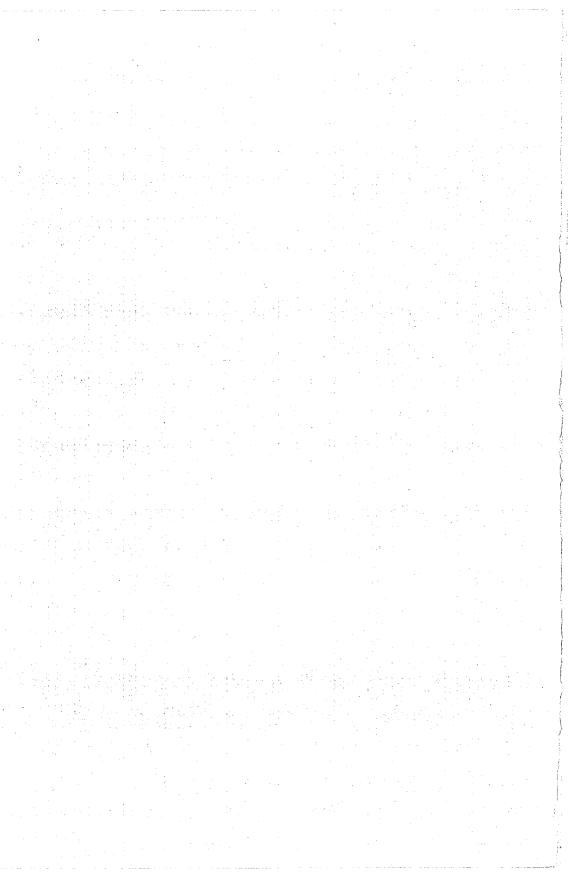


GEOLOGY STUDIES

Volume 22, Part 3—July 1976

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Brigham Young University Geology Studies

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Aspects of Coal Geology, Northwest Colorado Plateau Some Geologic Aspects of Coal Accumulation, Alteration, and Mining In Western North America: A Symposium

Papers prepared for presentation at a symposium at the annual meeting of the Coal Geology Division of the Geological Society of America, Salt Lake City, Utah, October 20, 1975, and adjunct papers pertinent to the annual field trip, October 17-19, 1975, in the Western Book Cliffs, Castle Valley, and parts of the Wasatch Plateau, Utah. The Field Guide and Road Log appears as Volume 22, Part 2—October 1975, Brigham Young University Geology Studies.

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The Fluorescence of Liptinite Macerals

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ABSTRACT.—An expanded nomenclature is presented for the liptinite (exinite) group of rosal macerals, including materials which have been mobilized following their incorporation into coal. More accurate measurement of the proportions of liptodetrinite, and better differentiation between macerals of the liptinite group, can be achieved using the
blue light fluorescence of these materials that occur in coals of up to high volatile A
bituminous rank. Such an approach will improve the understanding of the extent to
which liptinite macerals will be important as components of feedstocks for possible future conversion of western coals into liquid fuels.

Unaltered sporinite occurs in residues derived from coal hydrogenation experiments in which the vitrinite was partially converted. The fluorescence properties of the untreated and hydrogenated sporinite are compared.

INTRODUCTION

In two recent papers, M. Teichmüller (1974a, b) has named and characterized a number of materials present in coal which have not previously been properly identified or distinguished from other coal constituents. The fluorescence of these macerals is the property used most in identification.

New maceral names which have been proposed by Teichmüller are:

fluorinite

bituminite, and its higher-rank equivalent, meta bituminite exudatinite (Ger. exsudatinite), and its higher-rank equivalent, metaexudatinite

In addition, Teichmüller has described some of the phenomena by which the mobility of "bitumen" in coal is manifested when blue light irradiation is used for examination; these include "oil" exudations, smear films, and fluorescent vitrinites.

The optical properties of the three new macerals (summarized in Table 1, in figures 1 and 2, and below) reveal that these are quite different substances from the better-known liptinite (exinite) macerals, such as sporinite, resinite, and alginite.

Fluorinite

This pure organic substance has frequently been assumed by petrographers to be lenses or layers of clay minerals because of its black appearance, sometimes with internal reflections, in reflected light using oil immersion. However, Teichmüller has noted that this material has a strong yellow fluorescence when irradiated with blue light. Fluorinite has a maximum fluorescent intensity at a lower wavelength than other liptinite group macerals at the same level of rank. Consequently, the red/green quotient, that is, the ratio of relative intensity at 640 nm to relative intensity at 500 nm, is always lower than those of the other macerals. Figure 1 illustrates the low wavelength of the maximum intensity of fluorinite compared to bituminite and exudatinite.

TABLE 1

ORIGIN AND PROPERTIES OF NEW LIPTINITE GROUP MACERALS
(MODIFIED AFTER TEICHMÜLLER, 1974)

		BLUE LIGHT	E LIGHT						
MACERAL	ORIGIN	FORM	APPEARANCE IN REFLECTED LIGHT (OIL)	INTENSITY	COLOR	MAXIMUM FLUORÉSCENCE INTENSITY (A)	RED/GREEN QUOT IENT (Q)	ALTERATION OF FLUORESCENCE INTENSITY WITH TIME	
FLUORINITE	PLANT OILS FATS	LENSES	BLACK, OCCASIONAL INTERNAL REFLECTIONS	STRONG	BRILLIANT: YELLOW	510-570 NM	ca. 0.5	WEAK, EVEN NEGATIVE	
BITUMINITE	BACTERIAL LIPOIDS FATS PROTEINS	STREAKS AND AS GROUNDMASS	REFLECTANCE INTERMEDIATE BETWEEN VITRINITE & SPORINITE	WEAK	Orange to Brown	са, 635 мм	CA. 2.6	VERY STRONGLY POSITIVE	
EXUDATINITE	EXUDATES FROM LIPTINITES & VITRINITES	CAVITY FILLINGS	Black	VARIABLE	MOSTLY YELLOW TO ORANGE AND RED-BROWN	CA. 635 & 580 nm.	CA. 2.2	OFTEN AN IN- CREASE THEN DECREASE	

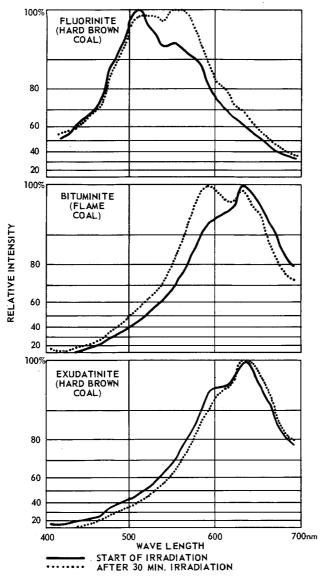
Bituminite

This is the most frequently occurring of the three new macerals. It is seen as irregularly-shaped shreds, wisps and layers of a material with reflectance intermediate between those of vitrinite and sporinite. Bituminite even may form the groundmass of some durites and sapropelic coals. Previously, it has often been identified as liptodetrinite, the attrital form of liptinite. However, Teichmüller notes that bituminite has a fluorescence property that clearly distinguishes it from all other macerals: its fluorescence intensity increases by as much as 200 percent after a 30-minute period of irradiation (figure 2). The usual fluorescence colors are orange to brown. The maximum fluorescence of bituminite occurs at a longer wavelength than those of other macerals. Teichmüller (1974a) has suggested that the generation of some micrinite in coals results from the diagenesis of bituminite.

Exudatinite

The manner of occurrence of exudatinite, once it has been identified, indicates that it is a secondary maceral which has been soft and mobile during the coalification process. Exudatinite appears black by reflected light in oil immersion, and it is only by the use of a dry objective or fluorescence illumination that what appeared to be empty cracks and cavities are seen to be filled with a material, which in blue light irradiation typically has an orange to yellow fluorescence. In some instances, the cracks containing exudatinite lead from primary liptinite macerals such as cutinite and resinite; the reflectance and fluorescence intensity of the exudatinite are respectively more or less than those of the primary liptinite occurrences.

Teichmüller observed that the maximum fluorescence of exudatinite is at a significantly different wavelength than that of sporinite, regardless of the rank of the coal. Other distinguishing features of exudatinite are its broad fluorescent maximum and a tendency to display an initial increase in fluorescence intensity, followed by a decrease.



Text-figure 1.—Typical fluorescence spectrum of fluorinite, bituminite, and exudatinite by long-wave ultraviolet irradiation (365 ± 30 nm). (after Teichmüller, 1974a)

MACERAL ANALYSIS

The identification of new liptinite macerals raises the question of the need to modify existing analytical methods for measuring maceral composition, to render them more accurate and complete.

The use of accessory equipment for fluorescence microscopy often enables

Sample PSOC	Seam	R _o	!	Macerals, Vol. % mmf								
No.	County, State	max	Illumination	٧	S	С	R	A	F	SF	MI	MA
105A	Indiana No. 1 Block, Owen Co., Ind.	0.53	W B	62 58	10 15	0	0	0	3	7	6	12 11
107	Top Vein, Brazil Block, Clay Co., Ind.	0.78	W B	71	12	0	0	0	5	3 2	4 3	5
122	Channel, No. 5. Block, Clay Co., W. Va.	0.75	W B	57 48	13 26	0	2	3	5	4	6	10 8
123	Lithotype, No. 5 Block, Clay Co., W. Va.	0.71	W B	27	24 33	0	2	0	15 11	4 3	5 4	13
124	Lithotype, No. 5 Block, Clay Co., W. Va.	0.55	W B	18 18	36 26	0	0	3	6	2 2	9	26 25
155	Lithotype, Unknown Seam, Kane Co., Utah	0.44	W B	90 74	2	0	0	0	4	0	1	3 2
181	Upper Block, Owen Co., Ind.	0.52	W B	55 39	9 33	0	1	0	13	4 3	6	12
182	Upper Block, Clay Co., Ind.	0.59	W B	64 53	10 26	0	2	0	3 2	9	4 3	- <u>8</u> 7
183	Upper Block, Clay Co., Ind.	0.59	- W B	61 50	6 21	0	1	0	9	5	5	13 10
184	Upper & Lower Block, Clay Co., Ind.	0.54	W B	65 55	11 23	0	2	0	6	8	2	<u>6</u> 5
185	Indiana No. 6, Sullivan Co., Ind.	0.53	W B	81 78	5 8	0	1	0	3	3	0	7
239	B Seam, Carbon Co Utah	0.60	₩ B	86 84	4	0	1	0	2	3	2	2

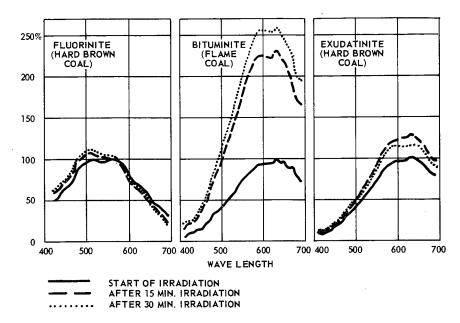
TABLE 2
A COMPARISON OF CONVENTIONAL AND FLUORESCENCE PETROGRAPHIC ANALYSES

W: white light; B: blue light irradiation; V: vitrinite; S: sporinite; C: cutinite; R: resinite; A: alginite; F: fusinite; SF: semifusinite; MI: micrinite; MA: macrinite

distinctions to be made between liptinite macerals which might appear uniformly dark in reflected white light. Plate I, figure 2, makes clear the liptinite maceral boundaries and some internal structures not as well defined in the corresponding white light photomicrograph, Plate I, figure 1. White light and blue light irradiation photomicrographs of a thin section of the same coal are shown in Plate I, figures 3 and 4; figure 4 illustrates the difference in fluorescence intensity between alginite on one hand and sporinite and cutinite on the other.

When blue light irradiation is used during petrographic analysis, the percentages of the fluorescent liptinite macerals are often greater than when conventional white light is used. Identification is made more easily by observing the fluorescence colors on a dark background than by observing the dark reflectances on medium background in white light. Table 2 provides a comparison of selected analyses in both white and blue light, showing the larger contents of liptinite macerals which were recorded in blue light. Some analyses (e.g. PSOC-124) show how the use of blue light has enabled individual liptinite macerals to be differentiated more readily even though the total content of these macerals remains approximately the same as that determined in white light. In the fluorescence analysis, the percentages of liptinite macerals were determined in blue light; the balance due to nonfluorescing vitrinite and inertinite group macerals was apportioned according to the results of white light analysis.

No attempt was made to identify or count any of the new liptinite macerals in the analyses referred to above. However, the method could be adapted to include these materials.



Text-figure 2.—Alteration of intensity of fluorinite, bituminite, and exudatinite during 30 min. long-wave ultraviolet radiation (365 ± 30 nm). (After Teichmüller, 1974a)

Individual members of the liptinite group have several characteristics in common: physical similarities, such as color in transmitted light and low reflectance; broad chemical affinities, such as relatively high hydrogen and volatile contents; and a higher contribution by aliphatic substances than is found in other maceral groups. However, these members also represent coal components which have very diverse origins, including spore and pollen exines, cuticles, algae, and a range of resinous substances and tannin derivatives.

The origin of fluorinite, bituminite, and exudatinite (Teichmüller, 1974a) is much less clear, although some appear to be derived from previously existing primary liptinite macerals. Others may have originated in part from waxes, oil, fats, proteins, algae, and bacterial lipoids (see Table 1); Teichmüller suggests that even humic substances may have contributed to the generation of bituminite. A more thorough study is needed of the different physical and chemical properties of liptinites, partly in order to determine the relative importance of each maceral in industrial processes such as liquefaction. The techniques of spectrophotometry (van Gijzel, 1971; Ottenjann et al., 1974; Teichmüller, 1974a; Ting and Lo, 1975) have already begun to elucidate some of the unique characteristics of individual liptinite macerals. Further understanding may be gained by the application of other techniques such as Fourier-Transform infrared spectroscopy for in situ analysis of these substances.

OCCURRENCES OF NEW LIPTINITE MACERALS IN U.S. COALS

A microscope survey has been made of several U.S. coals, particularly those of the western states. The following descriptions have been assembled,

EXPLANATION OF PLATE I

- Fig. 1.—Alginite, sporinite, and fragments of vitrinite and fusinite (white) in finegrained matrix containing bituminite and micrinite (white specks). No. 5 Block seam, Clay County, West Virginia. Incident white light, oil immersion. x440
- Fig. 2.—The same field. Alginite and sporinite fluoresce yellow and the bituminite brown. Incident light, blue irradiation, oil immersion. x440
- Fig. 3.—Alginite (white, center field), sporinite and cutinite (pale yellow), and vitrinite in groundmass containing bituminite and liptodetrinite. No. 5 Block seam, Clay County, West Virginia. Transmitted white light. x440
- Fig. 4.—The same field. Transmitted light, blue irradiation. x440
- Fig. 5.—Lens of ?fluorinite (black) in center field. Black Mesa coal, Arizona. Incident white light, oil immersion. x440
- Fig. 6.—The same field. Note the bright yellow fluorescence of the ?fluorinite. Incident light, blue irradiation, oil immersion, x440
- Fig. 7.—Bituminite- and liptodetrinite-rich particle in lower left field contrasts with liptinite-poor particle in upper right. Unnamed seam, Cannel King mine, Kane County, Utah. Incident light, blue irradiation, oil immersion. x440
- Fig. 8.—Orange-brown fluorescing bituminite in upper part of the field. Santo Tomas cannel coal, Webb County, Texas. x670

EXPLANATION OF PLATE II

- Fig. 1.—?Cutinite (dark grey) is apparently bordered by black bituminite which has permeated the surrounding vitrinite (light grey). Unnamed seam, southwestern U.S.A. Incident white light, oil immersion. x440
- Fig. 2.—The same field. Note the diffuse yellow fluorescence of the ?bituminite. Incident light, blue irradiation, oil immersion. x440
- Fig. 3.—The chambers of the sclerotia (light grey) appear to be empty. Blue seam (lower split), McKinley County, New Mexico. Incident white light, oil immersion. x440
- Fig. 4.—The same field. The chambers of the sclerotinite are infilled with orange-fluorescing exudatinite. Incident light, blue irradiation, oil immersion. x440
- Fig. 5.—Particle of fusinite with apparently empty cell lumens. Unnamed seam, Cannel King mine, Kane County, Utah. Incident white light, oil immersion. x200
- Fig. 6.—The same field. Note the bright yellowish exudatinite filling the cell cavities. Incident light, blue irradiation, oil immersion. x200
- Fig. 7.—Note the orange-fluorescing apophyses (exidatinite) on primary liptinite which fluoresce yellow. Blue seam (Lower split), McKinley County, New Mexico. Incident light, blue irradiation, oil immersion. x200
- Fig. 8.—Wedge-shaped apophyses of orange-fluorescing exudatinite leading from lighter fluorescing resinite. Blue seam (lower split), McKinley County, New Mexico. Incident light, blue irradiation, oil immersion. x440

PLATE I

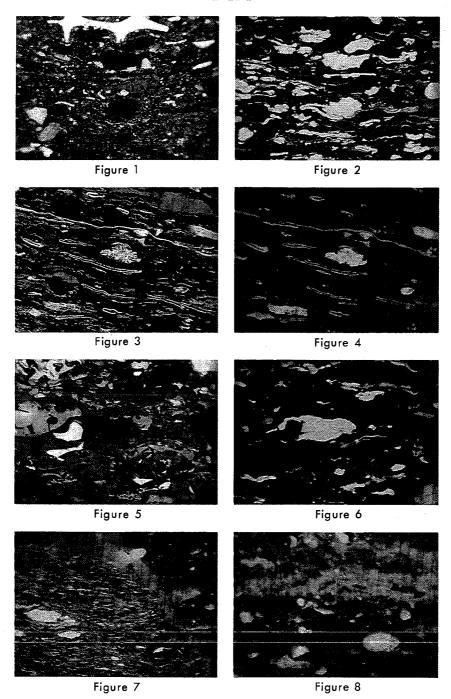


PLATE II

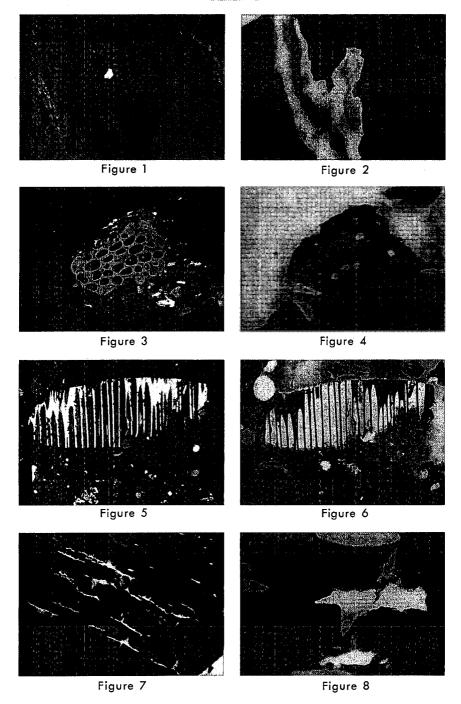
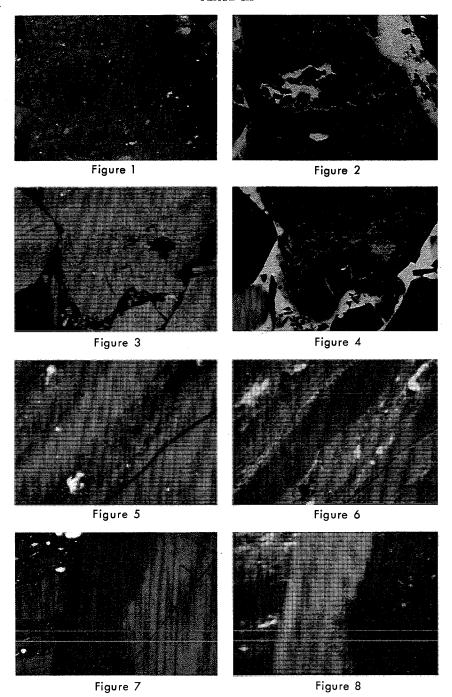
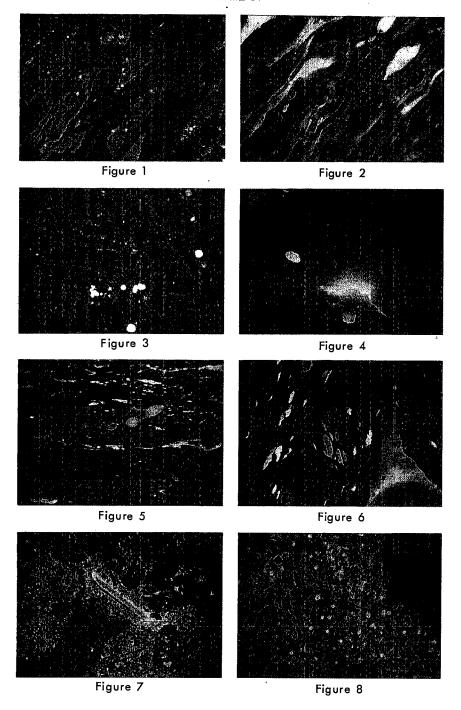


PLATE III



W. SPACKMAN, A. DAVIS, G. D. MITCHELL

PLATE IV



EXPLANATION OF PLATE III

- Fig. 1.—Exudatinite accumulations around corpocollinite (round, grey bodies). Blue seam (lower split), McKinley County, New Mexico. Incident white light, oil immersion. x200
- Fig. 2.—The same field. The exudatinite fluoresces orange to yellow. Incident light, blue irradiation, oil immersion. x200
- Fig. 3.—The exudatinite filling cracks and lining the lower left margin of vitrinite particle is barely detectable. Unknown seam, Big mine, Gunnison County, Colorado. Incident white light, oil immersion. x200
- Fig. 4.—The same field. The exudatinite fluoresces orange. Incident light, blue irradiation, oil immersion, x200
- Fig. 5.—The attrital bands within this vitrain appear darker, probably as a result of previous irradiation. B seam (B2 bed), Gunnison County, Colorado. Incident white light, oil immersion. x670
- Fig. 6.—The same field. Note the slight fluorescence of the attrital vitrinite. Incident light, blue irradiation, oil immersion. x670
- Fig. 7.—Dark "smear film" resulting from blue light irradiation. B seam (B2 bed), Gunnison County, Colorado. Incident white light, oil immersion. x440
- Fig. 8.—The same field. Note the fluorescence of the "smear film." Incident light, blue irradiation, oil immersion. x440

EXPLANATION OF PLATE IV

- Fig. 1.—Coalified secondary xylem in lignite. Vessels and fiber-tracheids appear darker grey, ray parenchyma is lighter grey. Zap seam, Mercer County, North Dakota. Incident white light, oil immersion. x440
- Fig. 2.—The vessels and tracheids show varying fluorescence from yellow to brown; the parenchyma appears black. Incident light, blue irradiation, oil immersion. x440
- FIG. 3.—Vitrinite particle with pyrite spheres in vicinity of horizontal fracture. The dark oval body (left, center) is resinite. Big D seam, Lewis County, Washington. Incident white light, oil immersion. x440
- Fig. 4.—Yellow fluorescence of exudation which diffused from the crack during examination. Incident light, blue irradiation, oil immersion, x440
- Fig. 5.—Crack across coal particle has exuded a dark reflecting substance which is seen here as pale yellow fluorescing droplets. Illinois No. 6 seam, Saline County, Illinois. Incident light, blue irradiation, oil immersion. x440
 - Fig. 6.—Residue of Cretaceous cannel coal (PSOC-155), hydrogenated at 300°C. Exudatinite (orange) filling fusinite lumens, resinite, alginite and bituminite (not shown) are essentially unreacted. Incident light, blue irradiation, oil immersion. x200
 - Fig. 7.—Residue of the same coal hydrogenated at 375°C. Elongate fluorescent substance in center field is thought to be immiscible plastic phase produced from alginite, showing slight solubility in the epoxy binder at its perimeter. Incident light, blue irradiation, oil immersion. x200
 - Fig. 8.—Residue of the same coal hydrogenated at 400° C. The small (generally less than 1μ) fluorescent spheres are alginite-derived immiscible phase. Incident light, blue irradiation, oil immersion. x440.

not as a detailed record of new macerals, but as examples of the types of occurrences observed. The descriptions also help emphasize that the new macerals may be found in a range of commonly known and sometimes important coal seams.

Examinations were conducted on samples known to contain relatively high total liptinite maceral contents. In most instances, polished coal pellet surfaces were viewed in incident light, but some thin sections were also studied.

The equipment used was a Leitz Ortholux indicent light microscope with illumination provided by an ultra-high-pressure mercury lamp; the exitation filter, BG 12 (3 mm), with a maximum transmission at about 400 nm, was used with a red suppression filter, BG 38. A dichroic mirror, LP 510, replaced the semi-silvered reflector in the vertical illuminator, and a barrier filter with maximum transmittance at 530 nm was inserted above the vertical illuminator. Examinations were made using a X10 dry objective and X25 and X60 oil immersion objectives.

Fluorinite

In this study of North American coals, fluorinite was observed only rarely. However, this lack may be simply the result of a biased sampling, since most of the coals examined were durainic, and fluorinite characteristically occurs in clarainic coals. Plate I, figure 6 shows the bright yellowish fluorescence in blue irradiation of what in the corresponding white light photomicrograph (Figure 5) resembles a claylike lens occurring in a subbituminous seam from the Black Mesa field, Arizona. Teichmüller (1974a) reported fluorinite from the Herrin No. 6 seam, Old Ben 24 Mine, Illinois.

Bituminite

Excellent examples of bituminite occurrences have been observed in several canneloid coals. A photomicrograph representing the No. 5 Block coal from Clay County, West Virginia, a high volatile A bituminous seam (Plate I, figures 1 and 2) shows alginite occurring in a bituminite groundmass, which has a brownish fluorescence in figure 2. In figure 1, some white specks of micrinite can be detected in the groundmass. Teichmüller (1974a) has suggested that the micrinite might be generated as a result of the diagenesis of bituminite, but its incorporation into such an attrital matrix might conceivably have taken place by ordinary sedimentary processes.

Plate I, figure 7 shows the relative fluorescence of bituminite-rich (lower left) and bituminite-poor particles in a canneloid horizon in an unnamed seam from the abandoned Cannel King mine, Kane County, Utah. An orange and brownish fluorescing groundmass is seen in a photomicrograph of Eccene cannel coal from the Santo Tomas district, Webb County, Texas (Plate I,

rigure 8).

Plate II, figures 1 and 2, show the apparent development of bituminite lying outside the original margin of some cutinite, having permeated the surrounding vitrinite. Newton's rings, which are often associated with bituminite occurrences, are present but not clear in the photomicrograph.

Exudatinite

The cell lumina of fusinite or semifusinite and the chambers of sclerotinite frequently provide the cavities for exudatinite infillings. Infillings in

sclerotia have been observed in the high volatile C bituminous Blue seam of New Mexico (Plate II, figures 3 and 4), the Utah B seam, and coal from the Black Mesa field, Arizona. The fluorescence colors displayed by these infillings were orange or orange-yellow in color. A reddish fluorescence was observed in some lumen fillings in the Zap seam of North Dakota.

Plate II, figures 5 and 6, display longitudinal sections of fusinized cells with the lumina occupied by an orange-yellow fluorescing exudatinite. The coal

is an unnamed seam of Kane County, Utah.

Cracks which occur in vitrinite are frequently the loci for exudatinite deposition. Sometimes these cracks diverge as "cross-bedding" apophyses from resinite and other primary liptinite macerals. The mobility of resinite in the later stages of coalification is a phenomenon which has attracted comment in the past. Now, Teichmüller has reported that some of the material occurring in this manner has different optical properties than the primary substances. Plate II, figure 7 shows a number of vertical apophyses which are darker in fluorescence than the liptinite bodies from which they have exuded; the phenomenon was observed in the Blue seam from New Mexico. Plate II, figure 8 shows some large wedge-shaped apophyses of a similar appearing exudate from a resin body in the same seam.

Other occurrences of exudatinite are associated with corpocollinites, when they may be observed around the peripheries of, and accumulated between, these rounded vitrinitic bodies. An example is provided in Plate III, figures 1 and 2, from the Blue seam, New Mexico.

In an example from an unknown seam, abandoned Big mine, Gunnison County, Colorado, some vitrain particles are covered by a network of fine cracks and holes which are filled with orange-fluorescing exudatinite, some of which, in Plate III, figures 3 and 4, lines the lower left margin of the central

particle.

Teichmüller (1974b) reported that the polyester resin which she used to embed her coal samples could dissolve "bitumen" from the coal. This dissolution was accompanied by a marked increase in the fluorescence of the polyester resin. Several of the coal pellets examined during the present study showed the same effect and, in one instance, a portion of an orange-fluorescing crack-filling had been dissolved by the epoxy resin to flow out of the crack as a fluorescing cloud.

MANIFESTATIONS OF MOBILE "BITUMEN"

"Smear Films" and Fluorescent Vitrinites

A few examples of vitrinites which can display a faint to moderate fluorescence have been encountered in U.S. coals. Figures 5 and 6, Plate III, show the banded vitrain from the B seam, a high volatile B bituminous coal from Colorado; the more attrital layers have the strongest fluorescence. In white light these layers are somewhat darker than the other bands, probably as a result of the development of exuded "smear films" as described below.

Across the base of one of the lighter-fluorescing bands of Plate III, figure 6, can be seen two lines of regularly spaced bright specks which seem to represent the characteristic bordered pits of vessel walls. Such an indication of a contribution to the fluorescent constituents of coals by woody cell wall substances is rare but significant.

Figures 7 and 8, Plate III, illustrate the development of "smear films" (see Teichmüller, 1974b) during actual irradiation of the specimen. The dark area on the left of the right-hand particle in Figure 7 represents the edges of two fields which had been examined in blue light for some minutes prior to the taking of the photomicrograph, and which had acquired the lower reflectance in response to the irradiation. The relatively higher level of fluorescence of these films, which Teichmüller has suggested are exuded bitu-

men, is apparent in figure 8.

In the Zap seam, North Dakota, a number of fluorescent zones can be distinguished clearly in blue light (Plate IV, figure 2) but are barely detectable in white light (Plate IV, figure 1). The photomicrograph is of secondary xylem, the fiber-tracheids and vessels displaying moderate fluorescence in shades of light brown. Ray parenchyma cells, in tangential section, appear light grey in reflected white light, and very dark in blue light. The yellow fluorescing areas may be resinous cell fillings, although they are very similar in reflectance to the vitrinitic (huminitic) areas. The same kind of differentiation of vitrinitic areas into distinct zones of fluorescence has been observed in the case of Whitby jet from the Upper Lias, Port Mulgrave, Yorkshire. Fluorescent vitrinite was observed in the No. 80 seam, Hanna, Wyoming.

The apparent differences in "bitumen" content that are sometimes evident between attrital and nonattrital vitrain bands may be significant in regard to observed differences in carbonization behavior of "vitrinite A and B" (Brown et al., 1964). Teichmüller (1974b) has already remarked on the high degree of plasticity of bitumen-rich coals, and on the fluorescence of desmocollinite, the attrital form of vitrinite which has also been called vitrinite B; these observations may be used to account for the better caking characteristics of vitrinite B, and for the poor caking behavior of some "pseudovitrinite" (see Benedict

et al., 1968).

The possible importance of "bitumen"-rich coals in liquefaction processes has also been noted by Teichmüller (1974b). Results of hydrogenation experiments performed by the U.S. Bureau of Mines (Fisher et al., 1945) and in work by The Pennsylvania State University in cooperation with Gulf Research & Development Company (Davis et al., 1974) indicate that the coals which produce the highest yields in liquefaction experiments are generally of high volatile bituminous rank; coals of lower rank give a more varied response, and higher rank coals give significantly lower yields. A parallel may be drawn between this behavior and the development of plasticity in caking coals and the distribution of hydrogen-rich "bitumens" in coal.

"Oil" Exudations

During examination in blue irradiation, certain coal samples evolved a substance which fluoresced a bright yellow before fading and dispersing in the immersion oil. The onset of this phenomenon was usually at the commencement of examination, and the suffuse fluorescence originating from a crack or hole within the coal increased rapidly in size and intensity before fading within a time period of 10 to 20 minutes. Figures 3 and 4, Plate IV, show the appearance of one of these exudations which has originated in the vicinity of some pyrite spherules. The exudation is barely discernible in white light. The photomicrograph shown is of the Big D seam, Washington; similar phenomena were observed in the B seam, Utah.

Other exudations onto the polished surface of coal pellets (see Teichmüller, 1974b) are apparent as brownish areas, sometimes with Newton's rings. Figure 5, Plate IV, shows the pale yellow fluorescing droplets which have exuded along a crack in a specimen from the Illinois No. 6 seam, Saline County, Illinois.

The fluorescence displayed by some clay partings in coal seams may be

due to absorption of oil generated by liptinitic substances.

PROGRESSIVE LIQUEFACTION EXPERIMENTS

In this phase of our investigation, the fluorescence of some liptinite macerals was used as an aid in understanding their relative reactivities during liquefaction. The macerals studied were exudatinite, bituminite, resinite, and alginite, all of which occur in significant quantities in the coal (PSOC-155, Unnamed Cretaceous cannel coal, Utah; see Table 2) used in these experiments.

The present series of liquefaction experiments used 20 g of minus 20 mesh coal with 4 to 1 parts by weight of tetralin as a hydrogen-donating vehicle in a nitrogen atmosphere. The stainless steel reaction vessel was heated by means of a fluidized sand bath for three hours at a selected constant temperature (see Given et al., 1974). Runs were made at 25° intervals from 300° to 450°C. At the completion of each run, the vessel contents were extracted in benzene for 48 hours, and the residues dried, embedded in epoxy resin, and polished.

In experiments reported by Given et al. (1974), a large increase in rate of conversion took place between 350° to 400°C, accompanied by a major disintegration of coal grains. Because of their frequent enclosure in a matrix of other constituents, there would be much less opportunity for the liptinite macerals to contact the tetralin vehicle prior to 350°C, so that dissolution should be minimal.

In support of the preceding remarks, Plate IV, figure 6 exhibits three fluorescent macerals (exudatinite, resinite and alginite) in the liquefaction residue of PSOC-155 from the run made at 300°C. Exudatinite is the only liptinite maceral which solvates at 325°C, well below the temperature of particle disintegration. With an increase in temperature to 350°C, bituminite and resinite acquire a granular texture and exhibit a decrease in fluorescence intensity. Unlike exudatinite, their complete solvation is dependent upon particle disintegration, which in these experiments took place at 375°C.

Alginite appears to be unique in that it produces a microscopically-distinct immiscible liquid phase during the 375° to 400°C interval. As shown in Plate IV, figure 7, some alginite becomes plastic to form a porous, soft, and oily substance. In many of the liquefaction residues, one can see the juxtaposition of unreacted alginite and the oil remnant, both showing approximately the same fluorescence intensities. On close observation an increase in yellow fluorescence of the epoxy binder can be detected, perhaps indicating solvation but incomplete hydrogenation of the alginitic oil. In the residue from the run made at 400°C, the alginite-derived immiscible phase has decreased in size (Plate IV, figure 8). Presumably, as temperatures are increased above 400°C, more complete hydrogenation of alginite would be attained.

The observation of fluorescence at all temperature stages of liquefaction contrasts with that made by Ting and Lo (1975) who reported the absence

of fluorescent macerals in coal samples which were heated above 350°C in a retort at atmospheric pressure.

SUMMARY AND CONCLUSIONS

- 1. A number of recently described macerals, whose existence is perceived best by examination of fluorescence properties, have been identified in several U.S. coals.
- 2. Increased amounts of liptinite macerals have been recorded during point-count analysis of some coals when blue light irradiation was used instead of the conventional white light examination. A procedure which uses the results of white light and blue light analyses in combination is recommended for coals which have high liptinite contents.
- 3. Certain bands of attrital vitrinite (desmocollinite, "vitrinite B") fluoresce more than the associated thick bands of nonattrital vitrinite (telocollinite, "vitrinite A," "pseudovitrinite"). This demonstration of the relatively greater "bitumen" contents of the attrital layers may explain their higher degrees of plasticity during carbonization. A similar explanation might be considered for the poor coking characteristics of some coals rich in "pseudovitrinite."
- 4. Coals of high volatile bituminous rank frequently have given the best response in liquefaction experiments. The coincidence of the maximum development of hydrogen-rich "bitumens" at this same rank level seems to be significant.
- 5. In a series of hydrogenation experiments, the liptinitic macerals retained their fluorescence properties until solvation. Examination of liquefaction residues in blue light enabled the following observations to be made:
- a) The secondary maceral, exudatinite, reacts before other liptinitic macerals and well in advance of the physical disintegration of coal particles.
- b) The solvation of bituminite and of resinite during batch liquefaction experiments were coincident at about 375°C. It was observed that neither maceral was completely solvated, but that a submicron size granular insoluble residue was produced, with a structure similar to micrinite.
- c) Alginite is the maceral which is most resistant to hydrogenation. At 375°C it produced droplets of a liquid phase which was immiscible with the vehicle; in runs made at higher temperatures, the droplets were progressively broken down and dispersed.

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