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KEROGEN-ISOLATION METHOD--A STUDY WITH KEROGEN DATA FROM SEDIMENTARY ROCKS

By

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INTRODUCTION

This research reports a method for rapid isolation of kerogen from sedimentary rocks and on some chemical and physical characteristics of solid organic matter in sediments.

Kerogen in this report refers to that fraction of organic matter in rocks that is not soluble in solvents ordinarily used for extraction of organic matter such as methanol, benzene, chloroform, acetone, and alkaline solutions of sodium or ammonium hydroxides. Because kerogen is immobile, it provides information on the type of organic matter in sediments and the conditions at original deposition with subsequent changes in the rocks. In contrast to kerogen, the extractable organic material is susceptible to migration.

The changes in kerogen from three areas (Idaho-Wyoming-Utah Phosphoria Formation, Anadarko Basin, and southern California Borderlands) were examined as a function of increased depth or temperature. These changes allow the evolutionary stage of the rock history to be inferred. These changes allow an assessment of the effectiveness of certain fine-grained rocks as petroleum source rocks.

In any comprehensive organic geochemical investigation of fine-grained rocks, kerogen analyses should be included; they can yield decisive information. Such information generally can aid in predicting if a frontier area is capable of producing hydrocarbons and what type.

Kerogens isolated from sediments were normally very fine, soft powders that varied from dark brown to jet black in color; many gave the appearance of coal dust. Elemental analyses show that carbon, hydrogen, and oxygen are the major constituents of kerogen (Forsman and Hunt, 1958). The elemental composition of kerogen before maturation reflects its origin from a wide variety of plants and perhaps even animals. The change in elemental composition of a given type of kerogen with depth is a very good indicator of the degree of thermal maturation of petroleum source rocks.

There is evidence that at least two, and possibly three, types of kerogen are found in sedimentary rocks. The three main types are coaly, algal, and coaly-oil shale (possibly an admixture of the coaly and algal types) (Forsman, 1963).

The amount of hydrocarbons that kerogen can generate is a function of the amount of hydrogen present in the kerogen (McIver, 1967). Metamorphism of organic matter caused by heat, changes the solid organic network partly to mobile products (petroleum generation), resulting in natural gas, oil, and condensate. During late-stage diagenesis, organic substances are converted to dry gas and pyrobituems, and ultimately to graphite under severe metamorphic conditions (Staplin, 1969). The major organic maturation levels which can be indicated by kerogen analyses are: 1) immature, which contains medium to heavy gravity oil and dry gas; 2) mature, which contains condensate, wet gas (C_1-C_4), and medium- to light oil; and 3) metamorphic, recognized by dry gas from drill bit cuttings and the presence of solid black organic matter (Evans and Staplin, 1971).

The three techniques most commonly used in estimating organic maturity (thermal alteration) of kerogen are:

1. Microscopic examination of the color alteration of kerogen (Staplin, 1969).
2. Reflectance of vitrinite (Dow, 1977).
3. Elemental analysis of kerogen (Tissot and others, 1974).

In this study color alteration and elemental analyses of kerogen were employed.

Changes are observed in the elemental analysis of kerogen with maturation. These changes are due to hydrogen, oxygen, and other elements being lost from the kerogen. Hydrogen is lost by formation of H_2O, formation of H_2S, and formation of methane and liquid hydrocarbons. Oxygen is lost from the formation of CO_2 and H_2O. These losses ultimately cause changes in the physical properties of kerogen, and these changes can be measured. However, subjectivity is involved in some of the measurements of physical and chemical properties.

A variety of isolation methods are used to obtain a relatively clean kerogen concentrate. Many investigators are interested in the changes of organic matter that occur as a function of maturation and, therefore, wish to minimize changes introduced by the isolation techniques (Forsman, 1963; LaPlante, 1974; Powell and others, 1975; Staplin, 1969). The method of isolation methods depends on the objectives of the study. Chemical isolation can, and will, alter some of the peripheral organic constituents of kerogen. This can be limited by carefully choosing the chemicals for releasing the organics from rock matrices and regulating the time of sample exposure to chemicals when it has been established that undesired changes are likely (Saxby, 1970). Physical isolation methods can be used to obtain the kerogen in rock samples without the small changes that accompany chemical methods (Forsman, 1963). Most of the physical methods result in high ash kerogen concentrate.

The kerogen isolation method used in this study encompasses basic techniques described in the literature for paleontological work as well as other methods. These techniques have been combined into a rapid method for kerogen isolation that is easily adjustable for specific studies when needed.

EXPERIMENTAL PROCEDURES

All rock samples used in this study had previously been ground to pass a 100-mesh sieve for solvent (soxhlet) extraction. The extracted rock samples were allowed to air dry after extraction, and then 20 g of rock sample was used for kerogen isolation (see flow sheet, fig. 1). The general procedure was carried out in well-ventilated hoods, in sets of eight samples as follows:

HCl treatments

The 20-g samples were placed into separate 250-mL polypropylene beakers. Each was checked with a few drops of 1N HCl for calcium carbonate. If calcium carbonate was present, more 1N HCl was added, with stirring, until all visible bubbling stopped. The samples with HCl were stirred intermittently during working hours and allowed to stand over night. Each sample was checked with a few drops of 6N HCl for further reaction. If the response was positive, that sample was treated with more HCl until no further reaction occurred. When the response was negative, the samples, still in the 250 mL beaker and in the acid pH range, were placed on a waterbath at 50°C for 25 to 30 minutes. This procedure removed dolomite in moderate amounts.

After the HCl treatment all calcium ions must be removed from the sample solutions by "washing". Each 250 mL beaker was filled with tap water and the agitated sample was allowed to settle completely. The water was removed by aspiration, without disturbing the water-sample interface, into a reservoir trap. This was repeated a minimum of four times (the actual number of washes depends on each sample), and the last wash solution was checked with pH paper. When the pH of the wash solution matched the pH of the tap water used, the samples were assumed to be free of calcium ions that could form the

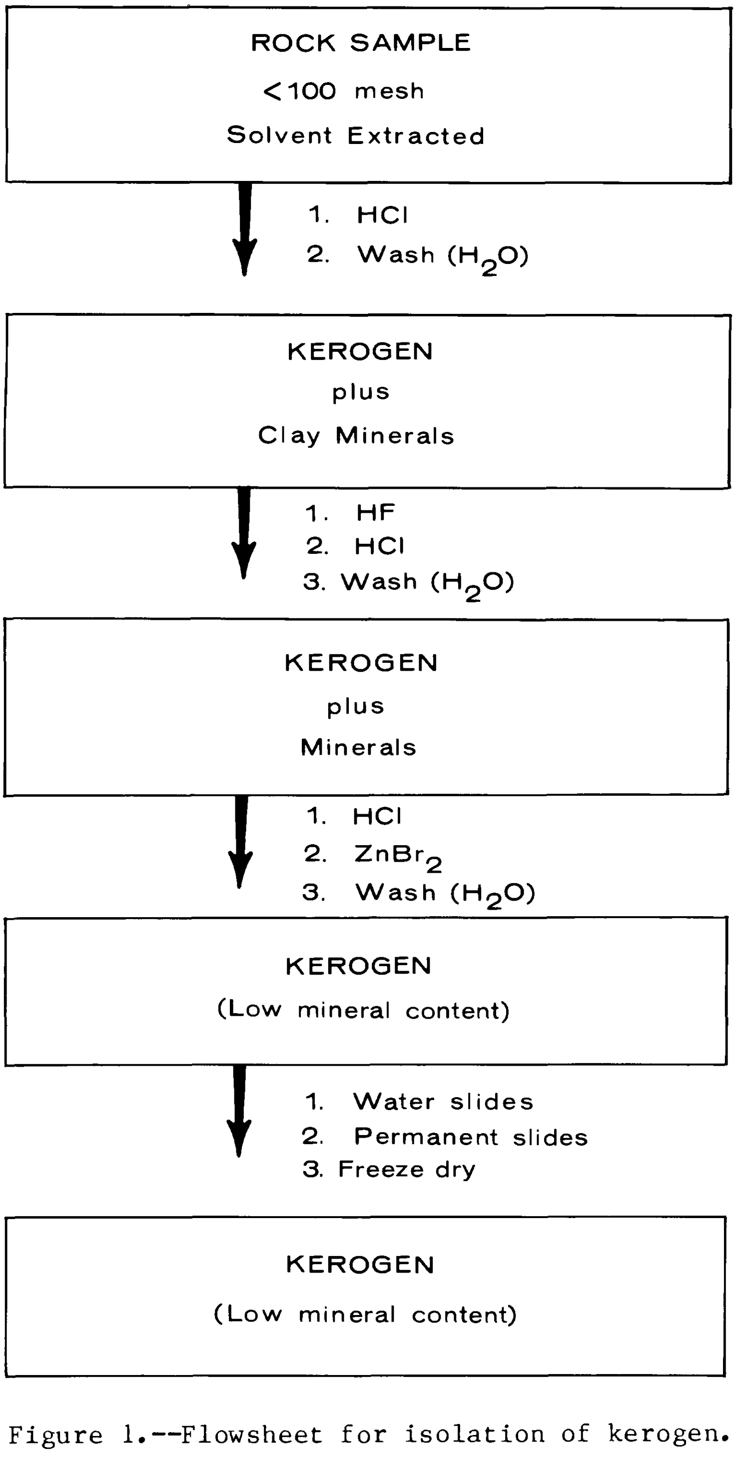

Figure 1.--Flowsheet for isolation of kerogen.

insoluble CaF_2, $K_{sp} = 3.4 \times 10^{-11}$, with fluoride ion. Complete removal of Ca^{++} is necessary before adding HF solution.

HF treatment

The samples were acidified with 15 mL 1N HCl to keep in solution those flouride compounds easily precipitated during this step. Hydrofluoric acid (48 percent) was added with stirring to the samples, in small quantities, to the original 250-mL beakers until vigorous reactions ceased, indicated by cessation of bubbling in the beakers. The beakers were filled with water and left overnight. The samples were washed, following the procedure previously described for HCl removal, then each sample was transferred to a 100 mL polycarbonate centrifuge tube.

$ZnBr_2$ flotation

Saturated, commercial solutions of $ZnBr_2$, or laboratory prepared $ZnCl_2$ were diluted with distilled water to a specific gravity of 2.0 to 2.1 and stored in 1 gallon containers for use as needed. Before adding the $ZnBr_2$ solution to the kerogen isolates, the samples were acidified (and agitated) with about 10mL of 1N HCl.[1] The acidified samples were centrifuged in the 100-mL tubes and the HCl solution was decanted to remove as much liquid as possible. $ZnBr_2$ was added to a depth of 2 mm above the sample, and the sample was thoroughly agitated on a vari-whirl mixer. The sample was poured into a 50-mL centrifuge tube, and the 100-mL tube was rinsed with $ZnBr_2$ to transfer all of the sample, then the samples were centrifuged for 30 minutes at 2,500 rpm, or longer at lower speeds. After centrifuging, the $ZnBr_2$ solution with

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[1] An alternate procedure used saturated $ZnCl_2$ solutions in place of $ZnBr_2$.

the floated kerogen was poured into the original 100-mL tube. The 50-mL tube was washed with approximately 5 mL 1N HCl which readily removes all remaining kerogen float without distrurbing the sediment. Fifteen mL of 1N HCl was added to the $ZnBr_2$ solution with the floated kerogen, to prevent precipitating $Zn(OH)_2$ during the next purification stage.

The kerogen suspension was mixed with water, then the tubes were centrifuged, and the supernatant liquid decanted. Twenty mL of 1N HCl was added to the first washing. Water was added to the tubes and the process repeated. A minimum of six washings was required. A small amount of the kerogen was set aside after the last washing for slide preparation for a visual check of mineral content, and the bulk of the material was prepared for freeze drying.[2]

Slide preparation

Two methods were used to prepare permanent slides. In the first method, a clean glass slide was heated to 50°C on a hotplate. This allowed caedax, the mounting medium, mobility under a cover slip when applied. A few drops of distilled water with kerogen were put onto the slide and allowed to completely dry. Caedax was added to the slide, by the drop, until the dried kerogen was covered. When the caedax was fluid enough, the dried kerogen was scratched from the slide surface and thoroughly mixed in the caedax on the slide. A

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[2] Four samples were processed using the kerogen isolation technique described here. That portion that sank in the $ZnBr_2$ flotation procedure and some of the floated kerogen were analyzed in another laboratory by stepwise pyrolysis--gas chromatography. The chromatograms of the two fractions were identical, which suggests that a good representation of particulate OM is contained in the floated kerogen (J. S. Leventhal, oral commun., 1977).

heated cover slip was immediately dropped onto the mixture. Enough pressure was applied to remove bubbles and extend the mixture to the edges of the cover slip.

The correct amount of caedax and kerogen can be judged accurately after a few trials. The caedax starts to set after a few minutes, and the slide can be removed from the hotplate. Excess mounting medium was removed with a clean razor blade, and slides were cleaned with xylene, a caedax solvent, immediately rinsed with methanol, and dried with paper tissues. The finished slide was labeled and placed on the slide warmer to continue curing.

The second method required more time and equipment. Solutions of methanol and water (25, 50, 75, and 100 percent methanol by volume) were used to dry kerogen samples. A small amount of kerogen was placed in a 15-mL centrifuge tube and washed once with each of the methanol-water solutions in the order given above. After the 100 percent methanol wash, the sample was washed once with a mixture of xylene:methanol, 50 percent by volume, for a water test. If this wash was milky, to any degree, water was present, and the methanol:water washes were repeated. Water must be excluded from permanent slides. When the kerogen passed the water test, caedax diluted with xylene was added to the kerogen and the mixture was transferred to a 0.5 dram screw cap vial. Slide preparation was similar to the above method. Complete curing takes 1 to 2 days when solvent volumes are adjusted properly.

Slide preparation by the first method is much quicker, but the second method of slide preparation allows one to store kerogen in the mounting medium for future slide preparation.

Freeze-drying

More than 99 percent of the kerogen from the $ZnBr_2$ flotation was freeze-dried. The kerogen was transferred with distilled water into 2- or 8-dram screw cap vials, dependent on the amount of kerogen. The vials were centrifuged at very low speeds to remove excess water, the 2-dram vials in a clinical centrifuge, and the 8-dram vials in a floor model centrifuge set up to handle 50-mL tubes. After centrifugation and decantation of excess water, each vial was swirled on the vortex-stirrer to put a thin film of kerogen on the surface walls of the vial. This increased the actual surface area of kerogen exposed to the vacuum, which insured more efficient freeze-drying. Small square filters, cut from kimwipes[1], covered the capless vials to protect the freeze dryer and pump. These filters were held in place with size 8 rubber bands. The kerogen was frozen at −50°C in the vials, before being placed into the dryer chamber. The drying was completed in 6 to 10 hours.

Freeze-drying kerogen, compared to air-drying or heat-drying kerogen, yields a fluffy and dry end-product ready for analytical use. Air or heat drying kerogen takes longer with more of a chance for oxidation to occur and normally produces a very brittle product. Air- or heat-dried kerogens require grinding for homogenization, and because of the extra handling, there is a chance of sample loss or contamination.

Carbon, hydrogen, and nitrogen elemental analysis

The freeze-dried kerogen samples were analyzed (table 1) on a Perkin-Elmer 240 Elemental Analyzer[1].

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[1] Trade names used in this report are for descriptive purposes only and do not reflect endorsement by the U.S. Geological Survey.

Table 1.--Kerogen data. Organic geochemical analyses of Phosphoria Formation (outcrop samples and one core at an outcrop site; inferred maximum depth of burial, in meters), Bertha Rogers well No. 1 (well depth in meters), and C.O.S.T Core (core depth in meters).

[Organic carbon; N, C, and H percentages; Atomic Ratios, H:C and C:N; Color of kerogen particles, in reflected light; Ash, percent of inorganics in kerogen; -, no data]

Sample No.	Depth (meters)	Organic carbon weight %	N %	C %	H %	Atomic H:C	Atomic C:N	Color	Ash %
				Phosphoria Formation					
74M-29FI	<1,500	11.9	2.28	67.70	6.82	1.20	35	Orange-brown	5.33
74M-33AK	2,000	2.8	1.48	53.84	1.87	.41	42	Brown	8.21
73M-44J	2,050	19.1	2.05	64.40	6.30	1.16	37	Yellow-orange-brown	7.03
73M-46H	2,250	0.6	.40	12.85	1.22	1.14	37	Dark brown	
73M-42K	3,000	4.0	1.01	28.61	2.38	1.00	33	Orange-brown	58.81
74M-2BK	3,500	1.9	1.77	69.95	5.98	1.02	46	Brown	10.26
73M-34D	3,750	2.9	1.37	66.10	6.99	1.26	56	Orange-brown	14.30
73M-34A	3,800	.4	.20	18.12	1.80	1.18	106	Orange-brown	76.30
73M-32E	4,400	.5	1.43	40.63	3.31	.97	33	Dark orange-brown	45.45
73M-31G	4,500	3.2	1.79	57.05	2.9	.61	37	Dark brown	14.61
73M-33G	4,600	4.2	1.60	69.70	2.34	.40	51	Black	9.74
73M-11D	7,000	2.7	1.48	56.16	2.10	.45	44	Dark brown	9.43
73M-21L	8,250	2.9	1.54	51.44	1.74	.40	39	Brown	11.33
74M-42BE	8,500	1.0	.93	31.46	1.54	.59	39	Dark brown	41.24
73M-17J	9,000	3.5	1.60	51.44	1.81	.41	38	Dark brown	12.10
74M-43AD	9,500	2.5	1.00	55.46	1.90	.40	65	Dark brown	10.56
				Phosphoria core (same sample site as 74M-2BK)					
	0 - .15	1.0	1.74	71.25	6.82	1.14	48	Brown	-
	0.6-.8	2.6	1.76	69.42	6.71	1.15	46	Brown	-
	1.2-1.4	2.4	1.66	68.87	6.49	1.12	48	Brown	-
	1.9-2.0	2.6	1.64	65.30	6.13	1.12	46	Brown	-
	3.3-3.4	6.7	1.96	66.56	6.43	1.15	40	Brown	-

Table 1.--Kerogen data. Organic geochemical analyses of Phosphoria Formation (outcrop samples and one core at an outcrop site; inferred maximum depth of burial, in meters), Bertha Rogers well No. 1 (well depth in meters), and C.O.S.T Core (core depth in meters)--continued

Sample No.	Depth (meters)	Organic carbon weight %	N %	C %	H %	Atomic H:C	Atomic C:N	Color	Ash %
				Bertha Rogers Well No. 1					
	1,676	0.1	0.54	11.85	1.21	1.21	26	Yellow-orange	-
	2,690	.4	2.13	58.85	4.42	.64	32	Yellow-orange-brown	23.20
	3,447	.3	1.37	62.00	3.42	.66	53	Orange-brown	21.80
	4,089	.5	1.08	63.76	4.07	.76	69	Orange-brown	
	4,671	.4	1.22	56.91	2.35	.49	54	Dark brown	28.56
	5,198	1.3	.93	63.07	2.26	.43	80	Brown	-
	5,779	1.1	.74	63.64	2.76	.52	100	Dark brown	13.28
	6,075	1.6	.56	58.40	3.83	.78	122	Orange brown	-
	6,600	1.3	.62	44.05	2.15	.58	83	Dark brown	-
	7,321	.3	.49	47.83	1.30	.32	114	Dark brown	33.43
	7,783	.4	.38	33.81	1.08	.38	104	Dark brown	-
	8,284	1.2	.41	45.66	1.03	.27	130	Dark brown	33.43
	8,451	3.6	.97	68.39	1.32	.23	82	Very dark brown	-
	8,635	.3	.54	26.23	1.51	.69	57	Very dark brown	-
	8,897	.6	.52	52.78	1.36	.31	118	Very dark brown	31.48
	9,082	.7	.40	21.16	1.49	.84	62	Dark brown	-
	9,412	.4	.30	38.65	.82	.25	150	Dark brown-black	-

Table 1.--Kerogen data. Organic geochemical analyses of Phosphoria Formation (outcrop samples and one core at an outcrop site; inferred maximum depth of burial, in meters), Bertha Rogers well No. 1 (well depth in meters), and C.O.S.T Core (core depth in meters)--continued.

Sample No.	Depth (meters)	Organic carbon weight %	N %	C %	H %	Atomic H:C	Atomic C:N	Color	Ash %
Continental offshore stratigraphic test (C.O.S.T.) OCS-CAL 75-70 No. 1 Core									
	1,008-11	3.3	2.73	60.27	6.62	1.31	26	Yellow-orange	7.43
	1,301-08	2.7	2.89	61.03	6.43	1.26	25	Yellow-orange-brown	7.45
	1,456-58	2.5	3.62	66.33	6.49	1.17	21	Brown	7.23
	1,642-44	.4	2.04	67.49	4.20	.74	39	Brown	5.98
	1,831-33	.7	2.23	68.35	3.10	.54	36	Brown	19.36
	2,531-34	.4	1.74	62.98	3.84	.73	42	Dark brown	16.52
	3,326-28	.6	1.93	78.26	3.68	.56	47	Dark brown	6.70

Ashing procedure

Selected samples were ashed in a furnace at 450°C for 20 to 24 hours in porcelain crucibles to judge the efficiency of the kerogen isolation technique. Sample weight was 3-26 mg depending on the amount of recovered kerogen. The samples were allowed to cool in a vacuum desiccator. The weight difference was recorded and the samples were ashed again for 2 hours at 450°C. The second weights of the ash were recorded and revealed no significant changes in the weights, indicating that all the organic material had been ashed (see table 1).

SAMPLE COLLECTION

Sampling of the Phosphoria Formation in southern Idaho, western Wyoming, and northeastern Utah was carried out during the summers of 1973 and 1974. The localities are given in Maughan (1976).

The samples were obtained from surface localitites, most of them from relatively fresh exposures. One shallow core was taken and an attempt was made to obtain unweathered samples at all localities. It is possible that the organic geochemical composition of these samples may have a low degree of weathering effects; it is probably of a fairly uniform nature and is not considered a significant factor in the interpretation of the results.

A suite of 16 samples was analyzed from the Bertha Rogers No. 1 well, Lone Star Gas Company, Washita County, Oklahoma. These samples were drill cuttings, supplied by L. C. Price (Price and others, in press).

The samples of cored sediment from the deep stratigraphic test OCS-CAL 75-70 No. 1 (Outer Continental Shelf-California) were also analyzed.

RESULTS AND DISCUSSION

Results of the kerogen analysis are given in table 1.

The marine Phosphoria Formation has a uniform amorphous organic matter (Sheldon and others, 1967; Maughan, 1975). Maximum burial depths were estimated by stratigraphic restoration of Phosphoria samples (Claypool and others, 1978). The uniform aspect of the organic material in this formation, makes it possible to observe changes in kerogen due to increasing depth of burial or increasing temperature (fig. 2) without interference from facies changes. The changes in the atomic H:C ratio from 1.26 to 0.40 (fig. 2) is due to temperature effects from depth of burial; and proximity to igneous intrusions 74M-33AK, a very shallow sample, taken from the periphery of the Idaho batholith (Maughan, 1975).

The open symbols shown in figures 3 and 4 are replicate samples from the Phosphoria Formation, analyzed to confirm the reproducibility of the method of isolation and analysis. Inferred depth of burial is equivalent to that of sample 74M-2BK.

The Bertha Rogers No. 1 well penetrated a section of marine Paleozoic rocks. The atomic H:C ratios of this well (fig. 3) showed a general decrease with depth of burial from 1.2. to 0.25. Two samples deviate significantly from the general trend with atomic H:C ratios of 0.69 and 0.84, at depths of 8.6 km and 9.0 km, respectively.

Kerogen data from a southern California borderlands offshore COST well was limited by the small number of samples (7) available. The atomic H:C ratios (fig. 4) change with depth from 1.3. at 1.0 km to 0.54 at 3.3 km. This amount of change through 2.3 km of sediment seems to indicate a higher than normal present day or paleo geothermal gradient, which is supported by kerogen data in table 1, or it may represent a change in the type of organic material present (stratigraphic control).

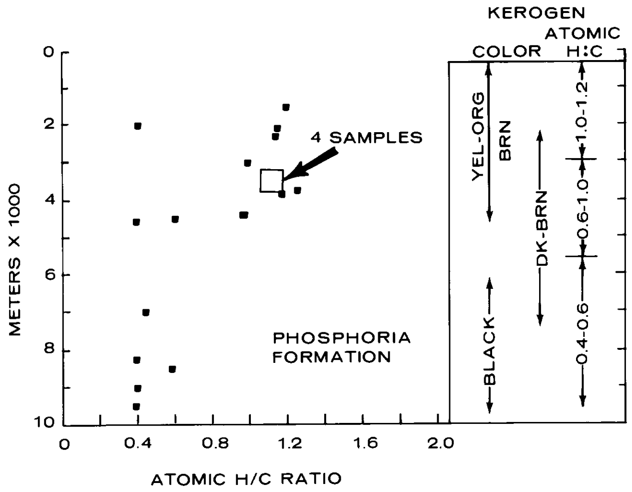

Figure 2.--Phosphoria Formation, atomic H:C ratio of kerogen versus inferred maximum depths of burial, with kerogen colors [Open symbols represent Phosphoria core samples, taken at same sample site as 74M-2BK]

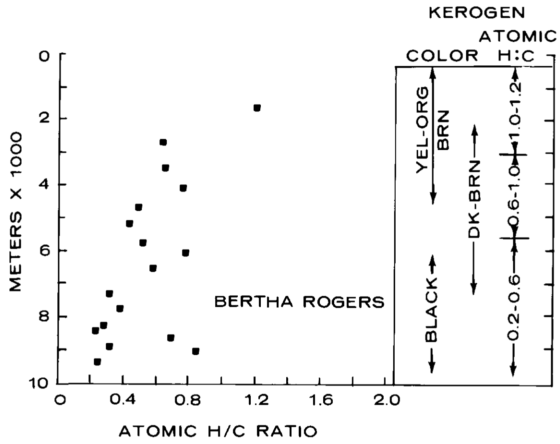

Figure 3.--Bertha Rogers well No. 1, atomic H:C ratio of kerogen versus depth of burial, with kerogen colors.

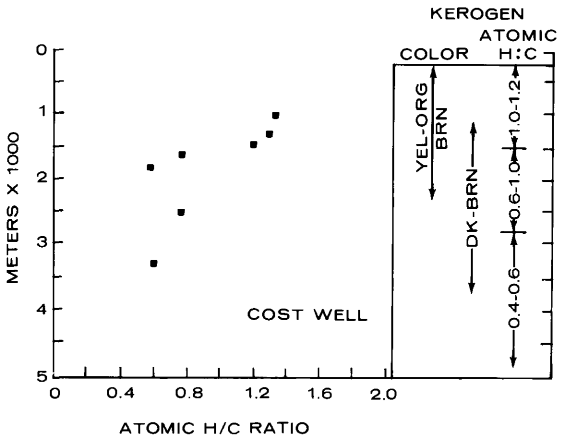

Figure 4.--COST well atomic H:C ratio of kerogen versus depth of burial, with kerogen colors.

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