

ORGANIC PETROLOGY UNIT

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PRESENTATION OF THE METHODOLOGY ELABORATED FOR OPTICAL ANALYSES OF BOREHOLE SAMPLES FOR OIL PROSPECTION

by

**B. Alpern, in collaboration with M.J. Lemos de Sousa, H.J. Pinheiro,
M.M. Marques and X. Zhu**

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INTRODUCTION

The following introduction has been prepared in order to facilitate in the best possible way the understanding between the laboratory and the readers who are unfamiliar with coal and organic petrology. However, it is recommended that recent publications by the laboratory are consulted for further information. Several of the illustrations in this presentation were obtained from these publications.

The main characteristics of the methodology practised at Porto are:

- a) conventional microscopic equipment and accessories, and unsophisticated analytical procedures,
- b) a high concentration of the organic particles in the light fraction,
- c) systematic study of whole crude rocks embedded in resin, after mild crushing. This aspect should be emphasized because most laboratories only use organic concentrates and are therefore bound to loose fundamental information.
- d) Development of a totally original analytical method based on the fact that the epoxy embedding resin behaves as a chemical extractor, comparable to chloroform or dichloromethane. As a result, hydrocarbons (HC) captured and fixed in the epoxy resin are detected, analysed and treated as a normal constituent of the sedimentary rock.
- e) Solid training and experience of the team in the science of coal and organic petrology. Apart from being members of international institutions such as the ICCP and TSOP, they have also worked, over the past years, under the direction of Invited Professor Boris Alpern, former President of international organizations totally devoted to the science of Phytoclasts and Macerals (CIMP and ICCP).

GENERAL REMARKS CONCERNING THE METHODOLOGY AND APPARATUS

1. Sample preparation

The samples are crushed below 200 μ , then dispersed (by ultrasonic) in a mixture of bromoform and alcohol. A **Light fraction** (float at density <1.7) is obtained by centrifugation at 3000rpm.

The **light fraction** is concentrated on a Millipore screen, mounted and polished on a plexiglass slide, thereby permitting all microscopic examinations: transmitted light, reflected light, fluorescence. It is labelled LF.

Supplementing this normal procedure, the **Whole Rock** sample (labelled WR), after light crushing when necessary, is embedded and mounted in Epoxy resin, then polished. This type of sample permits the study of the relations between organic and mineral matter, and to detect oil source rocks and impregnated reservoirs when they occur.

2. Optical equipment

A summary of the optical equipment utilized for quantitative and qualitative petrologic analyses is indicated below, jointly with the routine quantitative parameters usually measured:

a) EQUIPMENT

- Reflected light microscope equipped with stabilised sodium halogen lamp for reflectance, and mercury lamp (HBO 100W) for ultra-violet and blue light fluorescence
- oil or water immersion objectives (30 to 50 x)
- monochromatic filters: 500, 546 and 650 nm
- Leitz MPV Combi Photometer
- microphotographic camera

b) MEASUREMENTS

- **Reflectance** of Vitrinite at 546nm, or of selected Migrabitumen,
- **Monochromatic fluorescence:**
 - Excitation : 365 nm (UG 1)
 - I_{546} : Fluorescence intensity of embedding resin at 546 nm
 - $Q_m (650/500)$: Ratio of intensities measured at 650 nm and at 500 nm. This parameter is measurable on selected Liptinites, on embedding resin (HC dissolution) and on automorph HC.

The fluo scale, applicable to hydrocarbons (HC), is based on I_{546} and Q_m usually measured on the embedding resin.

DIAGNOSIS OF ORGANIC PARTICLES

1- Whenever possible the rules and nomenclature of the ICCP for coal macerals are used (Table 1).

2- Nevertheless, in other sedimentary rocks the situation is far more complicated because particles are often found broken, small and dispersed in mineral matter. Moreover, many **organoclasts**, other than macerals, can be present including zooclasts and mainly secondary solid bitumen. Fig. 1 illustrates the difficulties encountered in the identification of micro-organic fragments present in sedimentary rocks. Even a very experienced petrologist is sometimes obliged to conclude a diagnosis as "hypothetical" or "doubtfull".

3- The **vitrite reflectance** can be affected by:

- meteoric oxidation at low temperature (increasing R),
- oxidation at high temperature (oxidation rims),
- thermal effects from hot waters or igneous intrusions (transitions from normal coal to semi-coke, cenospheres, etc.),
- irradiation by radioactive elements (increasing R).

When detected, these facts are always taken into account in rank evaluation, and reported in the descriptive sheets.

GENERAL REMARKS CONCERNING THE LIGHT FRACTION (LF)

1. Morphology and abundance

1.1. Many useful informations are reported on the individual descriptive sheets. Amongst these are the identification of **size** and **abundance** of the organic particles as represented by their respective scales. For example, it is clear that small-sized, not very abundant, often spherical particles are most probably allochthonous and detrital when compared to bigger and abundant coaly fragments.

1.2. The richness of a slide corresponds to the **total** number of organic particles seen in reflected light **plus** in fluorescent light. A sample very rich in fluorescence can thus correspond to a level with **no reflectogram**. Furthermore, it is clear that the evaluation (rich, poor, etc.) for LF is given after strong physical concentration, and is related to the **original** richness of the rocks. However, the concentration factor is **inversely proportional** to the initial organic content of the rock: low for coals and oil shales, high for source rocks, nul for sterile rocks. For rocks poor in organic matter, many constituents are **only** detected in LF. Such a concentration permits greater success in obtaining statistically better data, for example, reflectance measurements.

2. Fluorescence

2.1. Fluorescence of Liptinites

2.1.1. The fluorescence properties of sporinite and telalginite (Botryococci, Tasmanaceae, Acritarchs, etc.) are preferably used in quantitative analyses. The fluorescence of other constituents such as resinite (too many diverse families), cutinite, bituminite, etc., are more qualitative indicators, but quantitatively more difficult to use due to the absence of reliable rank scales.

2.1.2. It is not always possible to distinguish algae from spores, but generally the former are more green (in low rank stages), and far **more** sensible to oxidation.

2.1.3. A color descriptive vocabulary is used, related to qualitative **uncorrected** fluo observations (blue light excitation at $\lambda=390\text{nm}$). The quantitative fluo (ultraviolet excitation at $\lambda=365\text{nm}$) is more **precise** and **objective**, thus also providing parameters. However, the corrected color is not necessarily the observed one.

2.1.4. The only advantage of **qualitative** fluo color evaluation is that a selection by size and homogeneity is not necessary. Consequently, all liptodetrinite and algal fine laminae, sometimes the bulk of the organic matter, can be integrated in the color evaluation (even in fluo matrices), but they can hardly be used for quantitative measurements.

2.1.5. The **red shift** of fluorescence color of liptinites often marks the entrance in the oil window. Each maceral has its own oil-window, and sporinites (Fig.2) are probably the last ones to produce HC because of their high activation energy. Whenever possible and justified, the Q ratio (red/green quotient) measured on a selected liptinite population, is established.

2.1.6. The **blue shift** (Fig.2), produced by generation of HC in the oil-window, is not easily detected by qualitative observation, particularly when it is **superimposed** on the proper color of the liptinite, which is progressing towards the orange-red color. But it can be detected in the associated organo-mineral groundmasses.

2.1.7. The fading of I_{546} during 15' of UV excitation is also a tool employed in the evaluation of maturity. For Botryococci, it passes from strongly positive when immature, to slightly negative when mature. This fact can only be used when Botryococci are present in the whole sequence (Fig.3).

2.2. Fluorescence of Vitrinites

The vitrinite secondary fluorescence is a well recognized fact. It is related to HC generation and impregnation in the coal matrix prior to total compaction. The maximum percentage of fluorescent vitrinite is situated at about $R_v = 0.9\%$, and this value corresponds to the peak of extractable HC (refer to Figs. 4,5,6).

2.3. Fluorescence of Matrices

The fluorescence color of the organo-mineral matrices progresses from yellow-brown to brown and reddish before it totally disappears when the matrices are extracted (post oil-window). The fading curves of these matrices can sometimes be used for rank evaluation: they are positive for immature rocks, negative for mature ones (Fig.7). Normally such matrices are studied in the WR.

It is clear from the curves established by Ottenjann (1985), that the negative fading is mainly related to impregnated HC in organo-mineral matrices (Fig.8).

BITUMEN - MIGRABITUMEN

In marine series, vitrinite is often absent or allochthonous. Most of the organic particles correspond to migrated bitumen (migrabitumen). They can have diverse origins:

- **pre-oil** bitumens,
- **post-oil** bitumens related to migrated oils which were biodegraded, oxidized, thermally affected or desasphalted.

1. Problems with Bitumen terminology

The situation with regards to Bitumen terminology is very confusing.

1.1. Whilst geochemists consider Bitumen to be the **soluble** part of the organic matter, organic petrologists consider it as **solid-viscous migrated** HC. For this

reason petrologists have proposed "**Migrabitumen**" as the term that precisely implies a relation to migrated solid products with various solubilities.

1.2. The traditional classification is presented in Fig.9. It is clear that solubility and fusibility are not optical keys. A further point worth mentioning is the fact that, unlike "pyrocarbon" and "pyrolitic carbon" which normally imply pyrolysis by thermal effects (igneous intrusions, lavas, etc.), the term "**Pyrobitumen**" is herewith misused for maturation. For these reasons, and in order to avoid misinterpretations, the term Pyrobitumen has not been retained by Jacob in the proposed classification presented to the ICCP (Tab.2).

1.3. Nevertheless, since fluorescence and reflectance are the petrologic instruments par excellence (Fig.10), it is not possible to microscopically distinguish between the Gilsonite-Grahamite branch (highly soluble in CS₂) from the non-soluble Wurtzilite-Albertite one (Fig.11).

1.4. Consequently, this laboratory has adopted, for internal use, the terms Lipti-, Vitri-, and Fusibitumen for those particles which respectively exhibit optical properties similar to Liptinite, Vitrinite and Inertinite, and with which confusion is sometimes unavoidable.

1.5. A glance at the chemical properties of bitumen, as illustrated by van Krevelen diagram (Fig.12) and the triangular diagram of Cornelius (Fig.13), illustrate the fact that the number of bitumen categories is probably very high (as for oils) and, with the exception of Ozokerite (rich in Saturates), nearly all other categories are rich in Resins and Asphaltenes proving a frequent "desasphalting" process of deposition.

2. Valorization of Migrabitumens (MB) reflectance

It is an established fact that many oils and MB can co-exist in the same sample. Samples with the following characteristics have often been observed:

- fluorescing HC impregnations (automorph and dissolved),
- dark MB (Liptibitumen), some of them producing oil during UV excitation,
- various categories of grey and white MB (Vitri- and Fusibitumens).

Consequently, the problem revolves around selecting the **appropriate** population for rank measurement, ie., the one that has **suffered the thermal history** of the host sediment - this is not an easy exercise, and its success and accuracy are dependent on the petrologist's experience.

Nevertheless, some relations between vitrinite and MB reflectances have been published. According to Jacob (Fig.14) it is recommended that the lowest R population be selected; the following corresponding equation is: $R_v = 0.618 \cdot R_B + 0.40$. However, this may not always be the case, particularly when asphaltic residues have been deposited later, after light oil migration. Riediger (1993) has recently proposed a new equation for MB with reflectances higher than 0.52% (Fig.15): $R_v = 0.277 \cdot R_B + 0.57$.

It is particularly interesting to check the mode of occurrence of MB in the whole rock sections.

GENERAL REMARKS CONCERNING WHOLE ROCK GRANULAR POLISHED BLOCKS WITH PARTICULAR REFERENCE TO THE DETECTION OF HYDROCARBONS

Two kinds of HC are detectable in granular polished blocks examined under fluorescence (Tab.3):

- automorph HC, corresponding to drops, films, networks and non-spherical HC (Pl. I and II);
- dissolved HC in Epoxy embedding resin.

The main fact is that the fluorescence properties of HC are **transferred** to the resin with slight alterations. Two parameters are established in UV light (365nm): I_{546} and Q (650/500).

The parameter Q, measured on free oil on slide and on oils mixed with embedding resin, is related to the oil geochemical composition, and mainly to the Saturates/Aromatics + Heavy Products (Figs. 16 and 17). These curves are not used to predict but to estimate the nature and quantity (relation with S_1) of the HC dissolved in the resin.

When the chemical nature of HC is **constant** throughout a borehole, the I_{546} values measured on the embedding resin are sufficient to detect the place of the main source rocks, and the correlation with S_1 is good (Fig.18). When the situation is more complex, such as when heavy oils are trapped in source rocks and lighter oils have migrated to reservoirs, both I_{546} and Q values must be introduced in the abacus (Fig.19).

1. Recognition of source rocks and Reservoirs

The observations used to distinguish between source rocks and reservoirs are mainly related to the **morphography** of the rock and of the automorph HC. **Source rocks** are more often laminated; their organo-mineral matrices are related either to shales or to carbonates; in which drops and films are significant manifestations.

Reservoirs are related to clastic material which, after crushing, liberate intercrystal non-spherical HC and networks (refer to Pl.II - photo 4).

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Table 1 - The International Stopes-Heerlen Nomenclature relating the macerals of Lignites and Hard Coals
 (International Handbook of Coal Petrography 1963, *ibid.*, suppl. 1971).

SUBMACERAL	MACERAL	SUB-GROUP	GROUP	GROUP	MACERAL	SUBMACERAL
TEXTO-ULMINITE EU-ULMINITE	TEXTINITE ULMINITE	HUMOTELINITE			TELINITE	TELINITE 1 TELINITE 2
PORIGELINITE LEVIGELINITE PHLOBAPHINITE PSEUDOPHLOBAPHINITE	GELINITE CORPOHUMINITE	HUMOCOLLINITE	HUMINITE	VITRINITE	COLLINITE	TELOCOLLINITE DESMOCOLLINITE GELOCOLLINITE CORPOCOLLINITE
	ATTRINITE DENSINITE	HUMODETRINITE			VITRODETRINITE	
	SPORINITE CUTINITE RESINITE SUBERINITE ALGINITE LIPTODETRINITE CHLOROPHILLINITE BITUMINITE FLUORINITE EXSUDATINITE		LIPTINITE	LIPTINITE (EXINITE)	SPORINITE CUTINITE RESINITE SUBERINITE ALGINITE LIPTODETRINITE CHLOROPHILLINITE BITUMINITE FLUORINITE EXSUDATINITE	
	FUSINITE SEMIFUSINITE MACRINITE SCLEROTINITE INERTODETRINITE		INERTINITE	INERTINITE	FUSINITE SEMIFUSINITE MACRINITE SCLEROTINITE INERTODETRINITE	PYROFUSINITE DEGRADOFUSINITE
LIGNITES				HARD COALS		

Table 2 - Classification and properties of Migrabituemen (Alpern et al. 1992, after Jacob, 1989).

	Random Reflectance (oil) %	Fluorescence (special masked uranyl glass standard = 1 %)	Micro-solubility in immersion oil and petroleum ether	Micro-flowpoint °C
ozocerite	< 0.01 - ca. 0.02	ca. 9.0 - > 50	soluble	ca. 30 - ca. 90
wurtzilite albertite	< 0.01 - ca. 0.1 ca. 0.1 - ca. 0.7	ca. 0.1 - > 2.0 ≤ 0.1	insoluble insoluble	does not flow does not flow
asphalt gilsonite glance pitch grahamite	ca. 0.02 - ca. 0.07 ca. 0.07 - ca. 0.11 ca. 0.11 - ca. 0.3 ca. 0.3 - ca. 0.7	ca. 0.4 - > 4.0 ca. 0.05 - ca. 0.4 ca. 0.05 - ca. 0.2 ≤ 0.05	soluble soluble soluble slightly soluble or insoluble	< 104 ca. 104 - ca. 164 ca. 104 - ca. 164 > 164 - ca. 287
epi-impsonite meso-impsonite cata-impsonite	ca. 0.7 - 2.0 2.0 - 3.5 3.5 - ca. 10	≤ 0.02 < 0.01 < 0.01	insoluble insoluble insoluble	does not flow does not flow does not flow

FIGURES

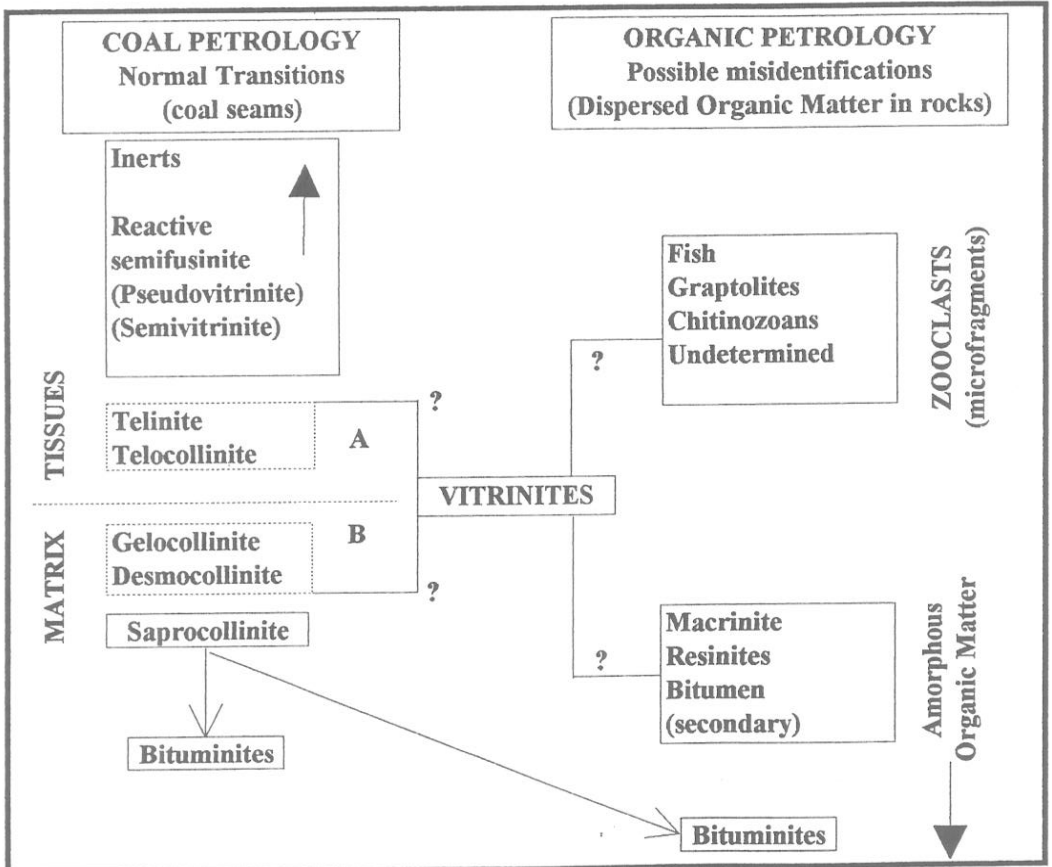


Figure 1 - Difficulties of vitrinite identification in coal seams and in rocks. (Alpern, 1987)

Table 3 - Classification of Liquid Hydrocarbons (HC) and Migrabitumen detected in polished granular whole rocks. (Alpern et al., 1993 in press).

Hydrocarbons (HC) embedded granular polished blocks	LIQUID HC	AUTOMORPH HC	drops films networks non-spherical	Conventional Terms	
	SOLID HC	Dissolved (in resin) HC		Asphaltite, Ozocerite	
		LIPTIBITUMEN R < 0.3%	fluo	Wurtzilite *, Gilsonite	
	Migrabitumen (MB)	VITRIBITUMEN 0.3% < R ≤ 0.7%	non-fluo	Glance pitch, Albertite (part)*	
		FUSIBITUMEN R > 0.7%	fluo	Grahamite	
			non-fluo	Albertite (part)*	
			isotropic	Impsonite*	
			anisotropic	Anthraxolite*	
		PYROBITUMEN	coke anisotropic spherobitumen		

* Non-soluble MB

NB: Spherobitumen containing radioactive inclusions are not included in this classification.

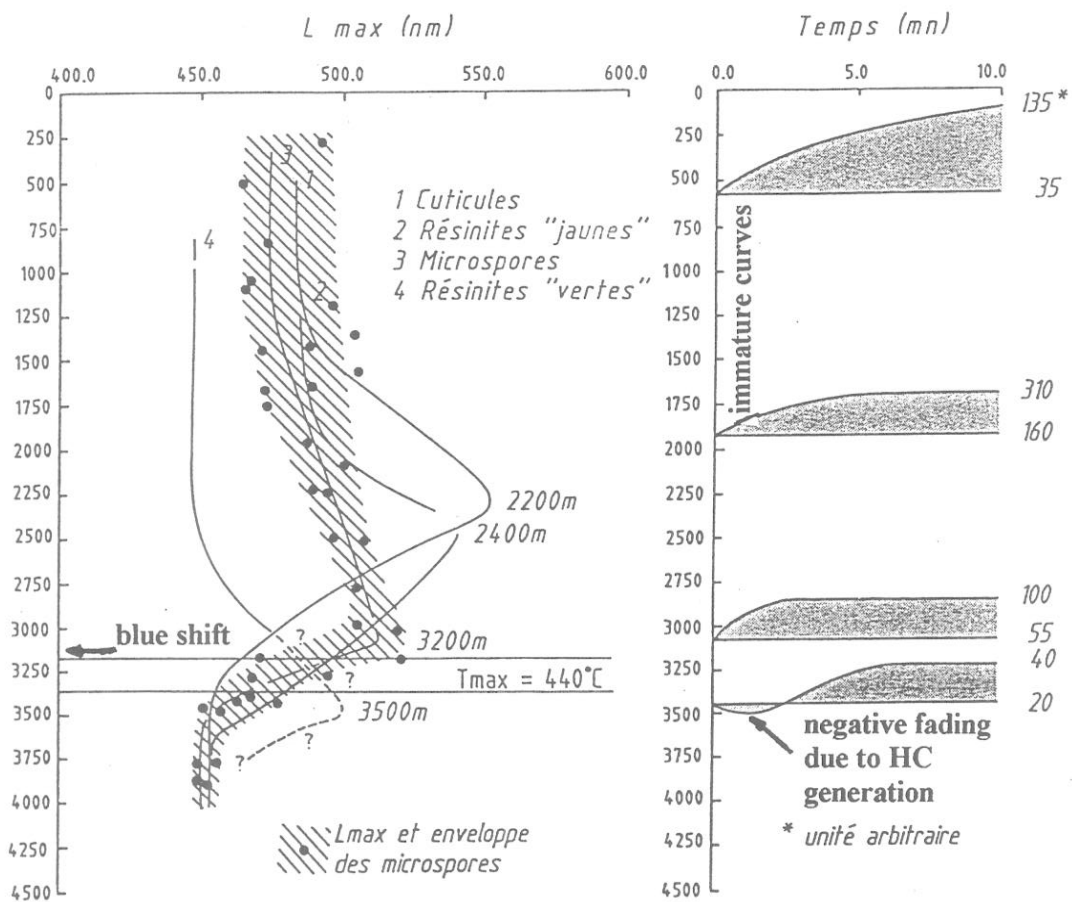


Figure 2 - Example of blue shift of various lipinite fluorescence (QF) in the Mahakam delta. The monochromatic fading ($10'$) varies from positive (immature) to negative then stable intensity of I546 (Pradier et al., 1987).

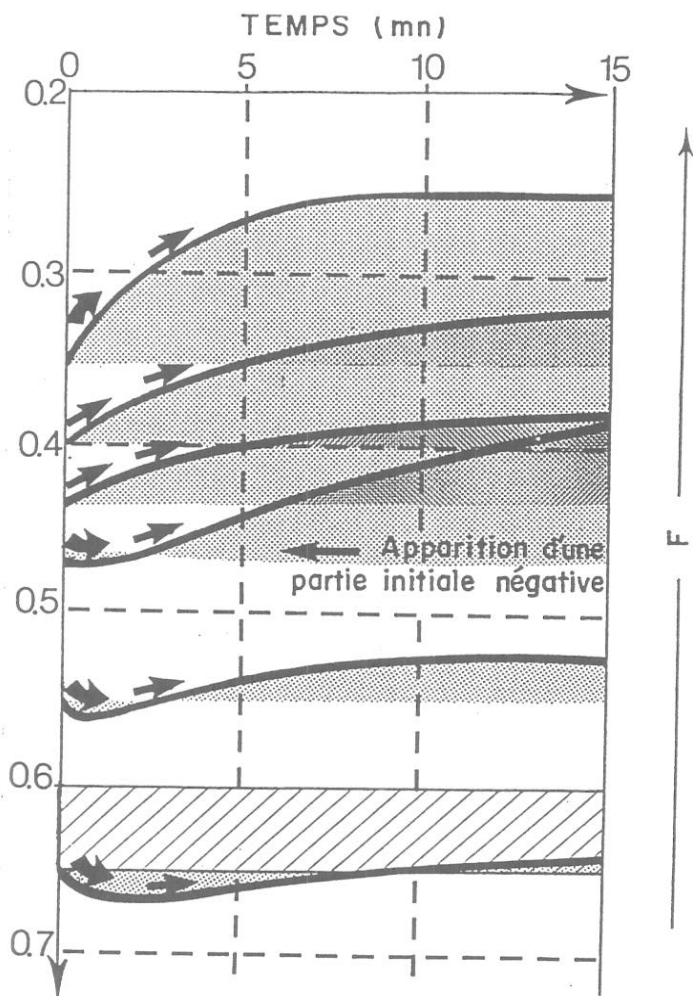


Figure 3 - Evolution of the fluorescence spectral alteration (F) of Cretaceous and Tertiary Botryococci with reflectance (Fourmont et al., 1989).

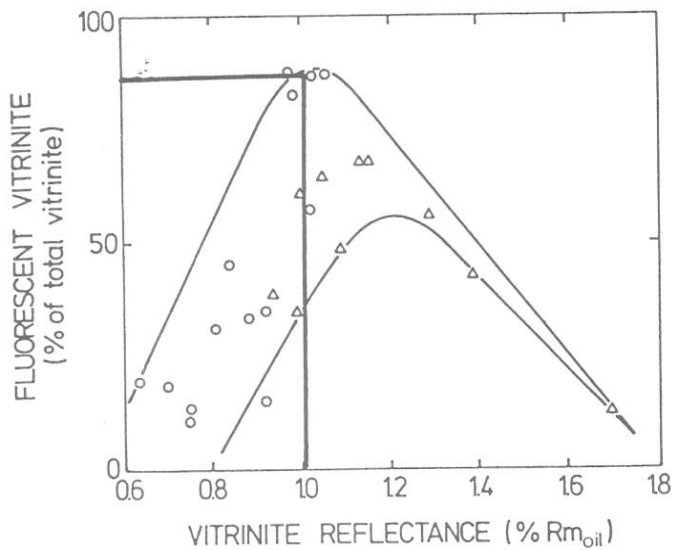


Figure 4 - Proportion of fluorescent vitrinite (% of total vitrinite) vs mean reflectance for coal samples from Kohlhaus and Uedem No.1 wells (circles and triangles respectively). Note that at 1% of reflectance, nearly 90% of the vitrinites are fluorescent in the Ruhr Basin. (Radke et al., 1980).

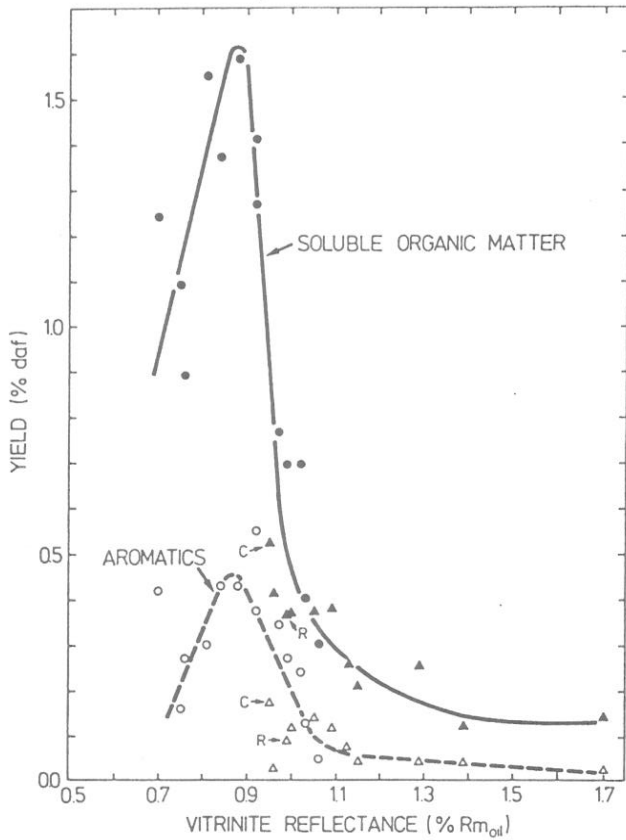


Figure 5 - Yields of soluble organic matter (solid signatures) and aromatics (open signatures) vs mean vitrinite reflectance for coal samples from Kohlhaus and Uedem No.1 wells (circles and triangles respectively). Note that the maximum of soluble organic matter is at $R=0.9\%$. [C=Cannel coal; R=Rock sample]. (Radke et al., 1980).

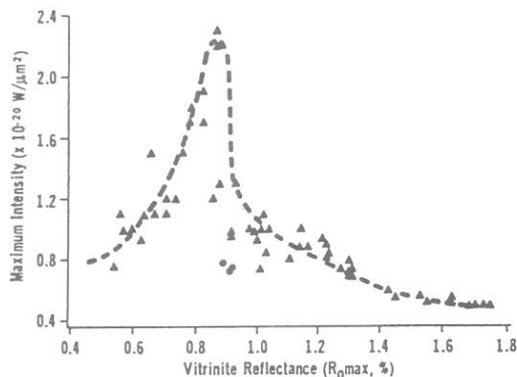


Figure 6 - Absolute maximum fluorescence intensity (blue light excitation) versus vitrinite reflectance. The fluorescence intensity of vitrinite passes by a maximum at $R=0.9\%$. It starts at about 0.7% and disappears at about 1.1%. (Lin et al., 1987).

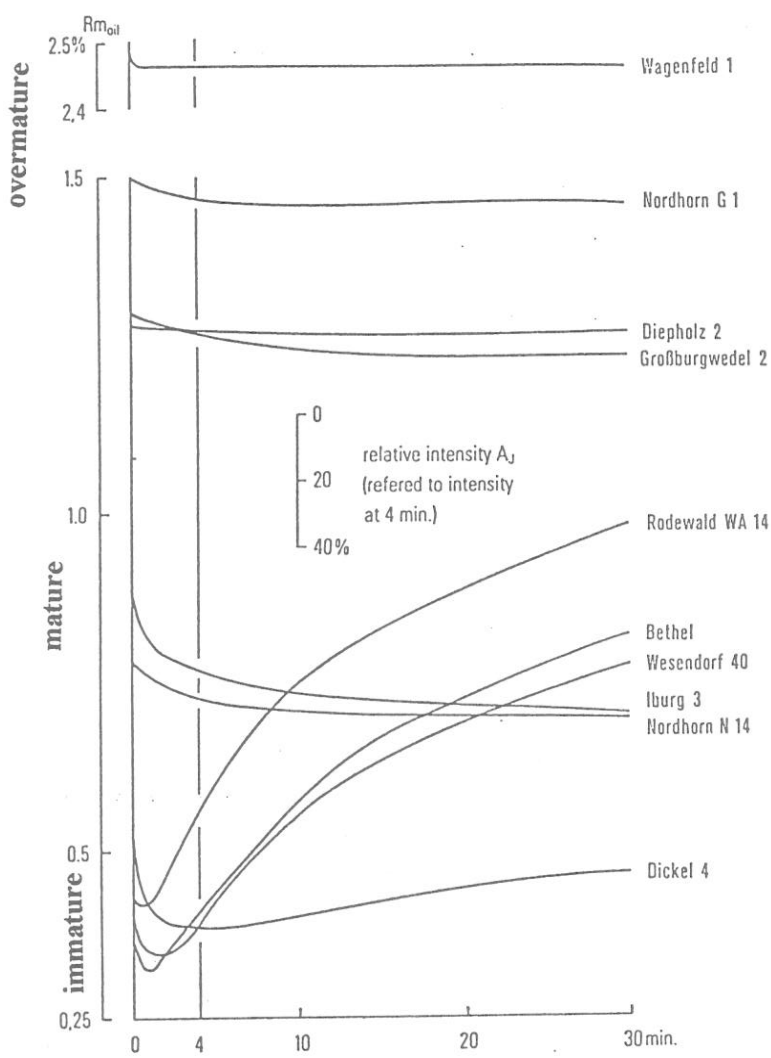


Figure 7 - Alteration curves measured on the mineral-bituminous groundmass of an oil source rock (Teichmüller, 1980 after Teichmüller & Ottenjann, 1977).

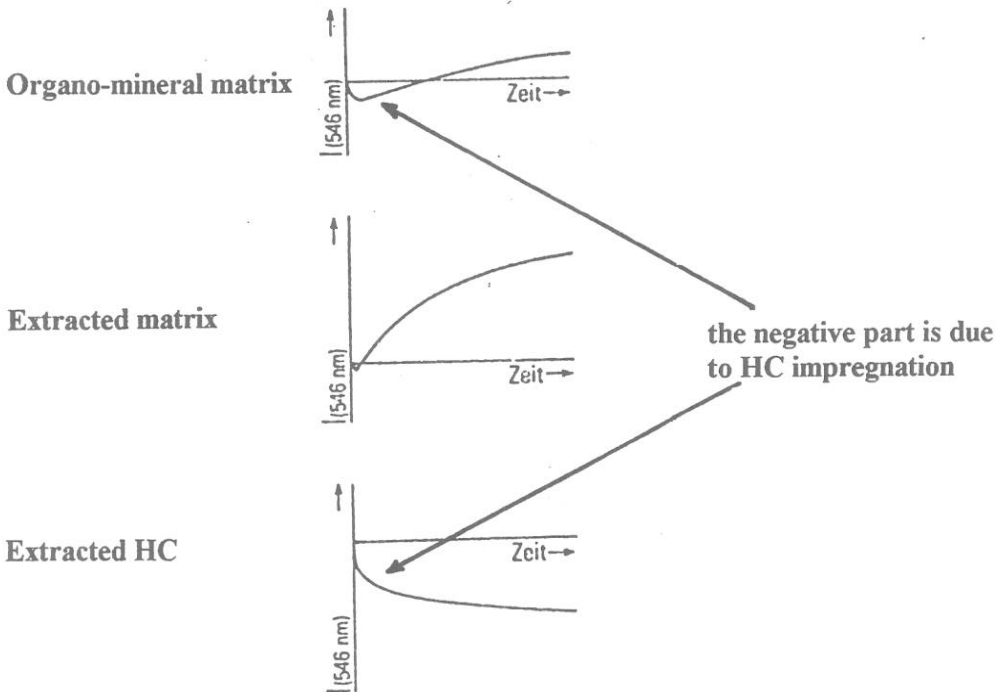


Figure 8 - Graphic representation of alteration curves obtained from the organo-mineral matrix of an oil shale, the extracted mineral matrix of the same oil shale and the extract. (Ottenjann, 1985).

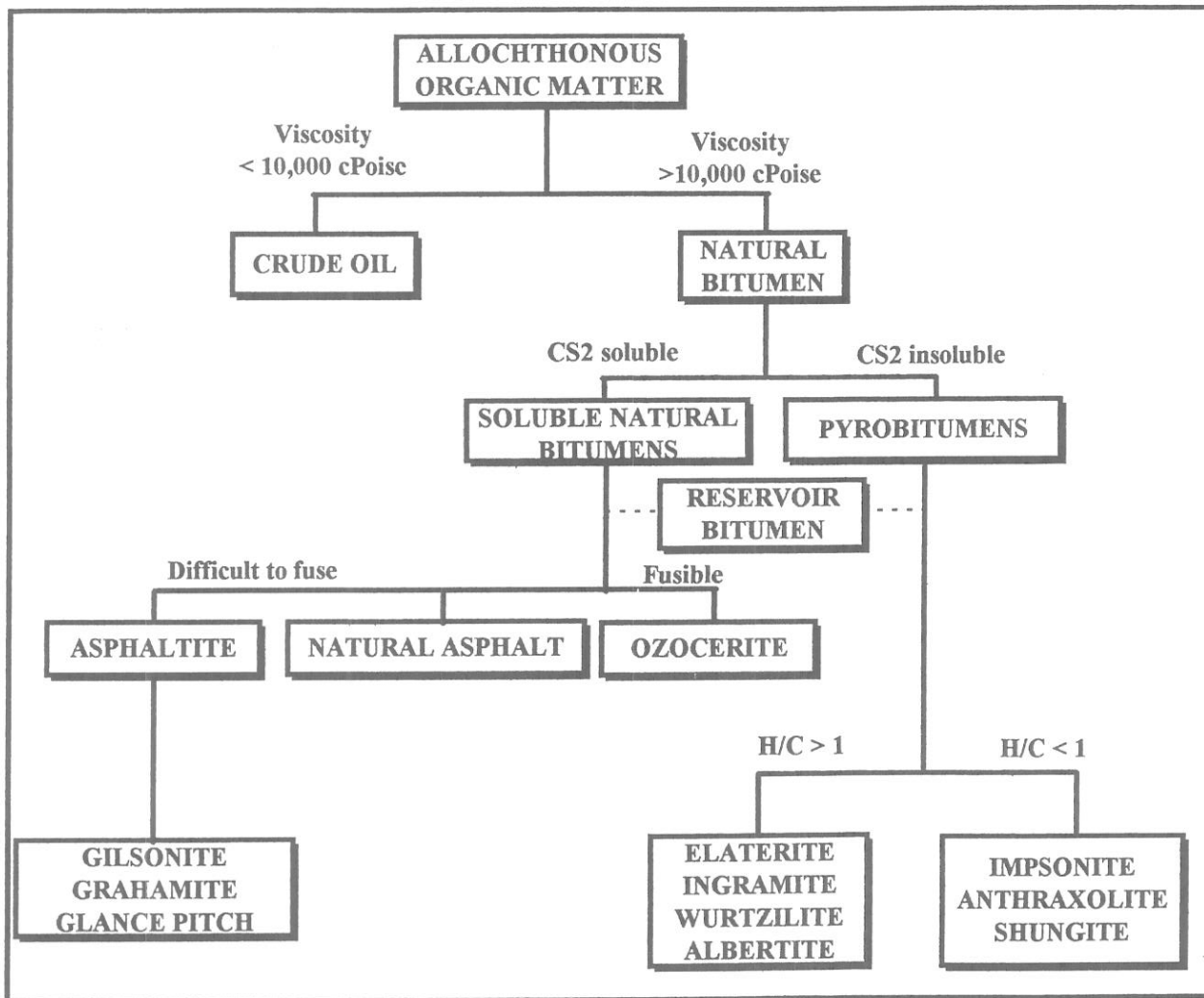


Figure 9 - Traditional classification of bitumens (Parnell, 1993, after Meyer & de Witt, 1990).

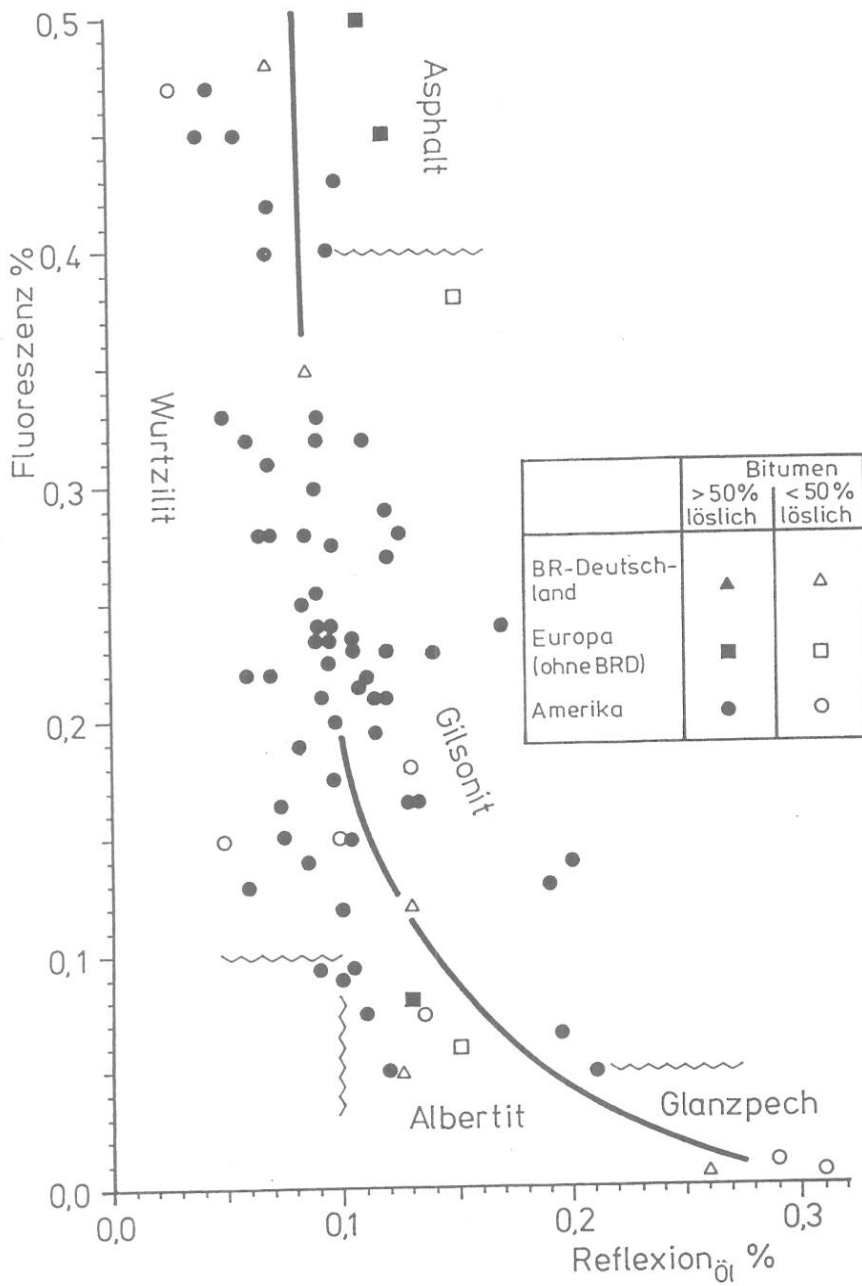
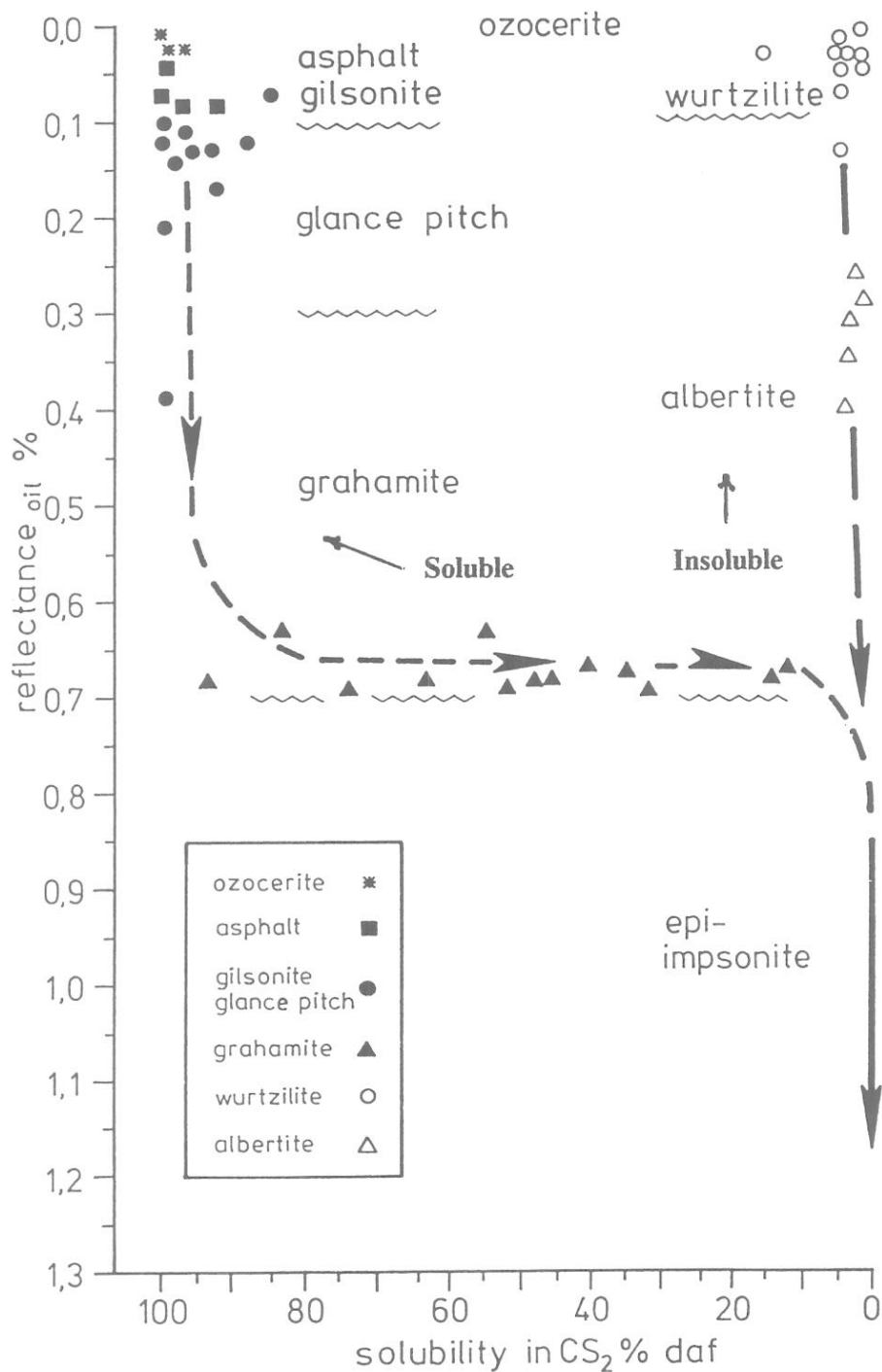


Figure 10 - Relation between reflectance and fluorescence of Liptibitumen (Jacob, 1983).



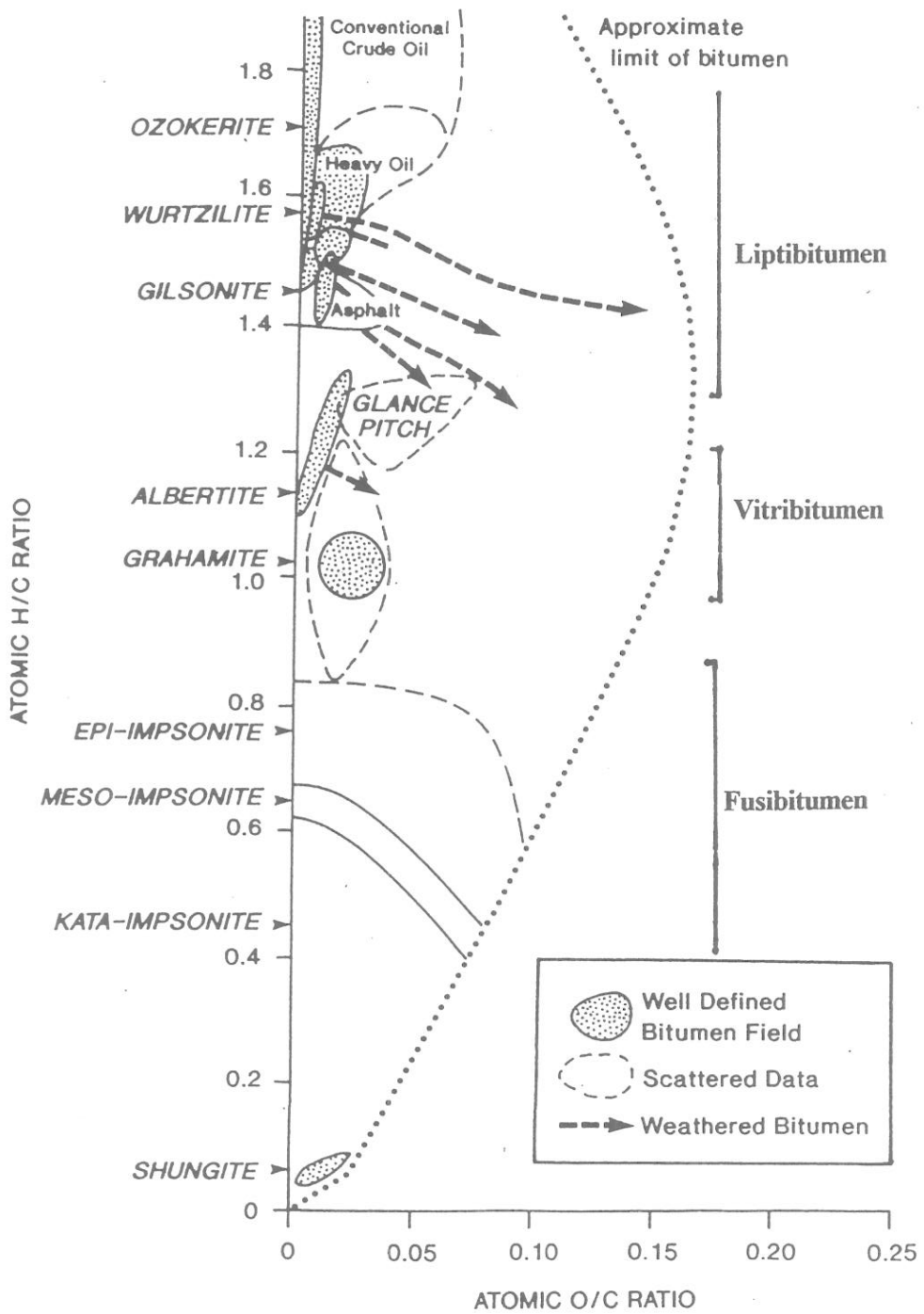


Figure 12 - Van Krevelen diagram of natural bitumens and crude oils (Cornelius, 1987).

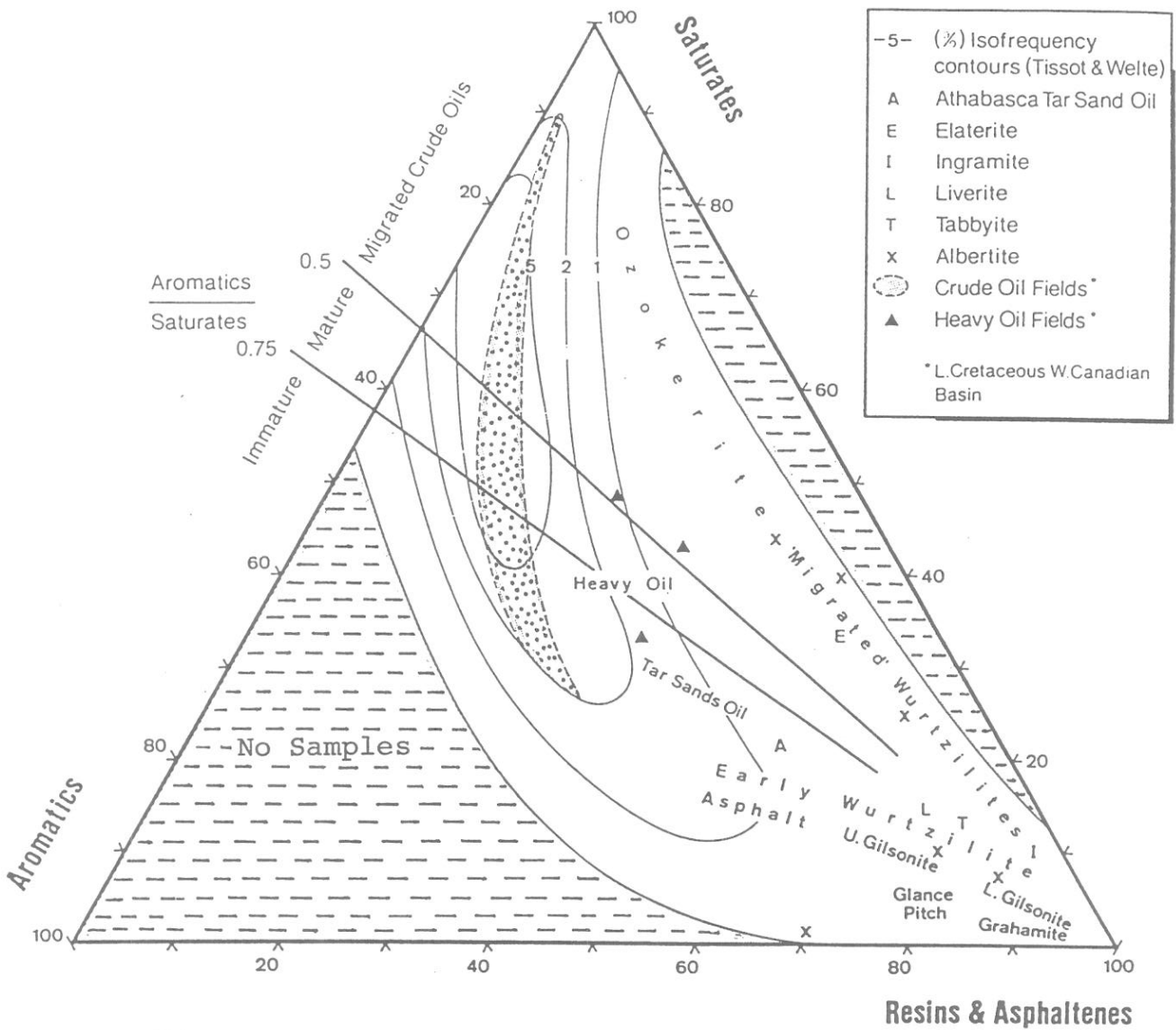


Figure 13 - Ternary petroleum composition diagram separating saturates, aromatics, and resins plus asphaltenes (Cornelius, 1987).

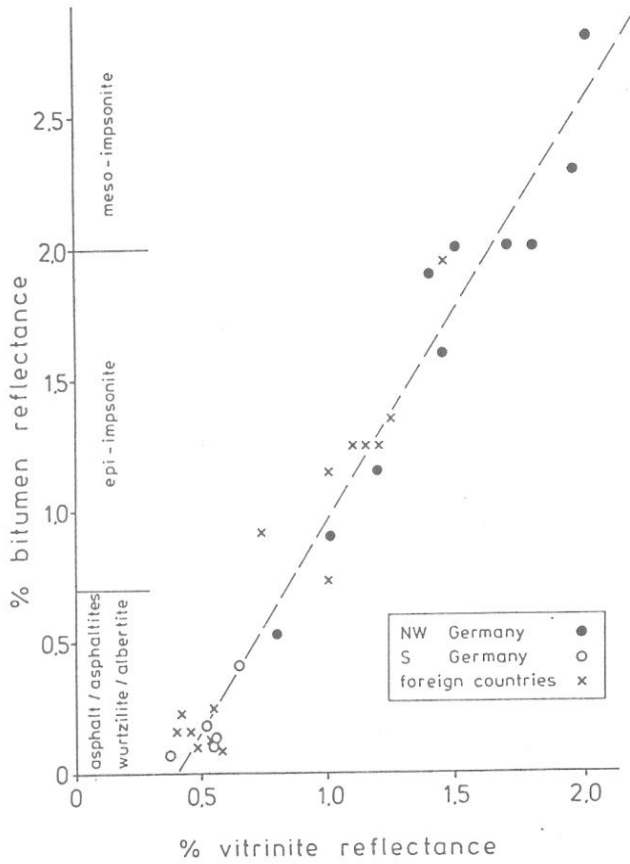


Figure 14 - Relationship between vitrinite and migrabitumen reflectance (in each case taking the lowest reflectance population). (Jacob, 1989).

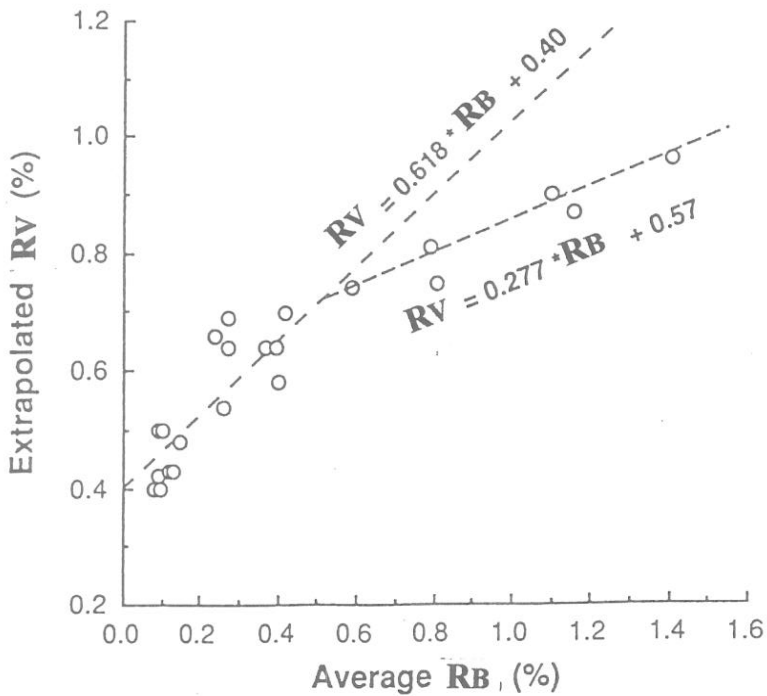


Figure 15 - Extrapolated reflectance of vitrinite against reflectance of bitumen. The relationship follows Jacob's equation up to RB of about 0.52% but beyond this value RB increases more rapidly than RV (Riediger, 1993).

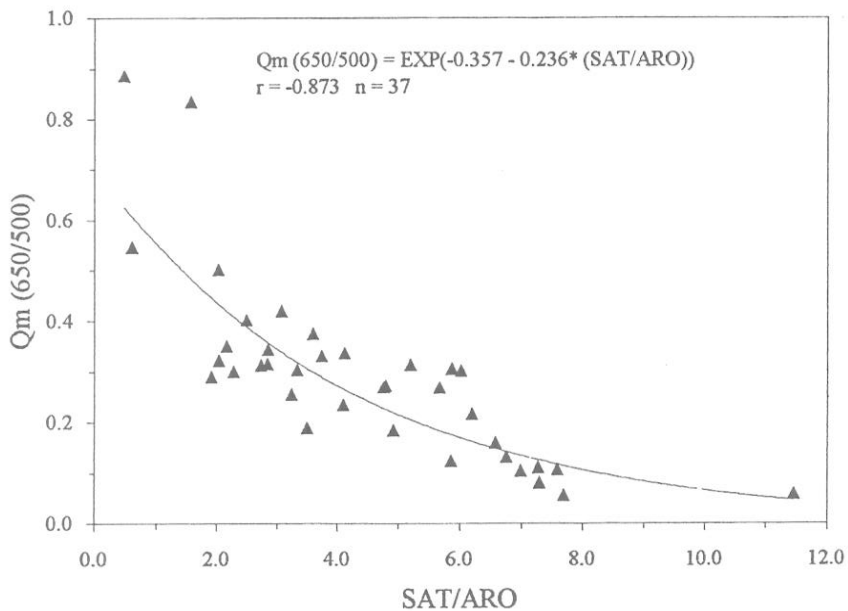


Figure 16 - Correlations between Q_m (650 / 500) measured on free crude oil, and oil quality (Saturates/Aromatics). (Alpern et al., 1993 in press).

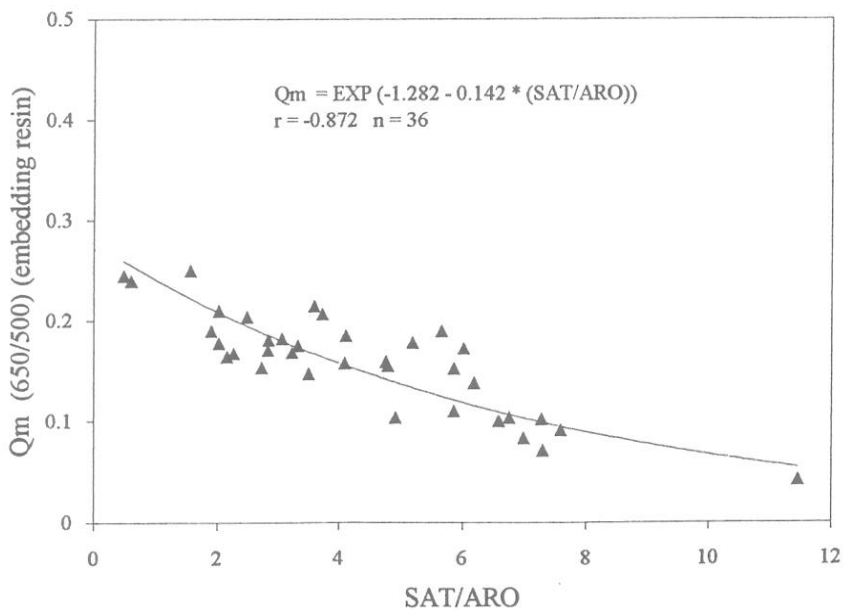


Figure 17 - Correlations between Q_m measured on crude oil mixed (1%) with EPOXY embedding resin and oil quality (Saturates/Aromatics). (Alpern et al., 1993 in press.)

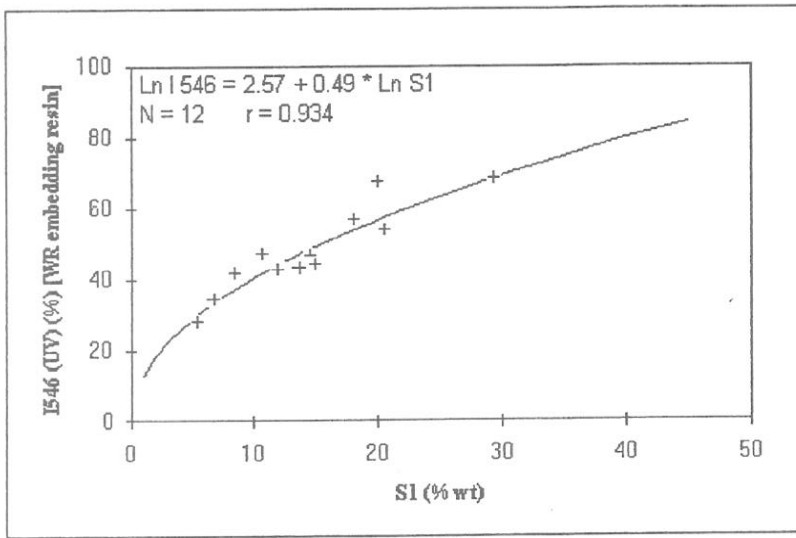


Figure 18 - A practical example (borehole CA 1) illustrating the correlation between I 546 of the whole rock (WR) embedding resin (polished block) and S1 from Rock Eval (Alpern et al., 1992).

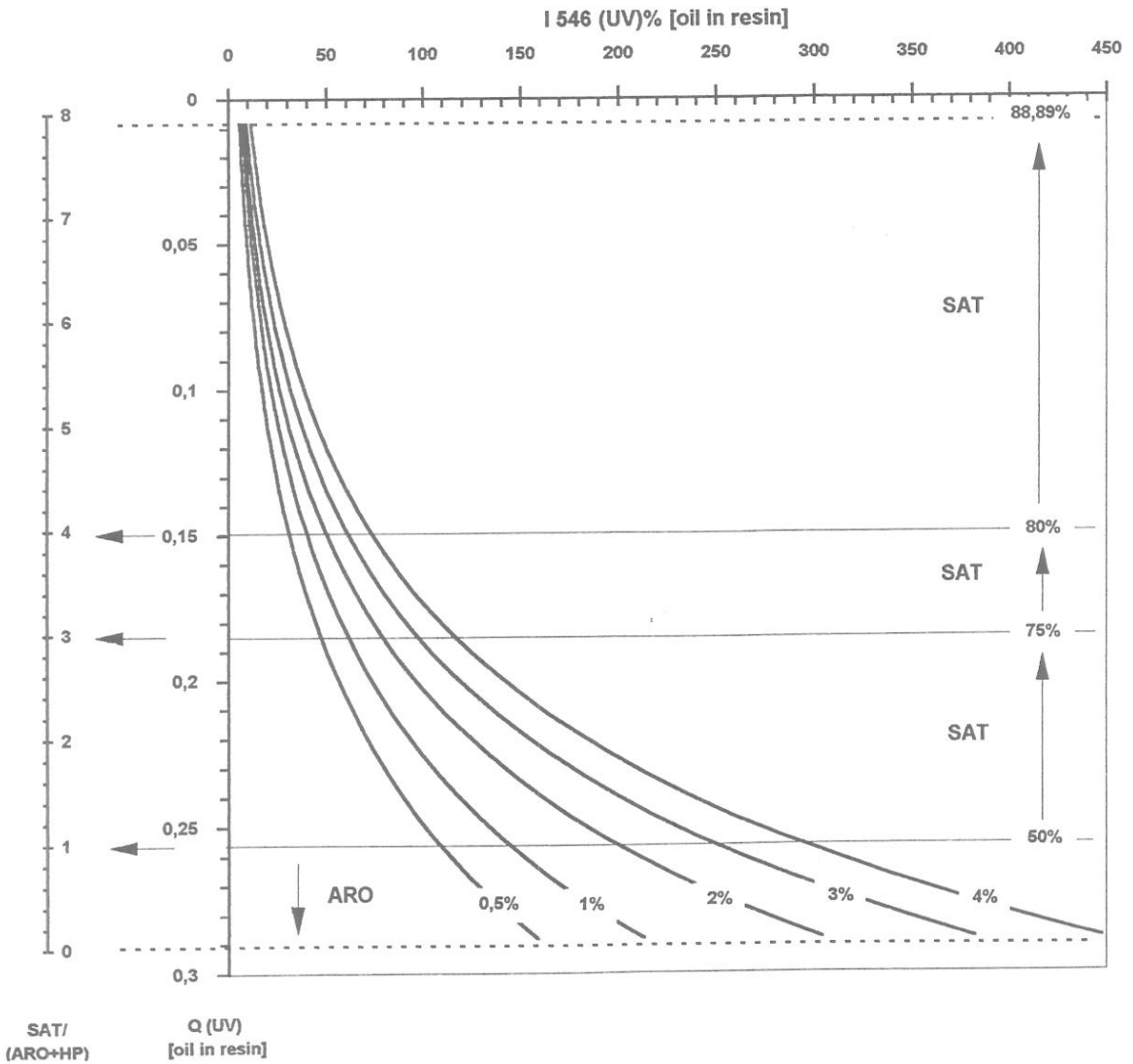


Figure 19 - Abacus based on the relation between Q (UV) and I 546 (UV) [both measured on the embedding resin] and Saturates/(Aromatics + Heavy Products). (Organic Petrology Unit, Porto, in-house research (1993)).

PLATES

PLATES

All photographs have been taken with a Leitz Orthoplan microscope equipped with fluorescence and a Photoautomat MPS 55. [Some of the microphotographs here illustrated are published in Alpern et al., 1992].

Blue light fluorescence was obtained with BG 12 excitation filter ($\lambda = 408$ nm), K 510 barrier filter and TK 510 dichroic mirror.

Magnification is provided by the scale below.

50 μ

PLATE I

1. Big greenish oil drop issuing from black matrix with orange Liptinite.

[Region: India; depth: 7740-7770ft]

2. Small to medium yellow drops issuing from dark brown matrices and trapped in high yellow embedding resin.

[Region: Angola; depth: 13740-13750ft]

3. Dark brown matrix without any visible organoclasts and surrounded by a necklace-type greenish-yellow film.

[Region: Angola; depth: 6670-6690ft]

4. Medium brown matrix, without liptinite, impregnated (in fissures) and covered by a thin yellow film. Weak green embedding resin.

[Region: Angola; depth: 6670-6690ft]

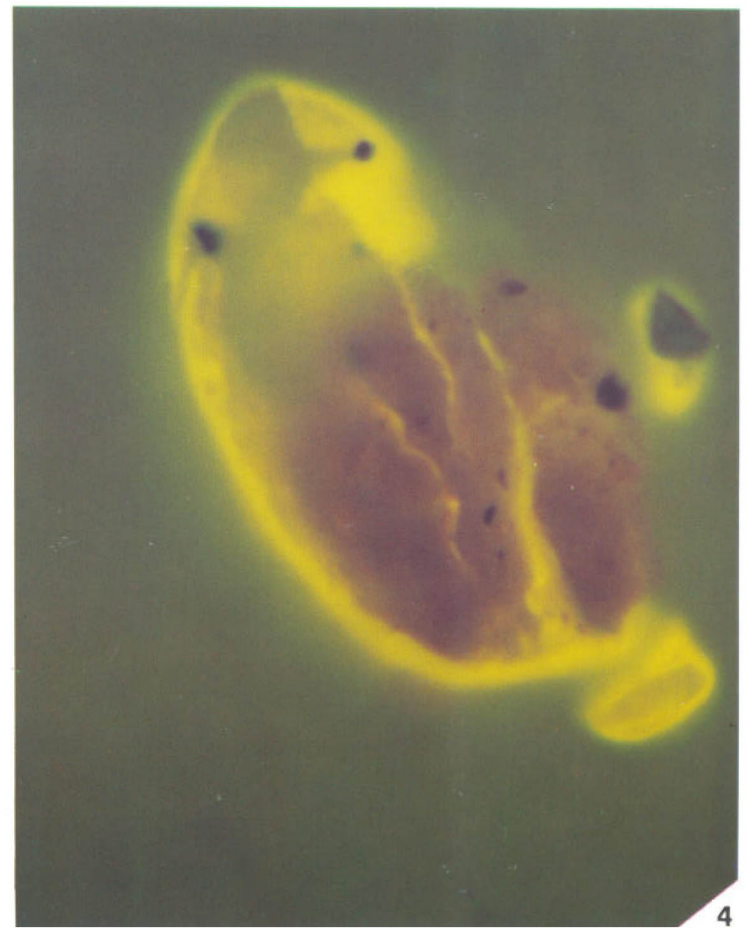
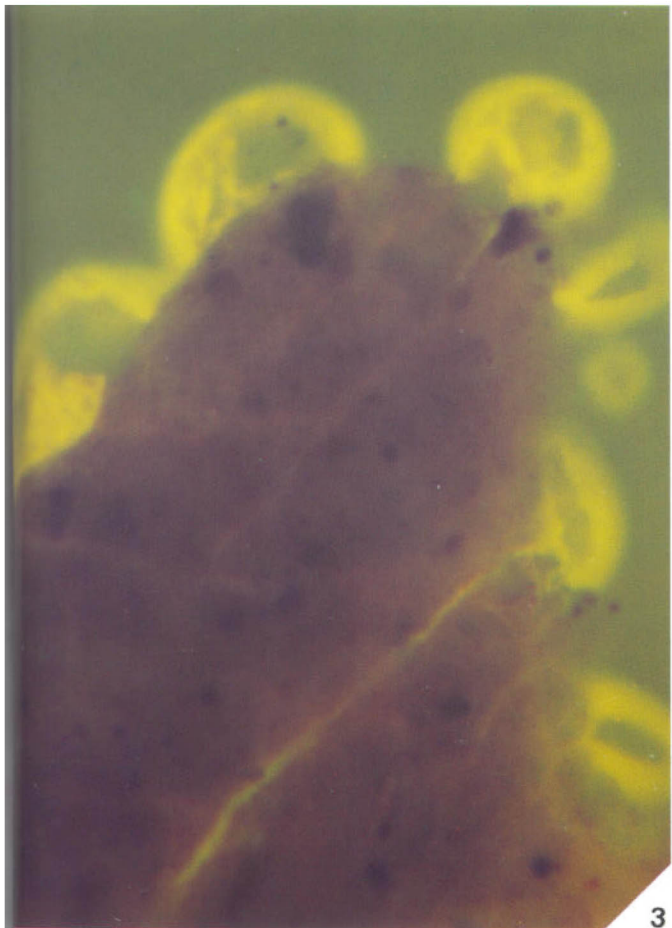
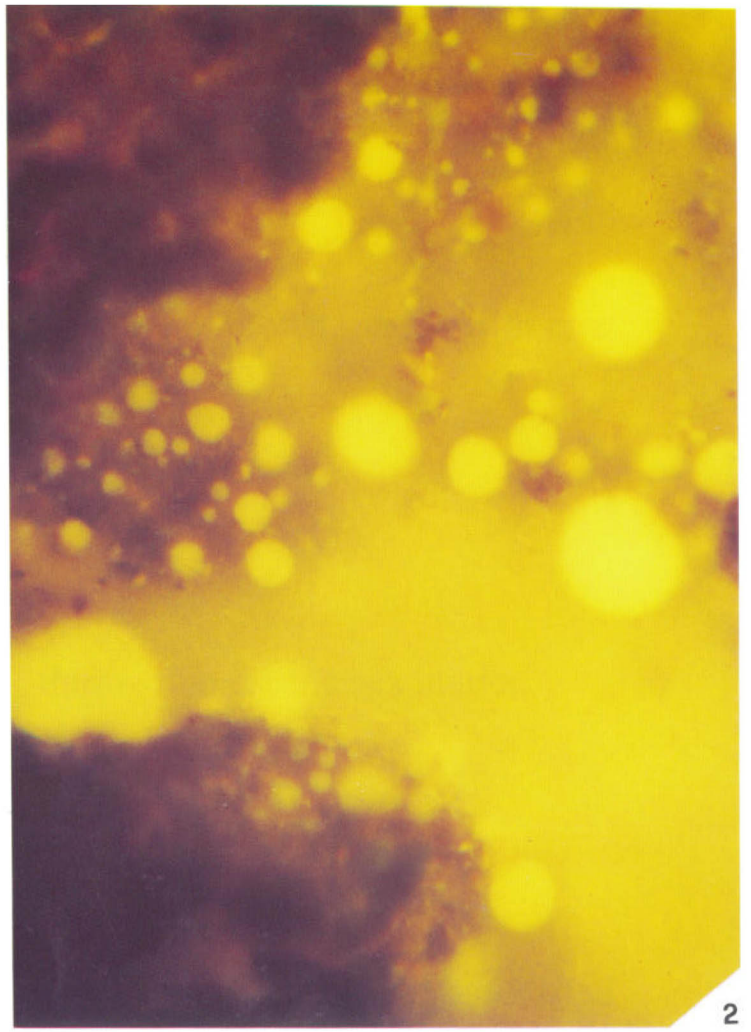
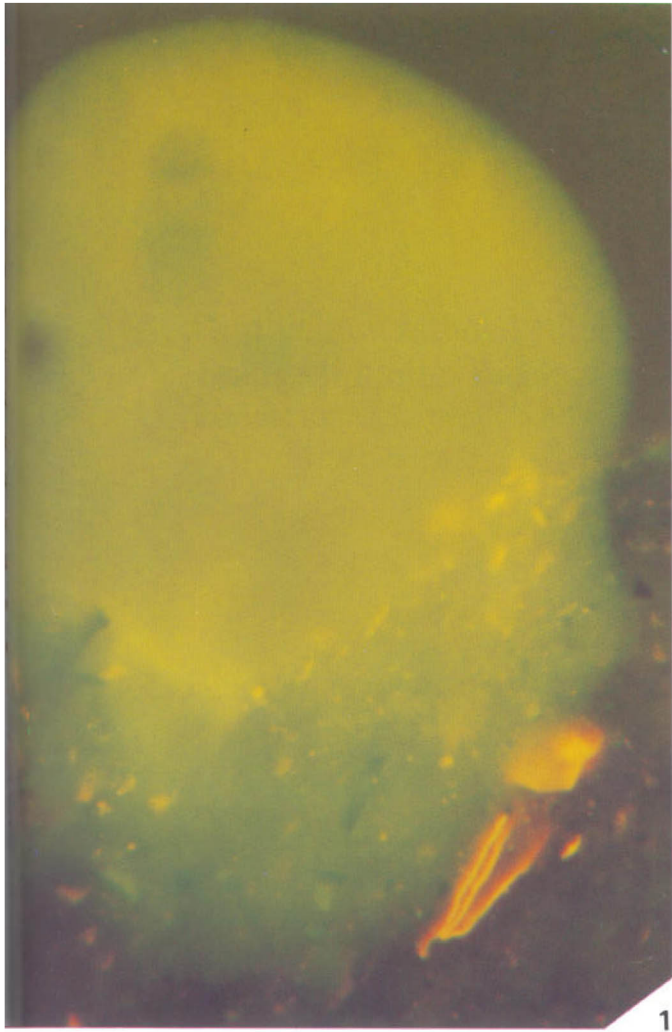


PLATE II

1. Thin yellow inflated film related to a weak brown matrix with rare orange liptinite. Weak green embedding resin.

[Region: Romania; depth: 3500m]

2. Black matrix strongly producing during fluorescence excitation.

[Region: Angola; depth: 6950-6970ft]

3. Dark matrix with unfigured orange-brown liptinite and associated clasts containing intercrystal greenish-yellow hydrocarbons.

[Region: Angola; depth: 13440-13450ft]

4. Clastic aggregate with non-spherical hydrocarbons filling inter-mineral spaces.

[Region: Argentina; depth: 798-800m]

