from a bright layer of the Miocene lignite from Morschenich, Lower Rhine District (Western Germany).

Polished thin section, 145X

1a. Reflected light-bright field, oil immersion

1b. Transmitted light, oil immersion. The fine-grained texture is more readily discernible in transmitted light.

Fig. 2 Bituminite ground mass as major component of a bright layer of lignite. In the lower left side of the picture, a pollen grain occurs; in the lower right side a particle of humodetrinite is present. Eocene coal suitable for low temperature carbonization from Oberröblingen (Eastern Germany).

Polished section, 360X

2a. Reflected light-bright field, dry objective

2b. Reflected light-bright field, oil immersion

2c. Reflected light, fluorescence in blue light excitation, dry objective. Only in this photomicrograph are the numerous liptodetrinitic inclusions visible. The poorly defined image is caused by over-excitation.

Fig. 3 Fine-grained bituminitic ground mass with inclusions of sclerotinite and inertodetrinite (black) and resinite (light gray) in a bright layer of a Miocene lignite from Morschenich, Lower Rhine District (Western Germany).

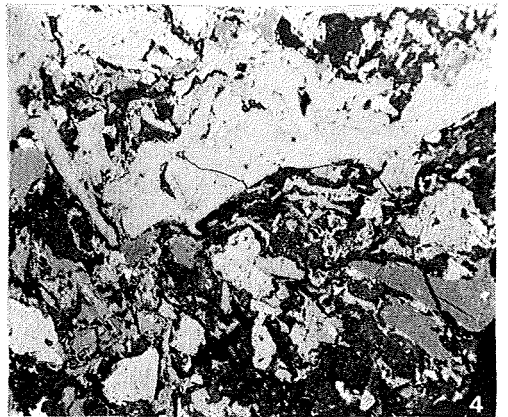
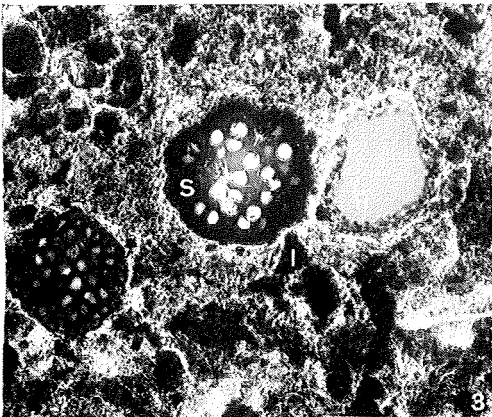
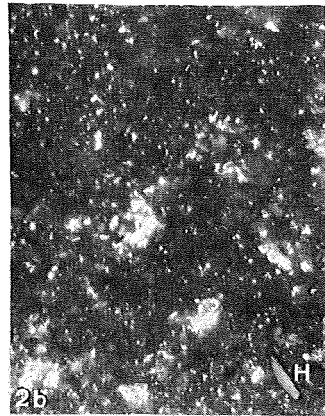
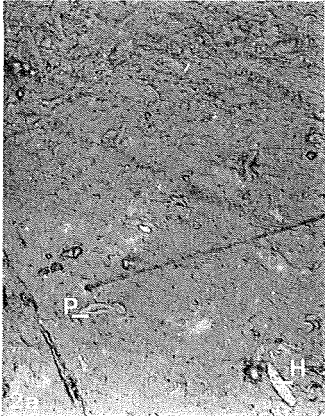
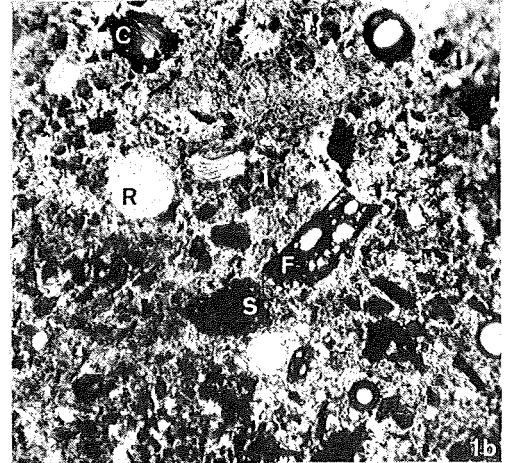
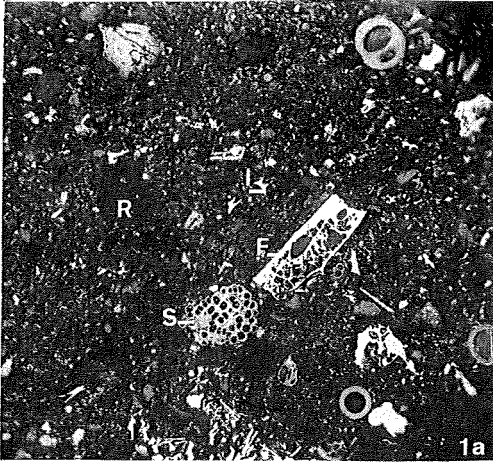
Polished thin section, transmitted light, dark field (Ultropak), oil immersion, 400X

Fig. 4 Fine-grained, dark-gray, bituminite as a ground mass in a durainic layer of a Miocene lignite from Handlova, Slovakia (Czechoslovakia). Polished section parallel to bedding, bright field, oil immersion, 360X

C = Corpohuminite
F = Fusinite
H = Humodetrinite

I = Inertodetrinite
P = Pollen grains (Sporinite)
R = Resinite
S = Sclerotinite

BITUMINITE — BITUMINIT



1975 Edition	International Commission for Coal Petrology Nomenclature Subcommittee	Lignite
	FUSINITE * (Stopes-Heerlen System)	Reflected Light Transmitted Light

I - ORIGIN OF THE TERM

The term fusinite was introduced in 1935 by M.C. STOPES** for an opaque coal constituent which displayed cell structure.

Etymology: Fusus (Latin) = spindle, fiber

Synonyms and analogous terms:

fusain (in part)(166); alpha, beta, gamma and delta fusinite (123); fusinoid-group (160); xylenofusain, xylenovitrenofusain (128); xylinito-fusinite, vitrinitofusinite (53); fusinite-telinite (119).

II - DESCRIPTION

A. Morphography

Fusinite consists of highly reflective cell walls which are usually thinner than the cell walls of the corresponding humotelinite and semifusinite. In lignite, fusinite frequently occurs as evenly constructed and well-preserved wood-cell tissue. Bogenstructure is common (see text on fusinite for bituminous coal). The cell cavities display varying sizes and forms, depending on the source material, the microbial destruction, and the orientation of the section. The cell lumina may be empty but occasionally may be filled with gelinite or mineral matter, e.g. clay minerals or pyrite (FeS_2).

Isolated fragments of cell walls with fusinite-like reflectivity are classified as inertodetrinite.

B. Physical Properties

Color: Transmitted light: black (opaque)

* Original Language: German

** STOPES, M.C.: On the petrology of banded bituminous coal. Fuel 14, 4-13, London, 1935.

Fusinite 2

Reflected light, bright field, oil immersion: white, often with a yellowish tint.

Reflected light, fluorescence: fusinite does not fluoresce.

Reflectivity: The reflectivity of fusinite is always relatively high, irrespective of the degree of coalification. Yellowish colored "pyro-fusinite" displays the highest values.

The following reflectivities are known for fusinite from lignites.*

Origin	Age	% R _o (546 nm)	Author
East Maritza, Bulgaria	Pliocene	} 2.23 - 4.74	(155)
Kjustendil, Bulgaria	Upper Miocene		(149)
Wallensen, West Germany	Pliocene	ca.1.5 - 3.0	JACOB ¹
USSR (several localities)		1.40 - 2.47	(154)

¹Oral communication

Anisotropy: In reflected light it is unknown and in transmitted light it is impossible to observe, since fusinite is opaque.

Remission: The remission capacity of fusinite is always relatively low. Fusinite from the Pliocene lignites of Wallensen, West Germany have a remission level of 4.4% (air-dry sample; measurement relative to MgO = 100, wave length 861 nm.). Fusinite from a high-moor peat near Langenheim, West Germany was measured under the same conditions and showed a remission of 3.5% (JACOB, oral communication). Fusinite from the Pliocene lignite deposits near East Maritza, Bulgaria display very low remission values of between 2 and 5% in the region of 400 - 1000 nm (SISKOV, oral communication). The spectral remission encountered is below that of semifusinite(158).

Density: Fusinite has a higher specific gravity than huminite from the same lignite. Fusinite from a Pliocene Bulgarian lignite (the East Maritza deposit) has, for example a density of $d_4^{20} = 1.47$ g/cm³ (157).

* The boundary between fusinite and semifusinite adopted by the various authors was more or less subjective.

(Lignite)

Fusinite 3

Density of fusinite from Jurassic lignites (130):

- 1) Kansk-Achinsk
(Siberia, USSR) 1.473 g/cm³
- 2) Orsk
(Urals, USSR) 1.640 g/cm³

Microhardness: The microhardness of fusinite varies over a wide range. It is dependent on the degree of fusinization (148). The abrasive hardness of fusinite is high and about equal to that of sclerotinite.

C. Chemical Composition and Chemical Properties

Fusinite is characterized chemically by a relatively high carbon content and low contents of hydrogen, oxygen and volatile components. Furthermore, it has developed a high level of aromaticity.

Concentrates of lignite - fusain can have the following composition:

Sample No.	1	2	3	4	5	6
% C (daf)*	85.7 - 91.7	70.89	73.12	82.75	76.95	74.3
% H (daf)	2.0 - 2.9	3.93	3.31	3.64	3.45	4.0
% O (daf)	3.5 - 8.1	N.D.	N.D.	13.03	N.D.	20.3
% N (daf)	0.8 - 2.2	0.60	0.78	0.43	0.60	0.9
% S (daf)	2.0 - 2.2	0.31	0.14	0.15	19.0 0+S	0.5
% water (af)**	6.0 - 7.8	8.89	7.83	4.9	9.6	N.D.
% ash (d)***	5.1 - 5.3	4.34	2.5	1.6	9.6	N.D.
% volatile components (daf)	11.0-18.4	39.1	33.0	27.8	28.8	38.2
% humic acid (daf)	N.D.****	N.D.	N.D.	3.0	5.0	N.D.

* daf = dry, ash-free

** af = ash-free, air-dried

*** d = dry

**** N.D. = not determined

also been found. A large portion of the fusinite in lignite, particularly the fusinite composing laterally extensive fusain horizons, is derived from forest and peat fires (fossil charcoal). Fusinite can also be created by decarboxylation with the aid of fungi and bacteria, but such "decomposition fusinites" are relatively rare in lignites (113). They usually have a semifusinitic level of reflectance.

Microscopy does not always determine with certainty the different origins of fusinite (e.g. from fires in moors, due to bacterial or fungal influence, or through complex chemical processes).

IV.- ALTERATION DURING COALIFICATION

With increasing coalification, the carbon content and reflecting capacity of fusinite also increase, while the percentages of hydrogen, oxygen, and volatile components decrease. Several authors believe that part of the fusinite found in bituminous coals has been formed during the coalification process from the textinite of lignite (143, 140, 164). This would explain the paucity of fusinite in many lignites and the abundance of fusinite in bituminous coal.

V - OCCURRENCE

Fusinite occurs in small amounts in most lignites. It occurs dispersed with attrinite and densinite. It occurs more abundantly in finely divided form in subaquatic facies types and in massive form in laterally extensive "fusite horizons" where it is found as fusain lenses. Fusinite is not restricted to specific seams or seam types. In the Miocene lignite of the Lausitz (East Germany) and of the Rhine area (West Germany) fusinite occasionally occurs as fossil charcoal *in situ* on very large stumps and on other xylites.

Fusinite-rich lignites have been found in the Eocene of North Dakota, the Cretaceous of Canada, the Tertiary formations of Southern India, as well as Kasachstan, Central Asia and, in lesser amounts, in Siberia. In a Jurassic lignite of Kasachstan, the fusinite content was 30 - 40% (168).

IV - PRACTICAL IMPORTANCE

a) Behavior during crushing

Fusinite concentrates in the finer particle sizes because of its friability.

b) Briquetting

During briquetting, fusinite stays inelastic and brittle. It does not cement with other petrographic components.

c) Coking and low-temperature carbonization

Fusinite behaves as an inert material.

d) Hydrogenation

Fusinite is not suitable for hydrogenation due to its relatively high carbon content and low hydrogen content.

e) Behavior with oxygen, spontaneous ignition

Fusinite is relatively difficult to oxidize. As far as is known, it does not tend to self-ignite.

f) Tendency to dust formation

Although fusinite concentrates in the portion of finest mesh size, its function in dust formation can be ignored, because it occurs only in small amounts in most soft coals.

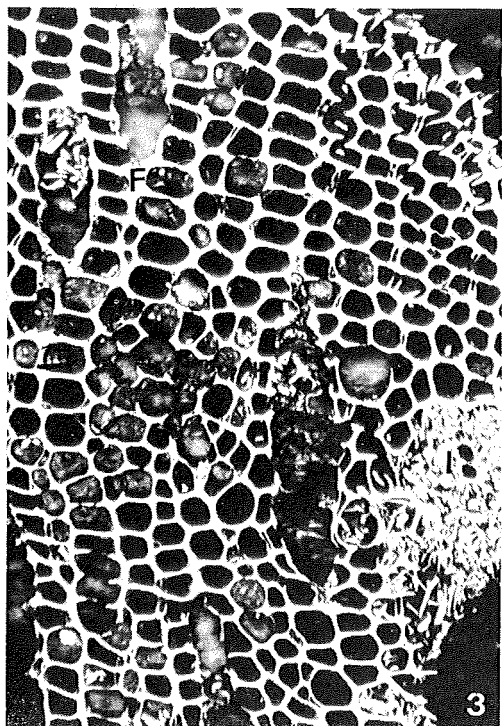
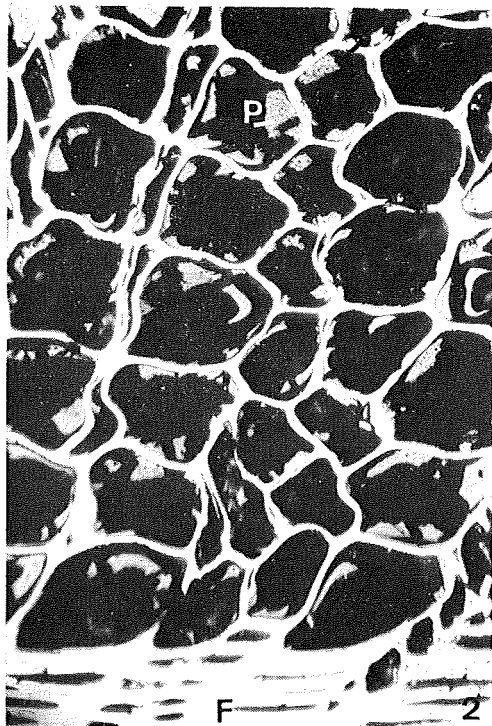
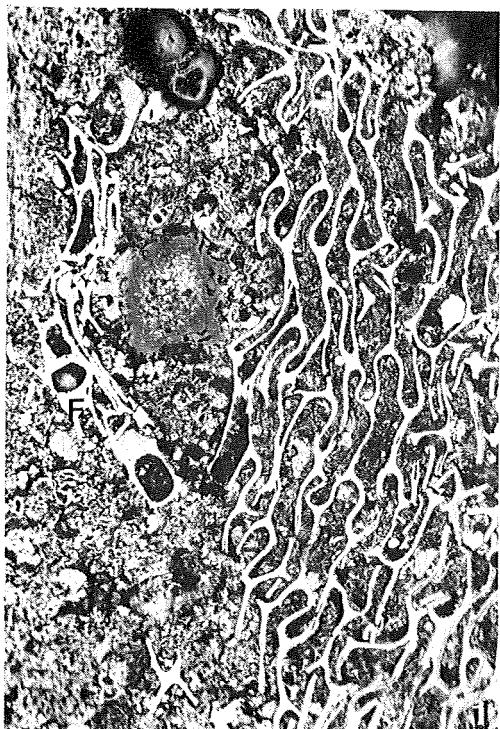
g) Stratigraphy

In large lignite seams, fusain bands occasionally stretch over large distances. In these cases, they can be utilized as key horizons for correlation purposes.

- Fig. 1 Fusinite (left) and transition fusinite/semifusinite (right) in Miocene lignite from the Lower Rhine Bay (Western Germany). Reflected light-bright field, oil immersion, 250X
- Fig. 2 Fusinite from springwood cells (above) and from summerwood cells (below) in Miocene lignite from the Lower Rhine Bay (Western Germany). Remains of porigelinite in the otherwise empty cell lumens. Reflected light-bright field, oil immersion, 250X
- Fig. 3 Fusinite with transition to inertodetrinite (right) in Miocene lignite from the Lower Rhine Bay (Western Germany). Reflected light-bright field, oil immersion, 250X
- Fig. 4 Fusinite in Jurassic lignite from **Kansko-Achinsk (Siberia, USSR)**
Transmitted light-bright field, dry optics, 45 X

F = Fusinite
I = Inertodetrinite
P = Porigelinite

FUSINITE — FUSINIT



1975 Edition	International Commission for Coal Petrology Nomenclature Subcommittee	Lignite
	MACRINITE * (Stopes-Heerlen System)	Reflected Light

I - ORIGIN OF THE TERM

The term macrinite was introduced in 1963 by E. STACH and B. ALPERN** for an inertinite-like maceral of bituminous coals. In 1975 it was accepted by the International Commission for Coal Petrology for an analogous maceral found in lignites.

Etymology: Makros (Greek) = large

Synonyms and analogous terms: semiopakdesminite and opakdesminite (146); bituminous coal-like particles (113); fusainodesminite (123); fusinit-collinit (119).

II - DESCRIPTION

A. Morphography

Macrinite is characterized by a more or less dense structure (small pore volume) and by its inertinite-like reflecting capacity. It occurs in various forms; as ground mass, which embeds the other macerals and, to a certain extent, cements with them, and as isolated particles in the huminitic ground-mass of the brown coals. The isolated macrinite particles have a definite outer form. For the most part, it consists of particles which are more or less round, about 30 to 400 microns (maximum value up to 2 mm) which are inclined toward check-formation (shrinkage cracks as a result of drying), and which contrast to the surrounding ground mass by having sharply defined, smooth grained borders. Generally, at the edges of the macrinite grains, the reflecting capacity is especially high. Morphographically, three maceral-types can be differentiated ("bituminous coal-like particles" of M. Teichmüller, 1950) -

* Original Language: German

** STACH, E. and ALPERN, B.: Inertodetrinit, Makrinit und Mikrinit. Fortschr. Geol. Rheinld. u Westf., 13, 2, 969-980, 1966.

telomacrinite, detromacrinite and gelomacrinite - according to whether they originated from humotelinite, humodetrinite or humocollinite (see Genesis). Detromacrinite often contains strongly reflecting material derived from liptinite. The original form and structure of the plant remains which make up these particles (tissue, detritus, gell, liptinite, etc.) are most recognizable in the soft brown coal stage (even without etching of the sections).

B. Physical Properties

Color: Transmitted light: Black

Reflected light, bright field, oil immersion: bright gray to white-yellowish, yellow.

Fluorescence: none

Reflectivity: The reflecting capacity can fluctuate between wide boundaries within a single bed. It is always higher than that of the corresponding humocollinite and can reach the same value as the fusinite (0.5 - 6.0% R_0 , determined at 546 nm.).

Microhardness: Relatively high abrasive hardness. In sections exhibiting relief, macrinite stands up in strong relief in distinction to the huminite, and relief increases with increasing reflecting capacity.

C. Chemical Composition and Chemical Properties

Macrinite in lignite shows, in contrast to huminite, a higher C-content (113, p. 456).

III - BOTANICAL AFFINITIES AND GENESIS

Macrinite occurring in isolation is distinguished in its botanical origin in no essential way from the huminitic material which surrounds it, excepting only that it is clearly more dense and possesses an inertinitic reflecting capacity. Therefore, the conclusion is reasonable, that it has been derived from strongly decomposed and gelified peat particles which have been secondarily fusinized and redeposited.

IV - ALTERATION DURING COALIFICATION

Presumably similar to that of other macerals of the inertinite group. Precise investigations are lacking.

(Lignite)

Macrinite 3

V - OCCURRENCE

In comparison with the Paleozoic bituminous coals, macrinite is extremely rare in the lignites of Europe and Australia. It occurs here predominantly as isolated particles in subaquatic sediments.

The macrinite which makes up the ground mass occurs predominantly in the Tertiary lignites of Middle and Eastern Europe.

VI - PRACTICAL SIGNIFICANCE

None, since brown coal macrinite is very rare.

MACRINITE — MACRINIT I

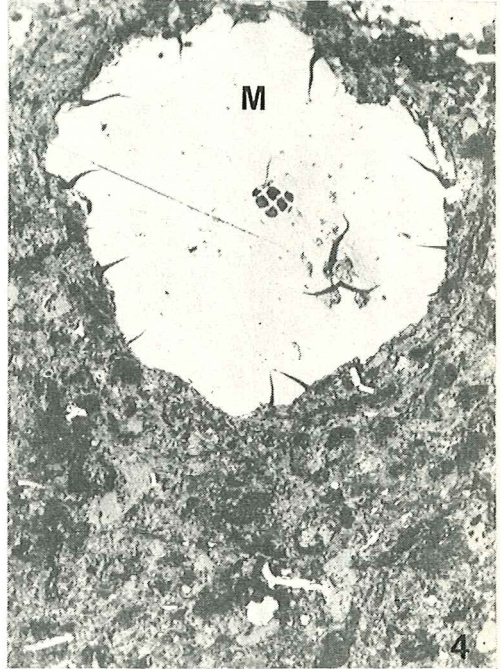
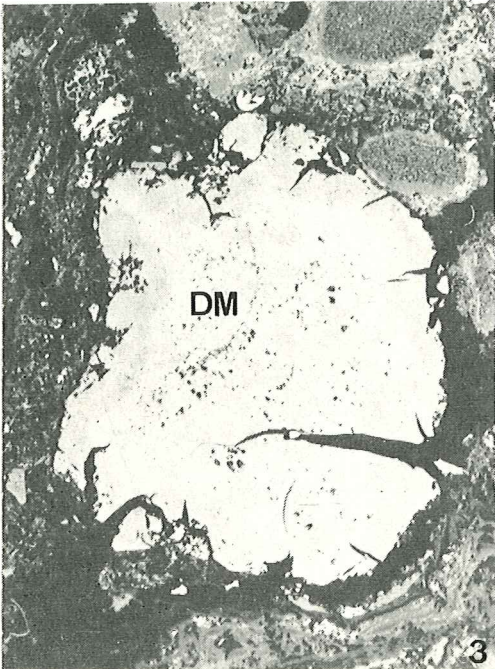
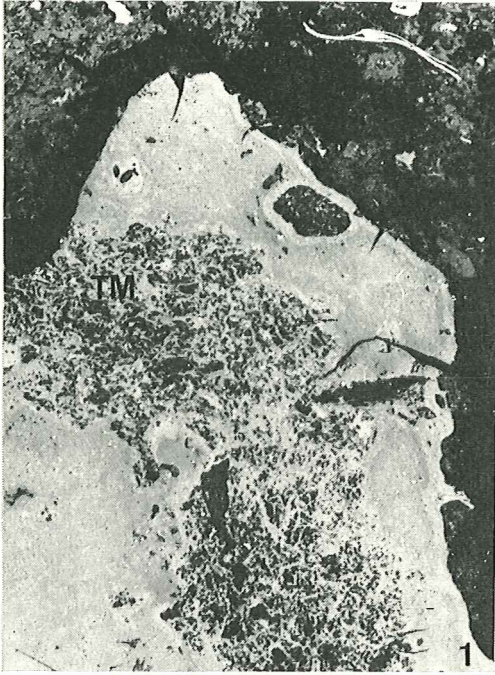


Plate 1

Fig. 1 Macrinite as the maceral variety texto-macrinite in Miocene lignite from the Lower Rhine District (Western Germany).
Reflected light, oil immersion, 500X

Figs. 2-4 Macrinite as the maceral variety detro-macrinite in Miocene lignite from the Lower Rhine District (Western Germany).
From Fig. 2 to Fig. 4 there is an increase in compaction and reflectivity.
Reflected light, oil immersion, 500X

M = Macrinite

DM = Detro-Macrinite

TM = Texto-Macrinite

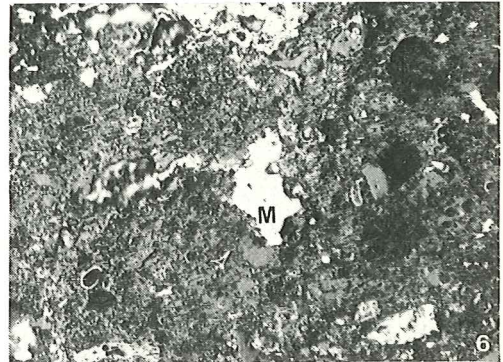
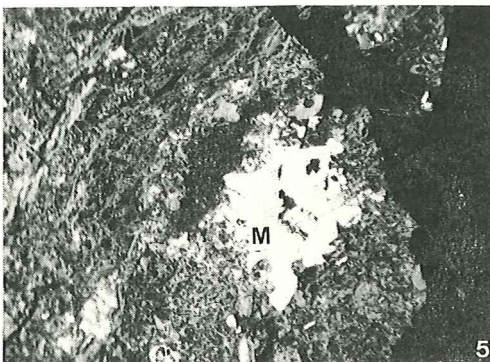
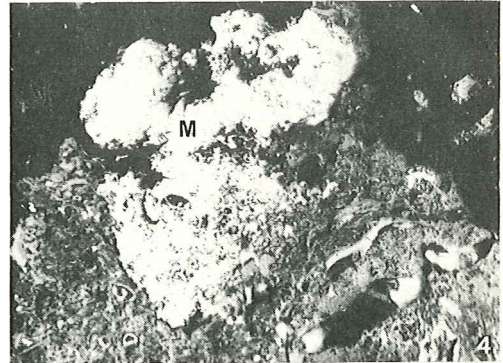
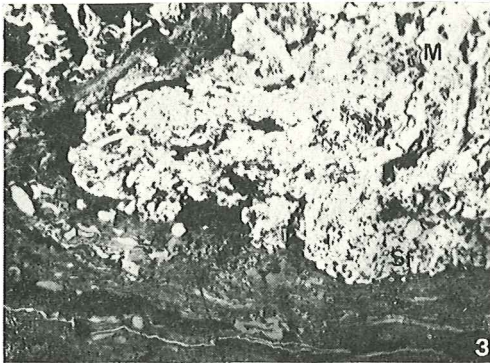
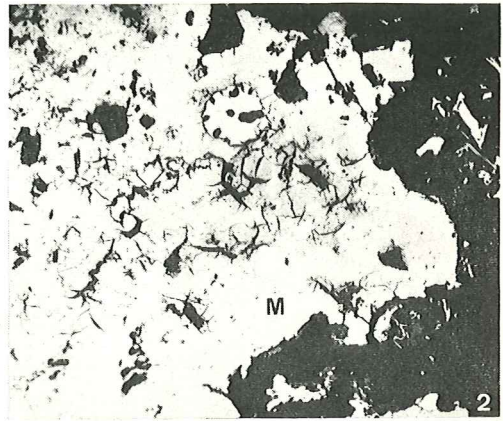
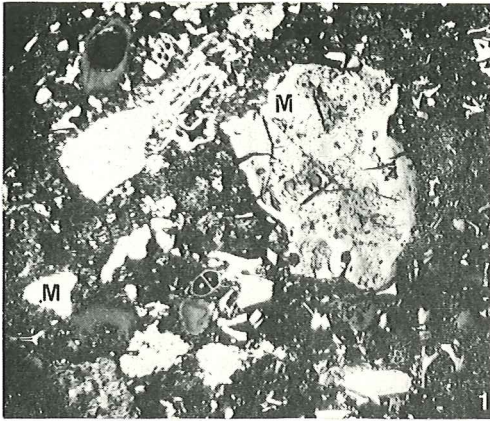
Plate 2

Macrinite 5

- Fig. 1 Various macrinite particles, among them a "bituminous coal-like particle" with typical formation of fissures, in Miocene lignite from the Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 2 Macrinite ("bituminous coal-like particle") with high reflectivity and typical formation of fissures in Miocene lignite from the Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 3 Macrinite with transition to "decay"-semifusinite in Pliocene lignite from Allershausen near Uslar/Lower Saxony (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 4 Macrinite developed from humodetrinite, in Miocene lignite from the Lower Rhine Bay (Western Germany).
Reflected light, oil immersion, 180X
- Fig. 5 Macrinite, compact particle in Miocene lignite from the Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 6 Macrinite, compact particle in Miocene lignite, from the Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 180X

M = Macrinite
Sf = Semifusinite

MACRINITE — MACRINIT II



1975 Edition	International Commission for Coal Petrology Nomenclature Subcommittee	Lignite Reflected Light Transmitted Light
	SEMIFUSINITE * (Stopes-Heerlen System)	

I - ORIGIN OF THE TERM

The term semifusinite was first used by JONGMANS et al (1938)** for a transition material between vitrinite and fusinite.

Etymology: Fusus (Latin) = spindle, fiber

Semi (Latin) = half

Synonyms and analogous terms: Xylain (13); vitrofusite (142); fusain (in part) (166); vitrofusite (90); alpha-, beta-, gamma-, and delta- semifusinite (123); semifusinoid group (160); semifusain, semixylofusain, semi-vitrenofusain (128); semixylinitofusinite (53); seminite (2); semifusinite-telinite (119); gelifusinito-telinite (119).

II - DESCRIPTION

A. Morphography

Semifusinite in lignite consists of cell walls whose reflecting capacity lies between that of the huminite and fusinite of the same coal. It therefore is a transition stage of textinite and textoulminite to fusinite. Semifusinite is formed of tissues with more or less well-preserved cellular structure. Cell walls of semifusinite are usually thicker than those of fusinite. The cell cavities are of different shapes and sizes (e.g., round, oval or elongated) depending on the source material, the microbial destruction, and the orientation of the section. Generally, they are

* Original Language: German

** JONGMANS, W.J., KOOPMANS, R.G., and G. ROOS: Nomenclature of coal petrology, Comptes Rendus 2nd Congr. Strat. Geol. Carbonif. Heerlen, 1935., Part.3, 1730-1737, Maastricht, 1938.

Semifusinite 2

smaller and less clearly demarcated than those of fusinite. They may be totally or partially filled with gelinite, oxidized resinite, phlobaphinite, clay minerals or pyrite (FeS_2). As opposed to fusinite, semifusinite displays ill-preserved cell structures. Transitions of semifusinite to fusinite occasionally appear in the same cellular tissue.

Isolated fragments of semifusinite-like reflecting cell walls are classified with inertodetrinite.

B. Physical Properties

Color: Transmitted light: red-brown to dark brown, also black (opaque) depending on the stage of fusinization and thickness of the section.

Reflected light, bright field, oil immersion: gray-white

Fluorescence: semifusinite does not fluoresce.

Reflectivity: Semifusinite in lignite has a stronger reflecting capacity than huminite and a weaker capacity than fusinite of the same coal. The following reflectance values of semifusinite from lignite are known:*

Origin	Age	% R_o (546 nm)	Author
East Maritza, Bulgaria	Pliocene	0.37 - 1.71	(155)
Kjustendil, Bulgaria	Upper Miocene		(149)
Wallensen, West Germany	Pliocene	0.3 - 2.9	JACOB (oral communication)
Various lignites, UdSSR		0.76 - 1.21	(154)

Anisotropy: Isotropic in transmitted light.

Remission: The remission capacity of semifusinite lies between that of gelinite and that of fusinite.

Semifusinite of Pliocene lignite from Wallensen, West Germany, for example, has a remission of 6.8% (air dry sample, measured relative to

* The boundary between semifusinite and fusinite followed by the various authors was more or less subjective.

Semifusinite 3

MgO = 100; wavelength of measurement 681 nm). Semifusinite of Pliocene Bulgarian lignite (East Maritza deposit) shows remission values of 2.5 - 7.5%, in the region 400 - 1000 nm. with a maximum of 10.0% at 612 nm (159). The remission spectrum of semifusinite is therefore between that of gelinite and that of fusinite.

Density: The density of semifusinite lies between that of gelinite and that of fusinite. In the Pliocene lignite of Bulgaria (open pit mine, East Maritza) it was $d_{4}^{20} = 1.35$ to 1.50 g/cm^3 (1957). Semifusinite from a Jurassic lignite from the Kanskó-Achinsk deposits (Siberia, USSR) has a density (specific gravity) of 1.504 g/cm^3 (130)

Micro-hardness: the micro-hardness of semifusinite is less than that of fusinite (148). The abrasive hardness and the relief of semifusinite in polished section are greater than those of gelinite and less than those of fusinite.

C. Chemical Composition

The chemical properties of semifusinite lie between those of huminite and fusinite of the same coal. The following analytical values from semifusinite concentrates are available:

Sample No.	1	2	3
	Elemental Analysis		
% C (daf)*	71.4 ± 2.6	76.17	74.82
% H (daf)	4.2 ± 0.3	3.92	3.69
% O (daf)	15.9 ± 1.9		
% N (daf)	1.4 ± 0.01	19.91	0.58
% S (daf)	6.6 ± 0.8		(O+S) 20.91
	Proximate Analysis		
% Moisture (af)**	10.2 ± 4.5	7.7	9.1
% Ash (mf)***	22.4	4.7	12.1
% Volatiles (daf)	45.3 ± 7.7	N.D.	44.4
% Alcohol-Benzene Extract (daf)	3.3 ± 1.9	N.D.	N.D.
% Humic acid (daf)	N.D.	N.D.	10.3

* Dry ash free
 ** Ash free
 *** Moisture free
 N.D. Not Determined

- 1) Pliocene lignite, East Maritza, Bulgaria (156)¹
- 2) Jurassic lignite from Kansko/Achins (Siberia) (130)¹
- 3) Jurassic lignite from Orsk (Urals) (130)¹

¹ Semifusinite identified through transmitted light studies.

Infra-red spectra of semifusinite from the East Maritza deposit in Bulgaria, shows that semifusinite contains fewer OH and C-O groups than the huminite in the same coal (158).

III - BOTANICAL AFFINITIES AND GENESIS

Semifusinite has its origin in the cellulose and lignin of plant cell walls. Causes of fusinization may be the following: fire (incomplete carbonification); effect of bacteria and fungi (decomposition fusinite); and other unknown causes. "Fire-semifusinite" is different from decomposition semifusinite in that it has relatively well-preserved cellular structures.

IV - ALTERATION DURING COALIFICATION

Since semifusinite in lignite is still quite similar to the huminite (see II C above), it changes more noticeably than fusinite with increasing coalification. Its reflecting capacity and its carbon content increase, while the percentages of hydrogen, oxygen and volatile constituents decrease.

V - OCCURRENCE

Semifusinite is found in small amounts in most lignites of all ages. It appears together with attrinite and densinite and sometimes forms local concentrations. In the thick Miocene lignites of the Lower Lausitz, East Germany and the Rhineland, West Germany, semifusinite can be found *in situ* on very large stumps and xylites. In this case it is usually associated with fusinite. The following are relatively rich in semifusinite: Pliocene lignites from Wallensen, West Germany; lignites from the Tertiary of India; the Eocene of South Dakota, USA; the Cretaceous of Canada; and the Jurassic lignites from Kasachstan and Central Asia. The Jurassic lignites from Kasachstan contain up

(Lignite)

Semifusinite 5

to 30-50% semifusinite (168). In lesser amounts, semifusinite is also found in the lignite deposits of Siberia. Semifusinite is not restricted to particular seams or seam types .

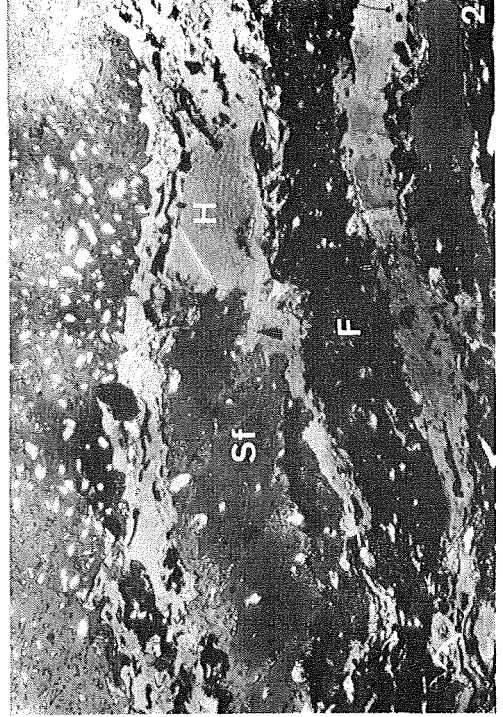
VI - PRACTICAL IMPORTANCE

The technological properties of semifusinite lie between those of huminite and those of fusinite, usually closer to those of fusinite. They have not been extensively studied. In briquetting, semifusinite does not bond well with other petrographic components, since semifusinite does not react plastically. In higher concentrations, semifusinite has detrimental effects on the quality of briquets (167).

- Fig. 1 Two semifusinite layers (center) in Pliocene lignite from the Mercure Basin, Calabria (Italy).
Reflected light-bright field, oil immersion, 250X
- Fig. 2 Semifusinite (dark grey) with huminite (grey) and fusinite (black), Jurassic lignite from the Kansk-Atschinsk basin (Siberia, USSR).
Transmitted light, bright field, dry optics, 150 x
- Fig. 3 Fragment of semifusinite (medium gray) in humodetrinite in Miocene lignite from the Lower Rhine Bay (Western Germany). On the left, textinite (dark gray), to the upper left, inertodetrinite (white).
Reflected light-bright field, oil immersion, 250X
- Fig. 4 Fragment of semifusinite in humodetrinite. Miocene lignite, Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 250X

F = Fusinite
H = Huminite
I = Inertodetrinite
Sf = Semifusinite
T = Textinite

SEMI-FUSINITE — SEMIFUSINIT



1975 Edition	International Commission for Coal Petrology Nomenclature Subcommittee	Lignite Reflected Light Transmitted Light
	SUBERINITE * (Stopes-Heerlen System)	

I - ORIGIN OF THE TERM

The term suberinite was introduced in 1935 by M.C. Stopes ** for coalified periderm. In 1975, the International Commission of Coal Petrology designated suberinite as a maceral of the Liptinite (Exinite) Group, having its origin in suberized cell walls.

Etymology: Suber (Latin) = cork

Synonyms and analogous terms:

α - suberinite, Δ - suberinite (123); subero-vitrinite (138); suberinito-telinite (120).

II - DESCRIPTION

A. Morphography

The term suberinite refers to coalified cell walls which differ from humotelinite by virtue of their leptinitic character. In polished section, suberinitic tissue; particularly cork tissue, displays a succession of more or less rectangular, brick-like, or irregularly polygonal 4 - 6 sided cells. The structural form depends to some extent on the orientation of the section. Suberinite often occurs together with phlobaphinite which is contained within the cork cells (48, 112). When the cells are empty and compressed, then the suberinite occurs as a laminar mass. The size of suberinite masses varies considerably. Those consisting of only a few cells have a thickness of about 50 microns. Sometimes a suberinite layer having a thickness greater than 500 microns can be seen. Fragmented suberinite that lacks recognizable structure is classed as lipto-detrinite.

* Original language - German

** STOPES, M.C., On the petrology of banded bituminous coal. Fuel 14, 4-13, London, 1935.

B. Physical Properties

Color: Transmitted light: light-yellow to gold yellow, red or brown; depends on section thickness, on degree of coalification (112) and on botanical origin.

Reflected light, bright field, oil immersion: almost black, dark gray, or medium gray; depends on degree of coalification.

Reflected light, fluorescence: u.v. excitation - light blue to green-yellow (37); blue light excitation - lemon yellow to yellow-orange and brown; depends on degree of coalification and botanical origin.

Refractive Index: Unknown

Reflectivity: Systematic studies are lacking because the thin cell walls make measurement difficult. A suberinite sample from a dark "Normalkohle", from Helmstedt, Lower Saxony (Eocene) has an R_0 of 0.08% (determined by L. Soos at 546 nm).

Anisotropy: Suberinite, in transmitted light, often shows weak double refraction. The optical characteristic is negative (112). Suberin-containing tissues that have been subjected to gelification or to partial fusinization during the early stages of coalification are isotropic and are not considered to be suberinite.

Hardness: Unknown.

Density: Unknown.

C. Chemical Composition

Suberinite originates from the suberin of cork tissue. Recent suberin, similar to cutin, is a polymer of very specific, high-molecular-weight, saturated and unsaturated oxy-fatty acid. There is no glycerol in suberin. A number of fatty acids from cork tissues have been isolated and characterized. These fatty acids contain 19 to 23 carbon atoms per molecule. They are mono- or divalent acids.

Analytical Data: Suberinite contains much hydrogen (112). Proximate and ultimate analyses are unknown.

Suberinite 3

Microchemical Behavior: Treatment with Schulze's solution ($\text{KClO}_3 + \text{HNO}_3$) produces the "ceric acid reaction"; small droplets are formed on the suberin lamellae. These droplets are soluble upon heating in alcohol or dilute potassium hydroxide (KOH) (64). Suberinite is not affected by concentrated chromic acid (H_2CrO_4) and concentrated sulfuric acid (H_2SO_4) at room temperature (64).

Solubility: Suberinite is not soluble in the common solvents - benzene, alcohol, gasoline, acetone (64).

III - BOTANICAL AFFINITIES AND GENESIS

Suberinite is formed from suberized cell walls, particularly in bark (periderm). Periderm is formed by the activity of a specific secondary meristem that is formed near the periphery of the plant organ that is growing in thickness; this is called the cork cambium (phellogen) (161). Layers of periderm, with very few exceptions, cover the older stalks, branches, stems, roots (48, 161), fruits and bulbs (143) of many plants.

Suberin lamellae are formed after injury in all spermatophytes (48) and to some extent in pteridophytes (139) as wound tissue.

IV - ALTERATION DURING COALIFICATION

Suberinite changes little during the first stages of coalification. In later stages (sub-bituminous and bituminous coal) drastic changes appear suddenly. Here suberinite is very similar to vitrinite with regard to optical properties (112).

V - OCCURRENCE

Suberinite occurs mostly in Tertiary coals. In zones of facies change, it is found only in small amounts. Suberinite is particularly common in coal derived from forest peats (37). There is no suberinite in Paleozoic coals.

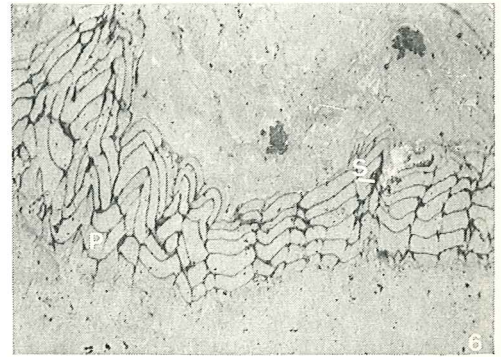
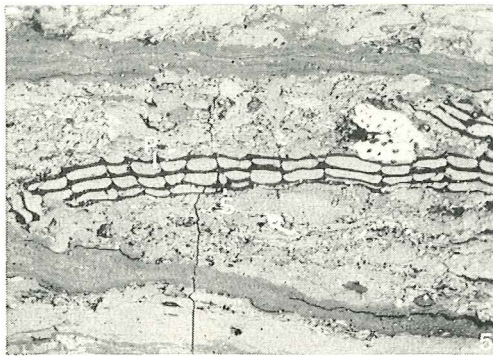
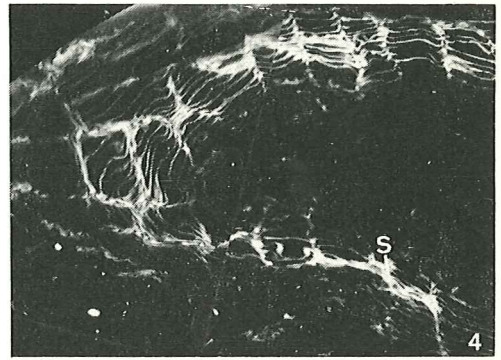
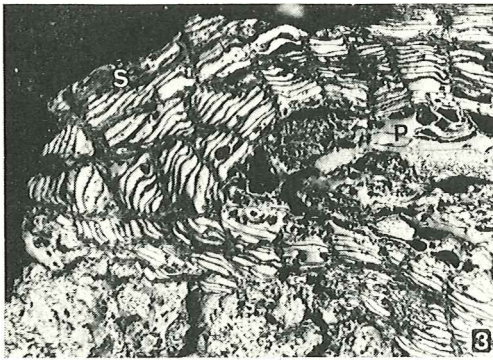
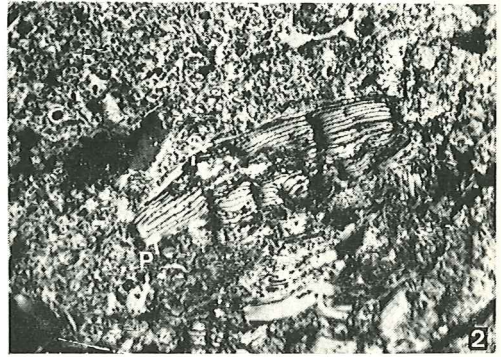
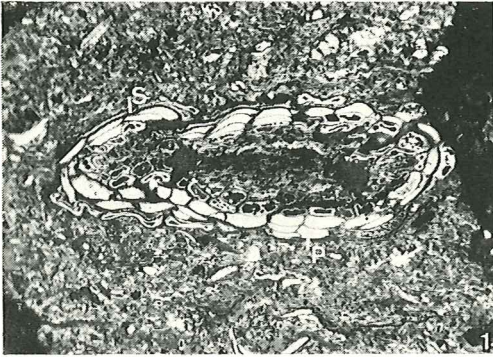
VI - PRACTICAL IMPORTANCE

Unknown, and also of little significance, since suberinite is found in only small amounts in all types of coal.

- Fig. 1 Suberinite and phlobaphinite in a transverse section of root bark in a Pleistocene lignite from Lieth/Holstein (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 2 Suberinite and phlobaphinite in a cork tissue in a Miocene lignite from the Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 3 Suberinite and phlobaphinite in a transverse section of root bark in a Miocene lignite from the Lower Rhine Bay (Western Germany).
Reflected light-bright field, oil immersion, 180X
- Fig. 4 Same as Fig. 3
Fluorescence (blue light excitation), reflected light, oil immersion, 180X
- Fig. 5 Suberinite and phlobaphinite in a cork tissue in Upper Miocene lignite from Trimmelkam/Upper Austria (Austria).
Reflected light-bright field, oil immersion, 360X
- Fig. 6 Suberinite and phlobaphinite in a cork tissue in Oligocene subbituminous coal from Hausham/Upper Bavaria (Western Germany).
Reflected light-bright field, oil immersion, 360X

S = Suberinite
P = Phlobaphinite

SUBERINITE — SUBERINIT



Edition	International Commission for Coal Petrology Analysis Subcommittee	Lignite Bituminous Coal
1975	FLUORESCENCE MICROSCOPY AND FLUORESCENCE PHOTOMETRY	Reflected Light

A. FLUORESCENCE MICROSCOPY

I. GENERAL

In contrast to bright-field microscopy, in which transmitted or reflected white light is used, fluorescence microscopy uses only a part of the light spectrum, the short wavelengths (blue to long-wave ultraviolet). This selected light causes certain inorganic and organic materials to glow. Only the organic matter of coals is considered here (see table 1); this restriction is significant in determining the preparation technique and the type of apparatus used.

Fluorescence intensity and ordinary reflectance are inversely proportional to each other (Jacob, 1969). With only very weak humification the fluorescence intensity of huminite can be as high as that of liptinite. Fluorescence microscopy is particularly important for study of lignite and peat, for only by this technique can many liptinite macerals be identified—especially fine detrital liptinite, which as a rule is abundant in bitumen-rich lithotypes. The use of fluorescence microscopy is also essential for study of low-rank bituminous coals (Amosov, 1956). In clastic sediments low-rank liptinitic material and low-rank huminitic material can be distinguished by use of fluorescence microscopy.

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Table 1. Fluorescence color and intensity for the different maceral groups at different coal ranks using blue-light excitation (excitation filter Schott BG-12,* 3 mm thick; barrier filter Schott OG-1**)

	Soft brown coal (lignite)	Hard brown coal (lignite)	Low-rank bituminous coal	High-rank bituminous coal
Liptinite***	Strong; green, yellow, orange, and brown	Strong to moderate; greenish yellow, yellow-orange, and brown	Strong to weak; yellow, orange, and brown	No fluorescence
Huminite or vitrinite	Strong to weak, yellow and brown; or no fluorescence	Very weak, brown; or no fluorescence	Very weak, brown; or no fluorescence	No fluorescence
Inertinite	No fluorescence	No fluorescence	No fluorescence	No fluorescence

*Broad-band filter with transmittance peak at about 400 nm.

**Edge filter with edge range from about 500 nm to 640 nm.

***Liptinite macerals differ in fluorescence. For details, see the individual Handbook sections on the liptinite macerals in lignites.

The fluorescence of organic materials in particular can change as a result of the illumination; this effect is called "Lichtätzung" (Haitinger, 1938; Jacob, 1964), "fading effect" (van Gijzel, 1971), or "alteration" (Ottenjann, Teichmüller, and Wolf, 1974). Changes of both the spectrum and the intensity are observed. The possibility of such changes should be kept in mind in all studies of fluorescence.

II. APPARATUS

Fluorescence attachments are built by most large optical firms, and they can be mounted on all research microscopes. In contrast to ultraviolet microscopy, fluorescence microscopy does not require quartz optics. Reflected-light microscopy is preferred to transmitted-light microscopy, for experience has shown that higher fluorescence intensity is reached with the former. It should be mentioned that thin sections can also be studied using fluorescence microscopy with reflected-

light excitation, for the excited objects are self-luminous and the cover glass interferes only slightly or not at all. However, the mounting cement must be fluorescence-free (Jacob, 1964). In the following discussion, only reflected-light fluorescence microscopy is considered.

The condenser arrangement is as important as the output and the flux density of the light source. If the incident light is not condensed adequately to a narrow beam, a large part of the peripheral rays is blocked and the light intensity is thus reduced. All diaphragms must be open (pay particular attention to the aperture diaphragm). Also ring apertures, half-stop apertures, or other accessories must not be inserted. If the vertical illuminator is provided with both a prism and a plane glass, the latter should be used; still better is use of a dichroic splitting mirror in place of the plane glass. A still greater fluorescence intensity can be obtained with a special apparatus, for example, with the Leitz fluorescence vertical illuminator (Ploem, 1969) or the Zeiss FL reflector. In addition, considerable intensity is gained by working without a polarizer. Sheet polarizers must in any case be removed, for the short wavelength radiation will destroy the fine polarizing crystals.

The larger the aperture of the objective, the greater is the fluorescence intensity. In addition, reflected-light objectives provide higher intensities than transmitted-light objectives. Objectives with a large aperture and magnification of about 20:1 to 60:1 provide good fluorescence intensity and resolution. Since the objects are self-illuminated, it does not matter whether dry or immersion objectives are used. Immersion objectives are preferred, however, when using alternately white-light and fluorescence observation. The immersion liquid should not be fluorescent; for this reason, cedar oil should not be used. For many years optical firms have generally provided immersion oils which are nearly fluorescence-free. Water or glycerin can be used with objectives of small aperture.

The 30 W to 100 W filament lamps normally used for coal microscopy are not suitable for fluorescence work because the light density is too low and the proportion of short-wave radiation is much too small; thus xenon or mercury vapor lamps are used for fluorescence microscopy.

The greatest part of the visible light must be removed by use of a violet or blue "excitation" filter; the filter thickness and fluorescence intensity are inversely proportional, but whatever type of filter is used, it must be thick enough to adequately absorb the unwanted radiation. Experience indicates that for coal microscopy blue glass filters (e.g., Schott BG-12 glass filter* with thickness of 2 to 4 mm) provide higher intensity than violet filters. A heat-absorbing filter should be inserted adjacent to the light source to protect the excitation filter and the object.

The excitation light reaches the object by means of the vertical illuminator and causes certain materials in the object to fluoresce. As a rule, however, the excitation light reflected from the object is more intense than the fluorescence; it interferes with observation and is damaging to the retina of the eye, and thus must be removed. For this purpose, light yellow glass is used with ultraviolet illumination and yellow-orange glass with blue illumination; these are the so-called barrier (suppression) filters (e.g., Schott OG-1). They can be mounted on the eyepiece or, better, with a proper holder, inserted in the compensator slot (Jacob, 1964).

In order to change rapidly from fluorescence to white-light observation, the mercury or xenon lamp should be mounted with the low-voltage filament light on a housing with a mirror to permit rapid light change. Thus, depending on the position of the deflecting mirror, either type of illumination is provided. It is advisable to work in a darkened room.

*A broad-band filter with transmittance peak at about 400 nm.

III. PREPARATION TECHNIQUES

If possible, fluorescence studies should be carried out on polished coal blocks without embedding. If briquettes of crushed coal are used, the size should be coarse enough that problems caused by fluorescence of the embedding material are minimized; this is particularly true when, in addition to fluorescence observation, quantitative measurements on the polished section are made. If a chemical treatment (maceration) is needed to enrich coaly matter in the sample, tests must be run to determine whether the treatment changes the fluorescence of individual macerals. The mounting medium should be nonfluorescent or very weakly fluorescent. Some of the common plastics can be used, such as polyester resin (e.g., Paladal, Castolite, or Jarrett 9101), methyl methacrylate (e.g., Acrilan, Degalan, Plexit, or Pleximon), or epoxy resin (e.g., Armstrong C-4, Araldit, or E.G.K.-19). Unfortunately, since the fluorescence is not a property of commercial importance, different batches of these industrial products have different fluorescence. The pure plastics are almost completely fluorescence-free under blue light. However, the commercial products often contain impurities that cause rather strong fluorescence. Thus each new supply of embedding medium must be checked to see whether it is fluorescence-free (Jacob, 1964).

The embedding medium should polymerize quickly (15 to 20 minutes); otherwise, fluorescent bitumens in some coals are partially dissolved by the monomer, causing fluorescent halos in the plastic.

There are no special techniques for grinding and polishing the samples. Of course, no fluorescent oils can be used as lubricants. The samples do not need to be polished as well for fluorescence microscopy as for bright-field microscopy.

Some macerals undergo positive or negative "fading" ("alteration," "Lichtätzung") as a result of short wavelength radiation. It is therefore

important to avoid exposing finished polished sections to bright daylight or to long excitation under the microscope (Schochardt, 1943, page 103; Jacob, 1964; Stach, 1969; and Alpern et al., 1972).

IV. FLUORESCENCE PHOTOGRAPHY

Most fluorescence photographs in black and white are very contrasty. In addition, only in rare cases can a picture as sharp as one in normal-incident light be obtained, because the fluorescing materials glow not only on the surface but also from within. Thus most black and white pictures are not satisfactory. Much better results are obtained with color photography; in addition, fluorescence colors have diagnostic value. No color correction is required, and daylight film is used (reversal or negative film). Additional filters are not needed for color photography. The exposure time depends on the fluorescence intensity of a particular object and on the "light intensity" of the entire apparatus. Even with modern apparatus, the range of exposure times is very large, from about 10 seconds to 30 minutes. For most liptinites in lignites and in low-rank bituminous coals, average exposure time of about 30 to 60 seconds can be assumed. The exposure time must be determined by means of a calibration curve or by an automatic exposure device. It is advisable to use sensitive or very sensitive film, for example, 18 DIN to 27 DIN or ASA 50 to ASA 400, in order to reduce "fading" ("alteration," "Lichtätzung") to a minimum.

B. MONOCHROMATIC FLUORESCENCE PHOTOMETRY

I. GENERAL

Monochromatic fluorescence photometry is particularly suitable for determination of the degree of humification of very low rank huminite. Fluorescence intensity and ordinary reflectance are inversely proportional to each other, although not linearly. As far as is known at present, measurable fluorescence

differences occur in natural solid organic matter (macerals and dispersed organic particles) only if the reflectance is less than about 0.3 percent (oil immersion, 546 nm wavelength); it follows that inertinite, vitrinite, and some huminite do not come under consideration for fluorescence study. Thus we are dealing with low-rank huminite, liptinite, and some of the solid petroleum bitumens.

In contrast to spectral fluorescence photometry, monochromatic photometry measures a specific wavelength of the emitted light (e.g., 546 nm \pm 20 nm). The apparatus and procedure are such that it is easy to combine monochromatic fluorescence photometry with reflectance photometry in a single working procedure (Jacob, 1973).

II. APPARATUS

The microscope equipment is the same as that described in the section "Fluorescence Microscopy." The illumination must be as strong and constant as possible; therefore, not all kinds of lamps are suitable. Direct current lamps are generally superior to alternating current lamps. Only high-pressure vapor lamps provide adequate light intensity and, especially, suitable spectral distribution even though they do not give as constant illumination as do filament lamps. In order to exclude additional instrument errors when using electronic photometers, it is necessary to stabilize the voltage (hence current) with suitable devices (Jacob, 1972).

To measure fluorescence and reflectance in combination, it is convenient to adapt a normal vertical illuminator that has both a prism and a plane glass by replacing the glass with a dichroic beam-splitting mirror, which provides a considerably brighter fluorescence.

Sporinite, suberinite, humotelenite, and the like are, for the most part, very small or thin macerals. As a consequence, the object spot sensed by the

photometer must be as small as possible (circa 1 to 3 μm in diameter). The excitation light must be centered with the sensed spot on the object. The total observed field may then be irregularly illuminated; however, in the part of the field measured, there will be a considerable intensity gain.

For monochromatic fluorescence photometry no special photometer is required. The same photometer and photomultiplier (e.g., EMI-6094) combination as used for reflection measurements is quite adequate (see the Handbook section "Determination of Rank by Reflectance Measurement of Vitrinite"). With blue-light excitation the fluorescent objects fluoresce green to orange. Therefore the photomultiplier used for reflectance photometry has a suitable sensitivity spectrum for fluorescence measurements. The greatest intensities are measured at a wavelength of 546 nm, and this wavelength can be used for combined fluorescence and reflectance photometry.

The way the measurements are recorded and evaluated depends largely on the number of measurements. With 50 to 100 points, manual recording from a galvanometer scale or digital indicator is adequate. With more than 100 measurements, electronic data processing (EDP) offers considerable advantage for data analysis and construction of histograms, either off-line by using punched paper tape or directly on-line with the photometer. The number of measurements required depends on the spread of values. With simple fluorescence distributions, about 50 measurements suffice. With complicated distributions, more than 100 values will be needed.

III. FLUORESCENCE STANDARDS

Unlike reflectance photometry, for which absolute standards are available, fluorescence photometry has no absolute standard at present. Therefore, relative standards must be used. Both liquid and solid standards have been used.

Sernetz and Thær (1970) proposed as standards fluorescein solution in quartz glass capillaries 6 to 8 μm in diameter. Different standard fluorescence intensity is obtained by using different capillary lengths or by diluting the fluorescein solution with a phosphate buffer at pH 7. These liquid standards must be renewed approximately every 2 months.

Jacob (1972 and 1973) used uranium oxide glass standards of different thickness (in the range of about 1 to 3 mm) and hence different intensity. Experience to date indicates that such standards have a stable fluorescence intensity (Sernetz and Thær, 1970; Jacob, 1972, 1973). The uranium glass standards are, however, independent of the objective aperture and the field diaphragm opening only if they are masked to a spot of about 10 μm . This small surface serves as a standard.*

Within a single laboratory it is thus possible to make reproducible fluorescence measurements with a relative standard. It is also possible to compare measurements from different laboratories if a particular individual standard is used to calibrate the fluorescence intensity of other relative standards.

C. SPECTRAL FLUORESCENCE PHOTOMETRY

I. GENERAL

Spectral fluorescence photometry is used for study of liptinite and huminite macerals in peats and lignites and of liptinite macerals in low-rank bituminous coals. The method will be described using sporinite as an example. Sporinite is particularly suitable for study of changes in rank. In contrast to other liptinite macerals, it is relatively resistant to brief primary chemical

*Humic tissues, humic detritus, and liptinite are generally so thin or small that they also are independent of aperture. This independence can be verified by opening the field diaphragm to check that fluorescence intensity does not change. To measure fluorescence of resinite, megasporites, or petroleum bitumens, it may be necessary to select fragments or edge segments.

action, occurs relatively commonly, and generally can be unequivocally recognized. Spores from coal seams as well as from other sedimentary rocks can be studied by this method.

Blue and long-wave ultraviolet light cause sporinite to fluoresce (see also Section A, "Fluorescence Microscopy"). Spectral fluorescence measurements are made using only ultraviolet excitation to permit measuring fluorescence at wavelengths as low as 400 nm.

The fluorescence emitted by sporinite has different color and intensity, depending on rank. Measurement of the absolute intensity of the entire spectrum is as yet not possible because of such problems as nonreproducibility of the excitation, lack of physically defined standards, and difference in size of the objects measured. Present indications are that the absolute intensity of the spectrum is probably in any case not as important for coalification studies as is the spectral composition of the fluorescence (van Gijzel, 1967, 1971, 1974; Ottenjann, Teichmüller, and Wolf, 1974). With the method described here, the relative intensities of various light wavelengths in the fluorescence are determined.

The polished sections are prepared in the usual way (see "Fluorescence Microscopy" and the Handbook section "Recommendations for Preparation of Polished Surfaces of Lump and Particulate Samples").

Because of the changes caused by prolonged excitation, prepared sections that are used for fluorescence observation cannot be used for fluorescence measurements unless they are first ground down and polished again. Likewise, reflectance measurements should not be made on polished sections that have been used for fluorescence measurement.

A minimum of 5 to 10 sporinite grains should be measured per sample. Angiosperm pollen in Mesozoic and Cenozoic coals and microspores in Paleozoic coals are usually suitable for measurement.

II. APPARATUS

The basic apparatus is the same as that used for monochromatic fluorescence photometry; a photomultiplier is mounted on a photo tube above a reflected-light microscope (see also the Handbook section "Determination of Rank by Reflectance Measurement of Vitrinite"). Some specific adaptations of the equipment are required for fluorescence photometry as described below (fig. 1).

Only a mercury vapor lamp with power supply can be used as the excitation lamp (1). This lamp provides the specific lines of the mercury spectrum. Attachment of a supplementary voltage regulator is recommended. By use of excitation filters (3) (or a monochromator), suitable intense mercury lines can be projected to the microscope for fluorescence excitation, for example, the lines at 365/366 nm transmitted by the excitation filter Schott UG-1.

Several filters of this type may be inserted in series to produce exciting light with a narrow half-peak band width if transmittance is adequate. This arrangement provides a sufficient separation of excitation and fluorescence at about 400 nm. A heat-absorbing filter (2) is inserted between the light and the excitation filters.

All objectives (6) with negligible internal fluorescence and large aperture are suitable for fluorescence work; examples are the Leitz oil-immersion objectives 22/0.65 or 54/0.95 or the dry objective Pl 40/0.65 or the Zeiss dry objective Epiplan 40/0.85. With the excitation source described, it is best to use a special fluorescence illuminator (5) that has a beam-splitting mirror (4) that provides a desired separation of excitation light and emitted light at about 400 nm (for example, the PLOEM-Opak of Leitz).

The measurement must be made with no barrier filter influencing the spectrum. Therefore it is desirable to provide an accessory plate with two positions, an open hole for measurement and a barrier filter (about 500 nm) to use while observing the specimen to position the object to be measured.

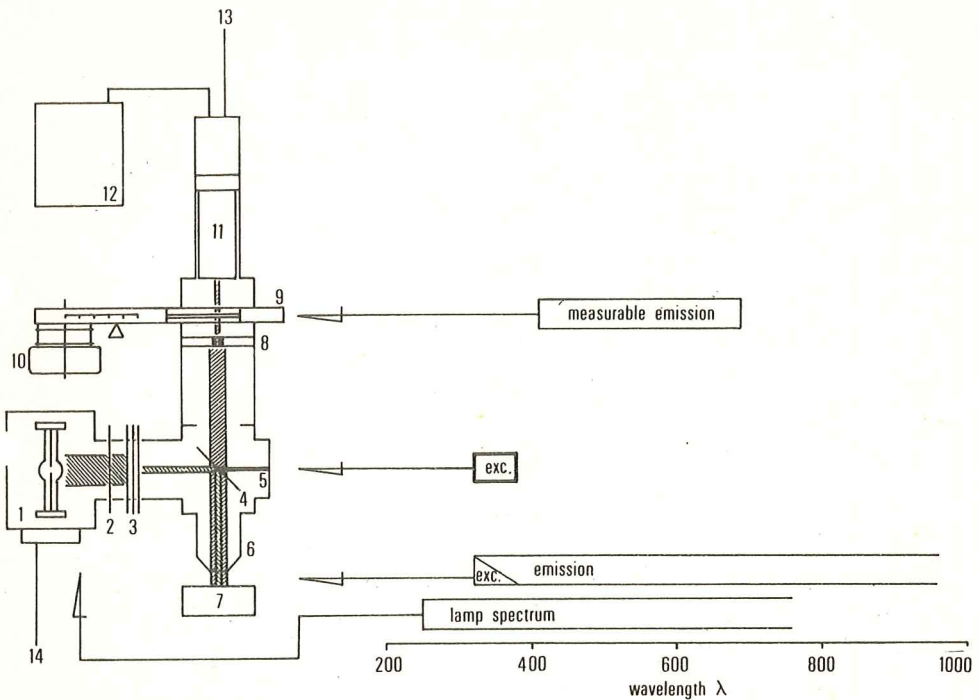


Figure 1 - Apparatus for spectral fluorescence photometry in reflected light using excitation filter Schott UG-1.*

1 - Lamp housing with 100 W high-pressure mercury lamp; 2 - Heat-absorbing filter; 3 - Excitation filters; 4 - Beam-splitting mirror; 5 - Reflected-light fluorescence illuminator; 6 - Objective; 7 - Polished section; 8 - Measurement aperture; 9 - Variable interference filter; 10 - Variable filter motor drive; 11 - Photo-multiplier; 12 - Chart recorder; 13 - Multiplier balance voltage; 14 - Lamp power; Exc. - Excitation range.

*A broad-band filter with transmittance maxima at about 360 nm and (outside the measured range) 760 nm.

The photomultiplier (11) must have adequate sensitivity to red light and must have minimum dark current and maximum general sensitivity (for example, EMI 9558A-S 20).

The spectral analysis of the fluorescent light is made with a sliding variable interference filter (interference wedge) (9). For example, the filters VERIL B 60 or S 60 (Schott) are suitable for measurements in the range of

400 to 700 nm. The variable interference filter is used in the system at the same position as that of the 525 or 546 nm filters used for reflectance measurement.

A motor drive for the interference filter (10), coordinated with a chart recorder (12), permits rapid measurements. Synchronous motor drives for both the filter and the recorder provide a constant relation between the two for each measurement.

An arbitrary fluorescence standard is used to permit comparison of the fluorescence intensity of different samples. An example is a uranium glass masked to a 10 μm diameter (see Section B, "Monochromatic Fluorescence Photometry").

III. ANALYSIS PROCEDURE

The following procedure is used to prepare the equipment for measurement:

- a. Position the instruments so that no exterior light can influence the measurements.
- b. Check that the interference filter and the chart recorder are coordinated. Check the stability of the photomultiplier and the high-voltage supply (see the Handbook section "Determination of Rank by Reflectance Measurement of Vitrinite").
- c. Check that the center of the measurement aperture agrees with the cross hair or other reference point in the eyepiece. Position the fluorescent light beam exactly with the measurement aperture. The size and shape of the measurement diaphragm may be selected arbitrarily if the photometer optics limit the light beam at the position of the interference filter to a maximum size of 1 mm (the maximum width of the beam is limited by the half-peak width of the filter used). However, the position of the measurement aperture must be held constant.

- d. To calibrate the wavelength of the continuous filter, use a narrow-band interference filter (e.g., 546 nm) with the microscope light. With this light source locate the position of the variable interference filter that gives a maximum photometer reading. This scale value corresponds to the τ_{\max} wavelength of the test filter used. Adjust the reference mark on the scale of the variable filter so that it corresponds to the value of the test illumination (see the manufacturer's instructions). With calibration by this procedure, take into account the microscope correction factors (see "Determining the correction factors" below).
- e. Check that the proper excitation filters (and also the barrier filter for observation!) have been inserted.
- f. Select several little-used objectives for the measurements; the correction factor will be determined for these objectives (see "Determining the correction factors" below).
- g. Center the lamp exactly; position the image of the light arc in the center of the measured field. Move the collating lens on the lamp housing until the photomultiplier indicates that a test object is receiving a maximum excitation intensity. Normally, at this position the lamp illuminates only a small part of the visual microscope field.
- h. Close the field aperture of the vertical illuminator until only the area to be measured is illuminated.

An elapsed-time counter to register the lamp use is suggested. The lamp intensity may decrease with time, and knowing the rate of decrease will help to interpret fluorescence intensity. Also the elapsed-time counter indicates when the lamp must be changed. A new lamp must burn 6 to 8 hours uninterrupted to minimize further decrease in intensity and to increase the lamp light.

The sequence of operations in a measurement is as follows:

- a. Study the polished section in normal bright-field illumination. Select the object and suitable magnification (use dry objectives when possible).
- b. Make a test measurement with a particle that corresponds to the particle to be measured in size and fluorescence properties. By this test, select a proper photometer sensitivity.
- c. Position the object to be measured under the center of the object spot of the photometer and focus the microscope (when possible, these operations should be done with normal bright-field illumination). On some apparatus, a separate photometer ocular is used to position the object. Using fluorescence, check the focus (remember to use the barrier filter!). Close any ocular on the photometer to eliminate stray light. To reduce changes caused by the excitation, use the high-intensity light as little as possible during adjustment.
- d. Measure the spectrum by using a synchronous filter drive and a chart recorder, or by moving the continuous filter manually and recording the photometer output in steps. With the synchronous drive method, it is useful to establish automatic start and stop positions on the filter scale.

In measuring fluorescence spectra, the measurement operation should be as quick as possible to reduce "fading" ("alteration," "Lichtätzung") to a minimum.

The recorded spectra are incorrect because of errors induced by the apparatus, and they must be corrected. The errors must be determined before beginning measurement. The measurements are composed of the background value (sum of the dark current, stray light, fluorescence of the microscope optics, and reflected excitation light) and the fluorescence intensity (incorrectly shown because of changing multiplier sensitivity as well as differing transmittance of the variable filter and of the microscope optics at different wavelengths).

Determining the background value: A nonfluorescent test object (inertinite, vitrinite in high-rank coal, or a microscope slide) is measured through the entire span of wavelengths. The adjustment of the excitation light, the photomultiplier balance voltage, and the apparatus must be the same as those used for the measurement of the sporinite (or other maceral). The background value must be deducted from every spectrum measured, for instance, by subtraction at specific wavelength steps. The background values are only approximate since they cannot be determined directly on the object of the study.

Determining the correction factors: The factors for photomultiplier sensitivity and for transmittance of the variable interference filter with light of different colors can be taken from data furnished by the manufacturers. The microscope correction factors are determined by comparing monochromator spectra measured directly with the same spectra measured through the microscope. The difference is the microscope correction factor.

IV. EVALUATION OF RAW DATA

Spectra measured digitally in steps can be evaluated directly. The record of a chart recorder must be transformed to a table of values. This transformation is done by direct measurement of the curve with the help of a transparent straight edge marked according to the calibrated values of the continuous filter. This assumes that the chart recorder speed and filter speed have a constant relationship (Ottenjann, Teichmüller, and Wolf, 1974).

The difference between measured value and background value can be measured from a chart recorder directly with a millimeter ruler and entered in a table (table 2). This difference, corrected by the correction factor on the table, yields the step values of the sporinite spectrum. Figure 2 shows the final corrected spectrum.

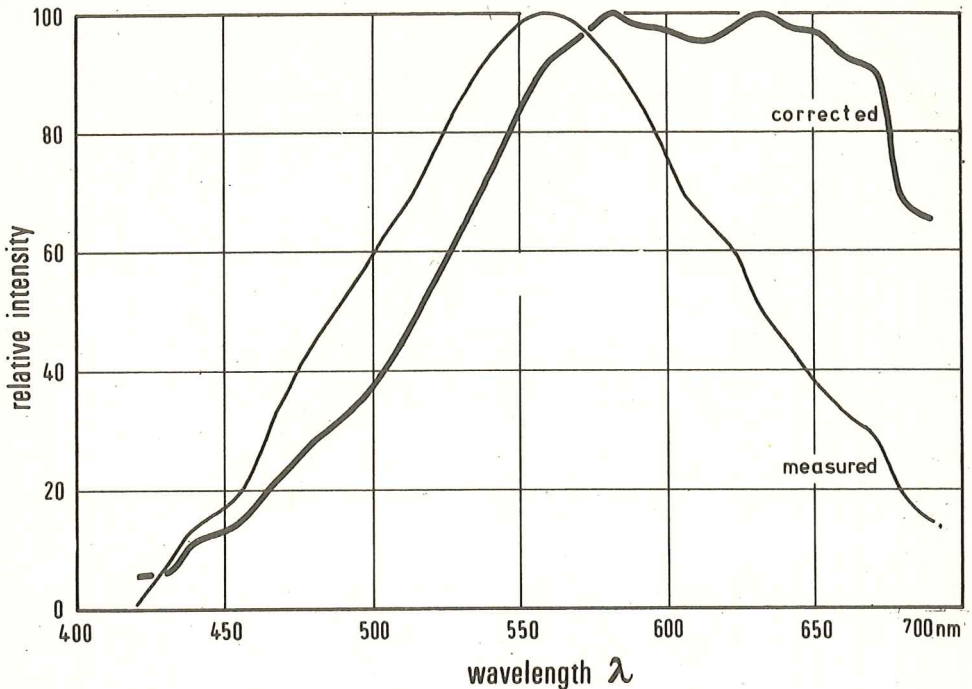


Figure 2 - Comparison of a corrected fluorescence spectrum with a curve of raw measured values.

If several measurements on comparable sporinite in a single sample can be made, an average spectrum can be calculated (Ottenjann, Teichmüller, and Wolf, 1974); this average serves as the foundation for determining various parameters of the spectrum. For statistical evaluation the numerical characteristics of each spectrum must be calculated from the individual curve (van Gijzel, 1967, 1971, in press). Examples of spectral characteristics that can be calculated are peak wavelengths (τ_{\max}), half-peak width, and one-third-peak width. Various ratios also can be calculated from the intensities of certain wavelengths (van Gijzel, 1967; Ottenjann, Teichmüller, and Wolf, 1974). Such parameters indicate the origin and nature of the materials studied (van Gijzel, 1963) and indicate, especially for sporinite, the rank (van Gijzel, 1967; Ottenjann, Teichmüller, and

Wolf, 1974). The parameters also allow comparison with other numerical data, for example, the various coalification parameters (Ottenjann, Teichmüller, and Wolf, 1974).

Table 2. An example of evaluation of a set of spectral fluorescence measurements (The correction values apply only to this case!)

(nm)	Measured value	Background value	Object value	Correction factor	Corrected value	$\frac{100}{\text{max. value}}$	Corrected relative intensity
410				7.00			
420	28	13	15	3.00	45		6
430	27	7	20	2.25	45		6
440	48	4	44	2.00	88		12
450	54	3	51	1.85	94		13
460	78	3	75	1.65	124		17
470	111	3	108	1.55	167		23
480	140	3	137	1.50	206		28
490	164	3	161	1.45	233		32
500	188	3	185	1.50	278		38
510	208	2	206	1.60	330		45
520	238	2	236	1.70	401		55
530	262	2	260	1.80	468		64
540	287	2	285	1.90	542		74
550	302	2	300	2.05	615		84
560	308	2	306	2.20	673		92
570	301	2	299	2.35	703		96
580	289	2	287	2.55	<u>732</u>		<u>100</u>
590	262	2	260	2.75	715		98
600	237	1	236	3.00	708		97
610	208	1	207	3.35	693		95
620	188	1	187	3.80	711		97
630	163	1	162	4.50	729		100
640	139	1	138	5.20	718		98
650	120	1	119	5.95	708		97
660	101	1	100	6.80	680		93
670	88	1	87	7.70	670		92
680	59	1	58	8.70	505		69
690	48	1	47	10.30	484		66

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