

## **CHAPTER 4**

### **COAL PETROGRAPHY**

Routine tools for source rock characterization in this chapter are based upon optical methods. These allow one to observe the components in their complexity, to identify their precursors, microfauna, pollen, spores, tissue fragments, etc, and to reconstruct the environment of deposition of the organic matter.

#### **4.1 DEFINITION OF MACERALS**

From International Committee for Coal Petrology (1971), macerals were defined as the microscopically recognizable individual constituent of coal and depending on their quantitative participation and their association, they control the chemical, physical and technological properties of a coal at a given rank.

#### **4.2 FLUORESCENCE MICROSCOPY**

Coal petrography is the study of the microscopically recognizable component of coal. It describes a coal in terms of its maceral composition and its rank level conveniently determined by reflected light microscopy.

Macerals of coal are microscopically differentiated on the basis of their reflectance, shape, structure and sometimes by such additional methods as etching, fluorescence, electron microscopy and luminescence.

Fluorescence is a useful technique for identification of liptinite material under reflected light microscope. An excitation wavelength (emission peaks between 366 and 435 nm of mercury lamp) in the UV or blue range of spectrum is used to produce a fluorescence, which is

generally restricted to the liptinitic components. Using this technique, liptinitic components are able to emit light from blue to red, this evidence is called fluorescence (Tissot and Welte, 1984).

Fluorescence is expected in molecules that are aromatic or contain multiple conjugated double bonds with a high degree of resonance stability. Both classes of substances have delocalized  $\pi$ -electrons that can be placed in low-lying excited single states. In polycyclic aromatic systems where the number of  $\pi$ -electrons available is greater than in benzene, these compounds and their derivatives are usually much more fluorescent than benzene and its derivatives. Substituents strongly affect fluorescence. Substituents that delocalize the  $\pi$ -electrons, such as  $-\text{NH}_2$ ,  $-\text{OH}$ ,  $-\text{F}$ ,  $-\text{OCH}_3$ , and  $-\text{N}(\text{CH}_3)_2$  groups, often enhance fluorescence because they tend to increase the transition probability between the lowest excited singlet state and the ground state. Electron-withdrawing groups that contain  $-\text{Cl}$ ,  $-\text{Br}$ ,  $-\text{I}$ ,  $-\text{NHCOCH}_3$ ,  $-\text{NO}_2$ , or  $-\text{COOH}$  decrease or quench the fluorescence completely.

### 4.3 COAL MACERALS

The macerals of coal can be divided in three different groups (Table 4.1 and 4.2) on the basis of reflectivity differences: huminite or vitrinite group, liptinite or exinite group and inertinite group. The differences in the reflectivities of the three maceral groups are most pronounced in the brown coals and they progressively decrease in the hard coal-rank, until in the anthracites. Reflectivity differences are virtually non-existent. In the anthracites, a coalification phase of the hard coals, both shape and Table structure are the most important distinguishing

characteristics of the macerals are as the following details (International Committee for Coal Petrology, 1971).

Table 4.1 Classification of coal macerals (after International Committee for Coal Petrology, 1971).

<b>Maceral Group</b>	<b>Maceral</b>	<b>Submaceral</b>
Vitrinite	Telinite	Telinite 1 Telinite 2
	Collinite	Telocollinite Gelocollinite Desmocollinite Corpocollinite
	Vitrodetrinite	
Liptinite	Sporinite	
	Cutinite	
	Resinite	
	Alginite	
	Bituminite	
	Exsudatinite	
	Fluorinite	
	Liptodetrinite	
Inertinite	Micrinite	
	Macrinite	
	Semifusinite	
	Fusinite	Pyrofusinite Degradofusinite
	Sclerotinite	Fungosclerotinite
	Inertodetrinite	

Table 4.2 Classification of low-rank coal macerals (after International Committee for Coal Petrology, 1971).

Maceral Group	Maceral Subgroup	Maceral	Submaceral	
Huminite	Humotelinite	Textinite		
		Ulminite	Texto-ulminite Eu-ulminite	
	Humodetrinite	Attrinite		
		Densinite		
	Humocollinite	Gelinite	Porigelinite Levigelinite	
		Corpohuminite	Phlobaphinite	
			P s e u d o - phlobaphinite	
	Liptinite		Sporinite	
			Cutinite	
Resinite				
Suberinite				
Alginite				
Bituminite				
Exsudatinite				
Fluorinite				
Liptodetrinite				
Inertinite		Fusinite		
		Semifusinite		
		Micrinite		
		Macrinite		
		Sclerotinite		
		Inertodetrinite		

#### 4.3.1 The huminite /vitrinite group.

Huminite denotes a group of maceral of brown coals whereas vitrinite group is used for a hard coal. They are subdivided into maceral subgroups, depending on the state of preservation of the plant material,

and into macerals and submacerals on a basis of degree of gelification or alternatively, on form and origin. Huminite consists of humic materials, which essentially arise from lignin and cellulose. Huminite is generally distinguished by average reflectivity in reflected light : a brown yellow to reddish brown colour in transmitted light, a relatively high oxygen content, a tendency to gelification (vitrinization) in the peat and brown-coal stage, a capacity to briquette satisfactorily in an ungelified or non-collinitic condition. Huminite is precursor of the vitrinite of hard coals. The explanations of each maceral in huminite group are as follows (Table 4.2):

#### 1. Humotelinite

Humotelinite is a maceral subgroup of huminite which consists of intact cell walls of tissue or isolated single cells in a humic state of preservation. Humotelinite is a precursor of tellinite and of telocollinite of hard coals. Depending on its degree of gelification, it is subdivided into the macerals textinite and ulminite.

#### 2. Humodetrinite

Humodetrinite is a maceral subgroup of huminite which consists of the finest humic fragments with a finely distributed humic gel between them. Humodetrinite is generally a precursor of desmocollinite and partly also of vitrodetrinite of hard coals. Depending upon its degree of gelification, it is subdivided into the macerals attrinite and densinite.

#### 3. Humocollinite

Humocollinite is a maceral subgroup of huminite which consists of an amorphous humic gel or of intensely gelified plant tissues and humic detritus. An exception is the submaceral phlobaphinite which represents one of the cell excretions formed by plants. In reflected light under oil

immersion humocollinite always appears homogeneous and it often occupies former cavities. Humocollinite is precursor of collinite of hard coals. It is subdivided into the macerals gelinite and corpohuminite.

The vitrinite group have the originate mainly from the humic-acid fraction of humic substances, which are dark-coloured compounds of complex composition. These compounds the elements carbon, oxygen and nitrogen which consist of aromatic nucleus and contain hydroxyl (-OH) and carboxyl (-COOH) functional groups. The compounds form through mouldering and peatification, even partly in the brown coals, chiefly from the lignin and cellulose of plant-cell walls (Stach and others, 1982). the vitrinite group consists of three macerals : tellinite, collinite, and vitrodetrinite (Table 4.1).

#### 1. Telinite

Telinite indicates the vitrinite which was derived from the plant cell walls whose structure was still displayed.

#### 2. Collinite

Collinite imply those vitrinite macerals which do not show any cellular structure. The collinite maceral includes four submacerals. Telocollinite occurs as wide homogenous bands, because of its microscopic homogeneity, its reflectance is commonly used as a rank indicator. Gelocollinite is believed to be formed from impregnation of humic gel into fissures and cell cavities; therefore, its boundary are defined by other associated macerals. When present as bands, it typically shows shrinkage cracks. Desmocollinite is often associated with other macerals such as sporinite. The term corpocollinite is used to significant

the vitrinite which occurs as homogenous and massive bodies in round or oval shapes, and which originates as cell filling (Lin and Davis, 1988).

### 3. Vitrodetrinite

Vitrodetrinite occurs in the form of detritus, these fragments mostly originate from plants or humic-peat particles which were degraded at a very early stage; rarely they are fragments of vitrinite which has been crushed by pressure, such as those which are embedded in collinite (Stach and others, 1982).

#### 4.3.2 The liptinite Group

Liptinite Group is originated from hydrocarbon rich plant material such as sporopollen, cutin, suberin, resin, waxes, balsams, latex, fats and oils, and from bacterial degradation products of protein, cellulose and other carbohydrates (Stach and others, 1982). Of the three maceral groups, liptinite macerals have a highest hydrogen contents and the lowest carbon contents (Lin and Davis, 1988). The liptinite macerals have long been of interest to the coal carbonisation industry because of their large yields of tar and gas. More recently they have been the object of interest of those in both the natural and synthetic petroleum industries because they are clearly a major source of naturally occurring liquid hydrocarbon (Cook and Kansler, 1982). The macerals in liptinite group consist of (Table 4.1, 4.2),

##### 1. Sporinite

Sporinite was first published in the International Commission for Coal Petrology (1957) (after International Committee for Coal Petrology, 1971) for a maceral of the exinite (liptinite) group, which arises from spores and pollens. Sporinite consists of the outer cell walls (exines and

perines) of spores and pollen. Sporinite typically is seen as elongated grains as a result of compression of exines, with a central line representing the compressed central cavity, when viewed on a polished surface perpendicular to the bedding (Lin and Davis, 1988). These cell walls are comprised of sporopollenin, which is a very resistant, highly polymerized, cross-linked, insoluble substance, consisting of oxidative polymers of carotenoids and carotenoid esters (Stach and others, 1982). Sporine, the ash-free substance of sporinite forms through the dehydration and dehydrogenation of sporopollenin, has variation in chemical constitution. Analysis elements of sporinite from hard brown coal from Moscow Basin, contain carbon 79.9%, hydrogen 6.0%, oxygen 6.3%, nitrogen 0.5%, and sulphur 7.2%. Sporinite occurs in varying amounts in all brown coals. In Tertiary soft brown coals, it is always below 10% of the whole coal (International Committee for Coal Petrology, 1971).

## 2. Cutinite

Cutinite is a maceral of the liptinite group, which mainly arises from the cuticles of leaves and stems. Cuticular layers and cuticles, the originators of cutinite, are formed from the protoplasts within the outer walls of the epiderms of leaves, stems and other aerial plant parts. The cuticular layers form a thickening of the outer epidermal cell walls, consisting of cellulose at the base, followed by cutin, wax, cellulose and pectin (cutocellulose), on top of which is deposited a water resistant, thin skin, the cuticle, which is comprised of pure cutin. Cutin is mainly comprised of saturated hydroxy-fatty acids and waxy alcohols. It has a highly polymeric character and chemically is close to suberinite (Stach and others, 1982). From elementary analysis of cutinite, it consists of

carbon 70-76.4%, hydrogen 7.6-11.8%, and oxygen 11.8-21.7% (International Committee for Coal Petrology, 1971). It usually can be readily identified by its typical serrated margin along one side. Cutinite is often only a minor constituent of coals; however, in some cases, it can reach great abundance (Lin and Davis, 1988).

### 3. Resinite

Resinite is used for a maceral of the liptinite group, which mainly arises from resin (International Committee for Coal Petrology, 1971). Resinite has differing origins, but its principal precursors are probably resins and waxes, although balsam, copals, latex, oils and fats are also considered to be source materials. Chemically the resins, copals, essential oils, balsam, and latex belong to the terpenes. Fats and waxes belong to the extractable lipids. On the basis of the characters of its precursors, resinite can be divided into three large genetic groups : terpene resinite, lipid resinite and secondary resinite (exsudatinite). Terpene resinite originated from resins. Chemically resins belong to the terpene group. Resins form in secretion cells particularly in conifer wood, in tabular resin ducts or form resin pockets which resin impregnated sites in the wounded tissues. Isolated resinite bodies are particularly common in Tertiary coals. Lipid resinite originates from fats and waxes. Fats are collect in the plasma of seed cells. Chemically, it composed of mixing between glycerin esters and different fatty acids. With its composition is very rich in hydrogen, thus fatty acids play an important role as precursors of petroleum. Waxes are esters of higher fatty acids with higher aliphatic alcohol. They also contain free fatty acids and are generally very similar to the fats. Waxes are deposits on the surface of leaves and fruits as fine grains, small rods, or as compact crusts (Stach

and others, 1982). From elementary analysis of cutinite, it contains carbon 77.0-85.0%, hydrogen 8.2-11.0%, and oxygen 2.7-13.1% (International Commission for Coal Petrology, 1971). They are often finely disseminated in Tertiary coals, these entities are grouped with liptodetrinite. Secondary resinite (exsudatinite) is believed to have been formed from petroleum like materials in bituminous and lower rank coals (Stach and others, 1982).

#### 4. Suberinite

Suberinite originates from corkified cell walls, which occur mainly in barks, but also at the surface of roots, on stems and on fruits, acting as a protection against desiccation. Cork cells consist of layers of cellulose, lignified cellulose and pure suberin. Suberin consists of glycerine esters of high molecular unsaturated and saturated fatty acids and oxy-fatty acids. Suberin is similar to cutin, but is less strongly polymerized and thus is more easily attacked (Stach and others, 1982). Suberinite is present in coals of all age (Permian to Tertiary) (Cook and Kansler, 1982).

#### 5. Alginite

Alginite, International Committee for Coal Petrology (1971) has designated alginite as a maceral of the liptinite (exinite) group, it is derived mostly from algae. Alginite quite often displays the original structure of the algal colonies from which it is derived. Stach and others, (1982) suggested there are two forms, *Pila* and *Reinschia*, are known from carboniferous boghead, which can be related to the recent green algae polymorphous colonies forms, *Botryococcus braunii*, occurring predominantly in fresh water and distributed throughout the whole world. Chemically all alginite are distinguished from the others by its relative

high hydrogen content and lability. It has the lowest aromaticity of all bituminous and low rank coal macerals (Lin and Davis, 1988). There are two main groups which, for convenience, are termed alginite A and alginite B. Alginite A appears as discrete algae bodies (both colonial and unicellular) which are either elliptical, spherical or disc-shapes and form the characteristic algae components of the oil-shale. Alginite B is green or blue-green algae formed as finely banded lamellar alginite intimately interbedded with mineral matter in well laminated (Cook and Kansler, 1982).

#### 6. Bituminite

Bituminite is used for a maceral of liptinite group that is distinct by virtue of its amorphous character (International Committee for Coal Petrology, 1971). It occurs as decomposition product of algae, animal plankton, bacterial lipids and similar precursors which is recognized by its lack of definite shape. Generally found in sapropellic coals and other subaquatic, mainly liptinite rich coal types. Bituminite is predominant maceral of oil shales and other oil source rocks (Stach and others, 1982). Chemically, 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 and will yield large amounts of volatile matter, extracted bitumens and tar (International Committee for Coal Petrology, 1971).

#### 7. Exsudatinitite

Exsudatinitite originated from oils or bitumen related material expelled during coalification that derived from other exinite macerals or possibly vitrinite macerals. General occurs in veins, wedge-shaped cracking, infilling bedding plane joints or infilling empty cell lumens.

Found in coals of bright brown coal to high volatile bituminous coals (Cook and Kansler, 1982).

#### 8. Fluorinite or chlorophyllinite

Fluorinite originated from lipids, essential oils, possibly of secondary origin in some occurrences. Characterised by a very high fluorescence intensity, extremely strong green or green-yellow fluorescence. Occurs as large, isolate, featureless mass or cell lumen infilling and as small lenticular masses. Present in coal of all ages (Cook and Kansler, 1982).

#### 9. Liptodetrinite

Liptodetrinite is a collective term for all liptinitic constituents which, because of their finely detrital nature and small particle size, cannot be grouped with other liptinite macerals. Comprised of fragments and fine degradation remains of sporinite, cutinite, resinite, alginite and suberinite (Stach and others, 1982). Moreover, fluorescing constituents of a few micron size, unknown origin and varying form, belong to liptodetrinite. Its chemical is still not investigated, however, can compare sporinite, cutinite, resinite, suberinite, alginite. Characteristically must have relatively high content of hydrogen and volatile matter. Liptodetrinite is usually abundant when other macerals of liptinite group are strongly represented (International Committee for Coal Petrology, 1971).

#### 4.3.3 Inertinite Group

Inertinite maceral group has precursor from cellulose and lignin from cell walls of plants which acting as fusinitization product substances. These substances contain relatively high carbon contents, lowest

hydrogen content of three maceral groups (Stach and others, 1982) (Table 4.1, 4.2).

### 1. Fusinite

Fusinite is used for an opaque coal constituent which displayed cell structure. Fusinite consists of highly reflective cell walls which are usually thinner than the cell walls of the corresponding humotellinite and semifusinite (International Committee for Coal Petrology, 1971). Two types of fusinite are distinguished on the basis of their origin. Pyrofusinite is believed to have been formed by forest fires; its cell walls are very brittle. On the other hand, degradofusinite is thought to have been formed from gradual oxidation and dehydration processes. Its cell walls are more swollen in comparison to pyrofusinite (Lin and Davis, 1988). Fusinite is characterized chemically by a relatively high carbon content and low contents of hydrogen, oxygen and volatile components. It occurs in small amounts in most lignites, dispersed with attrinite and densinite (International Committee for Coal Petrology, 1971).

### 2. Semifusinite

Semifusinite is used for a transition material between vitrinite and fusinite. Semifusinite in lignite consists of cell walls whose reflecting capacity lies between that of the huminite and fusinite of the same coal. Semifusinite is formed of tissues with more or less well preserved cellular structure. Cell walls of semifusinite are usually thicker than fusinite. The cell cavities are of different shapes and sizes depending on the source material, the microbial destruction, and the orientation of the section. Semifusinite is found in small amounts in most lignites of all ages (International Committee for Coal Petrology, 1971). May be partly of forest fire origin but much semifusinite is thought to be the result of

biochemical processes associated with fungal and bacterial attack (Cook and Kansler, 1982).

### 3. Macrinite

Macrinite is used for an analogous maceral found in lignites. Macrinite is characterized by a more or less dense structure 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 (International Commission for Coal Petrology, 1971). It to be have been formed from oxidized humic material which was gelified prior to oxidation (Cook and Kansler, 1982). generally appears with roughly rounded outlines without showing any cellular structure (Lin and Davis, 1988).

### 4. Micrinite

Micrinite appears as fine grained and highly reflecting with a granular texture. Some micrinite may form during early diagenesis but most appears to be a result of disproportionation reactions associated with hydrocarbon generation (Cook and Kansler, 1982).

### 5. Inertodetrinite

Inertodetrinite is composed of detrital or attrital inertinite derived from the breakdown of fusinite and semifusinite (Cook and Kansler, 1982).

### 6. Sclerotinite

Sclerotinite is used for all strongly reflecting fungal remains. In Carboniferous and Permian coals (mainly hard coals), hard, mainly oval secretion bodies of high reflectance are described as sclerotinite. Sclerotinite consists mainly of roundish, predominantly oval forms

which often display chambers (fungal spores, sclerotia) (International Commission for Coal Petrology, 1971). In Tertiary coal is almost exclusively of fungal origin (fungosclerotinite). It can be readily recognized by its typical morphology, occurring in round, oval and tabular forms with cavities (Lin and Davis, 1988). The chemical constitution of sclerotinite is unknown. Sclerotinite generally only occurs in small amounts. It can be associated with all other macerals (International Committee for Coal Petrology, 1971).

#### 4.4 FLUORESCENCE OF MACERAL

Liptinite group display high fluorescence intensities in peat and low rank coals. With increase in rank, the fluorescence intensity decrease. At the same time, the fluorescence colours shift to longer wavelengths. The fluorescence colours for the liptinite macerals are, however, not uniform, but differ from one maceral to the other. The fluorescence colours change from green-yellowish in the peat stage to orange brown in high volatile A bituminous coals. In medium volatile bituminous coals at reflectance level of 1.3 %, the fluorescence of liptinite macerals disappears.

Huminites/Vitrinite group : some of the huminites and low to medium rank vitrinite also fluoresce. The fluorescence colour change from yellow in lignites to brown bituminous coals. The fluorescence in vitrinite is probably caused by a relatively high proportion of hydrogen rich material from lipid substances which are absorbed or chemically incorporated in the vitrinite.

Inertinite group : do not show fluorescence (Table 4.3).

Table 4.3 : Fluorescence color and intensity for the maceral groups at difference coal rank using blue-light excitation (after Cook, 1982)

	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

## 4.5 PROCEDURE

### 4.5.1 Sample preparation

Due to the importance of petroleum source of the Pattani Basin, the cutting sample from 10 wells selected from 10 petroleum fields from this area were used to study by coal petrographic microscopy. Each sample consists of washed and dried cutting that was selected and mixed together to lie over 150 feet of samples. Samples were prepared as polished sections by impregnated with epoxy resin with a radial 1.5 cm and polished prior to study. Each sample was study under white reflected light and then excited by blue light fluorescence with oil immersion. Macerals were identified, counted the proportion of each macerals and

the petrographic recorded prior to scientific interpretation (Stach and others, 1982).

#### 4.5.2 Identification procedure

For fluorescence microscopy (MPM microscope photometer, Figure 4.1) the instrument is equipped with reflected light system F1, further components are

1. Fluorescence illuminator with collector and HBO 50w mercury lamp supplied from a separated power supply.
2. Three slots for sliders on the side of the microscope. The rearmost is generally used for a slider which either interrupts the illumination beam path (fully inserted), brings a red-attenuated filter BG38 into the beam path, which illuminates disturbing IR light (middle position), or provides free aperture. The other two slots are for accommodation of filter slider A with one 18 mm dia. Aperture for an additional exciter filter. A heat-reflecting filter KG1, which does not affect UV excitatio, is visible from the outside.
3. Lever for luminous field diaphragm.
4. Centering screws for luminous field diaphragm.
5. Reflector slider 3F1 fits has three apertures : the one in the middle is free for brightfield or phase contrast observation, the others accept suitable exciter filter/chromatic beam splitter/barrier filter sets
6. Sheet-metal cover which protect the fluorescence filters from dust; these covers must be inserted in the analyzer slot.

### **Procedure**

- Adjust the selected specimen feature in transmitted light brightfield or phase contrast using reflector slider (7.5) in the middle position (free light path) and lower illuminator with halogen lamp put block its light path with rear slider (7.2)

- Switch off transmitted-light illuminator (or reduce at least its brightness), remove all filters in magazine in the stand base from the beam path, select the left or right position of the reflector slider, depending on the type of excitation, and remove slider 7.2 from the beam path

- Since a narrow aperture diaphragm would reduce brightness in reflected-light fluorescence, only a luminous field diaphragm is provided. Use lever 7.3 to close it so far that it become visible in the image, then center with 7.4 and open the diaphragm until the field of view is free.

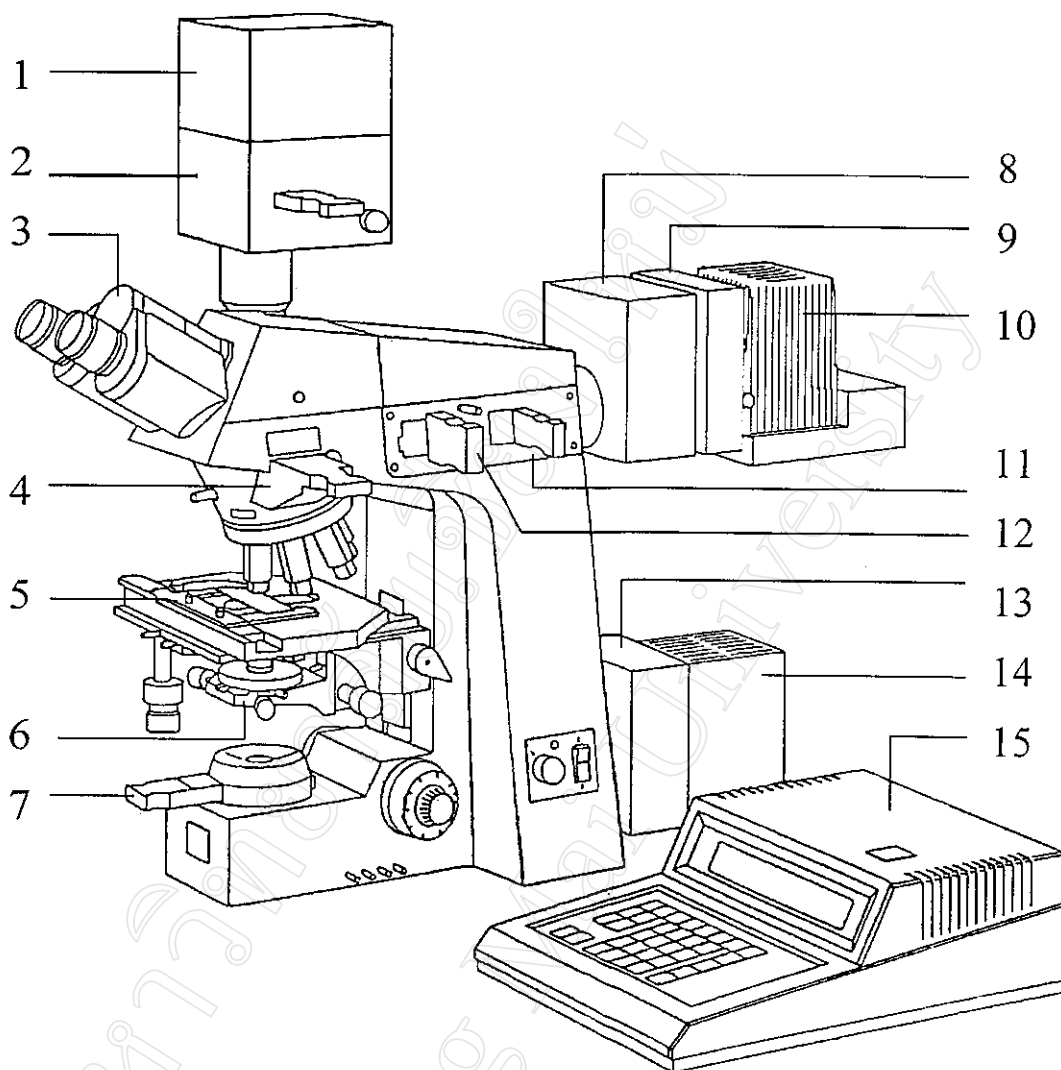


Figure 4.1 : MPM200 microscope photometer for fluorometry with axioplane universal microscope as base unit; 1. Detector unit with photomultiplier, 2. Photometer unit O with measuring diaphragm slider, 3. Binocular phototube with sliding prism 30/25P, 4. Reflector 3FL, 5. Mechanical stage 75x30R with low-mounted stage drive, 6. Condenser carrier Z with course and fine drive for vertical adjustment, 7. Luminous-field diaphragm slider inserted in luminous-field diaphragm attachment, 8. Electromagnetic 8-position filter changer, 9. Fast shutter, 10. Illuminator with HBO 100W/2 mercury lamp, 11. Incident light system FLP, 12. Slider with luminous-field diaphragm, 13. Light shutter, 14. Illuminator with 12V 100W halogen lamp, 15. MSP 20 microscope system processor (after Carl Zeiss Company, 1989).