

1943

CRETACEOUS SHALES OF MANITOBA AS A POSSIBLE SOURCE OF CRUDE PETROLEUM

by  
S. C. ELLS

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During recent years attention has been directed to reported occurrences of oil shale in the provinces of Manitoba and Saskatchewan. The shales are exposed along the escarpment of the Pembina, Riding, Duck, and Porcupine mountains, which border the lake plain and Red River valley of Manitoba. They are also found in the escarpment of the Pasquia hills, a northern extension of the same series. These hills, dignified by the name of mountains, constitute the erosion escarpment of the Cretaceous beds which form the first prairie step. The eastern edge is indented by drainage valleys of varying importance, which separate the hill features into groups. The Pasquia hills, and Porcupine, Duck, and Riding mountains, occupy an area which is bounded toward the north and northwest by the Carrot river, and toward the east by waterways, which include Moose lake, Cedar lake, and lake Winnipegosis. Broad, low-lying, slightly undulating, lacustral plains, which formed the bed of glacial Lake Agassiz, stretch away from the various waterways of the lower slopes of the hills. These lower slopes are marked by a series of old lake beaches, and rise by easy gradients, through some five or six hundred feet, to the more abrupt escarpment of the main ridge.

The whole area is well watered by numerous small streams few of which have a width greater than 60 feet. In descending from the table lands, these streams, for the most part, flow with rapid current along boulder-strewn channels, deeply entrenched in precipitous valleys and ravines, where active erosion and landslides are much in evidence. On reaching the lower slopes of the hills, the current slackens, and many excellent geological sections are exposed in cut banks at concave bends. Through the low-lying alluvial lands, the banks, as a rule, are low, and the channels of the meandering type.

Outcrops of shales examined on Birch river, Havel river, Sclater river, Pine river, Vermilion river and Ochre river, are all near the Winnipeg-Prince Albert branch of the Canadian National Railways, and may be easily reached by highway roads.

Toward the east and northeast, the slopes of the Pasquia hills are marked by somewhat sharp gradients. In passing westward, however, the slopes become more gentle, and rock exposures along the various streams less frequent. The elevation of the summit has not been accurately determined, but apparently ranges from 2,000 to 2,300 feet above sea level datum. The area throughout is densely wooded. Apart from a number of comparatively limited areas reserved as timber berths, the forest growth consists, principally, of large poplar and birch. Much of the land is of excellent quality, and, when cleared, should prove well adapted to agricultural development.

GEOLOGY

The general geology of the area was worked out many years ago

by Tyrrell, Dowling, and McInnes, and the reports then issued<sup>1</sup> remain authoritative. The general geological features are simple, and may be very briefly summarized as follows

The Pasqua hills, and Porcupine, Duck, and Riding mountains, are built up of Cretaceous sediments, resting unconformably on limestones of Palaeozoic age. Apart from type fossils, which are found at many localities, the principal subdivisions of the Cretaceous, within the area under consideration, may frequently be recognized by lithological characteristics.

A marker band of impure limestone, averaging some four feet thick, and containing typical fossils, occurs near the top of the Niobrara, and apparently extends over a wide area. Other marker bands are of local significance only. In the following general section, estimated thicknesses are given, out, pending the collection of further data, these must be considered as approximations.

	( Montana	( Pierre (upper) Odanah shales ... 400 feet
	(	( " (lower) Millwood shales.. 650 feet
	(	
Cretaceous	(	( Boyne Shale)
	( Colorado	( Niobrara (Morden shale) .... 130-240 feet
	(	( Assiniboia shale)
	(	(
	(	( Benton shale ..... 180 feet
	(	Dakota sandstone.
Devonian .....		Unconformity.

The detailed geological structure has not been accurately worked out, although numerous exposed sections, together with correlated elevations, indicate that the general dip of strata of Cretaceous age is almost horizontal. Between Winnipegosis and the well drilled by the Manitoba Oil Company, 11 miles southwest of Dauphin, the dip of Upper Palaeozoic limestones is apparently about four feet to the mile, in a southwesterly direction. South of Swan River valley, Cretaceous sediments also dip at a very low angle toward the southwest. North of this point, the strata flatten out, and finally dip toward the north. The same general horizontal structure apparently typifies strata underlying the Pasqua hills. Minor local folding was observed, but this in itself can scarcely be considered as of economic importance. The general physical characteristics of members of the Cretaceous system may be briefly summarized as follows

Pierre.-- Odanah series. Dark fissile shales, usually poor in fossils, and weathering to light grey fissile flakes. Often rusty along joint planes.

<sup>1</sup> Report on Northwestern Manitoba with portions of the adjacent districts of Assiniboia and Saskatchewan, J. B. Tyrrell and D. B. Dowling. Geol. Survey of Canada, 1892.  
The Basins of Nelson and Churchill Rivers, Wm. McInnes, Geol. Surv. of Canada, Mem. 30. 1913.

Millwood series. Soft grey shales, sometimes almost black in colour. Weathers to light grey flaky particles, which eventually disintegrate to dark plastic clay. Slopes adjacent to cut banks exhibit marked evidence of instability. Ironstone nodules are common, particularly near contact with Niobrara.

Niobrara.-- Dark grey clay shales, frequently mottled, and interbedded with heavy bands of hard calcareous shale, usually highly fossiliferous, and almost black in colour. Shale weathers to light grey colour. Comparatively weather resistant.

Benton.-- Very dark, soft, non-calcareous, somewhat carbonaceous, evenly bedded shale, poor in fossils. Weathers to small thin flakes, which rapidly disintegrate into dark plastic clay.

Dakota.-- White, light grey or greenish sandstone. Usually somewhat soft, but in places having the hardness of quartzite.

With the exception of the upper and the lower members of the Cretaceous system, the sediments exposed within the area under consideration thus consist chiefly of rather soft, greenish grey, clay shales, more or less darkened owing to the presence of hydrocarbons and iron sulphides. Moreover, many of these shales, when freshly broken, emit a marked odour of petroleum, which, on exposure, rapidly passes off. When disintegrated by the drilling tools, in the presence of water, the shale forms an emulsion of a dark green colour. This emulsion gives off a faint odour of petroleum, and, after standing for a time, exhibits a thin film of petroleum on the surface. It is, therefore, not surprising that the presence of free petroleum in wells drilled through the shales has been reported from time to time.

Of upper Cretaceous shales, the Niobrara formation is the most highly fossiliferous, hence it is within this horizon that bands of oil shales might have been anticipated. Fossils observed in this formation comprise a large number of foraminifera, among which *Globigerina cretacea* is conspicuous, and an abundance of shells of a large *Inoceramus*, together with the following species.

*Serpula semicoalita* (Whiteaves. N. sp.).  
*Ostrea congesta* (Conrad).  
*Anomia obliqua* (Meek and Hayden).  
*Inoceramus problematicus* (Schlotheim).  
*Belemnitella Manitobensis* (Whiteaves. N. sp.).  
*Loricula Canadensis* (Whiteaves. N. sp.).  
*Ptychodus parvulus* (Whiteaves. N. sp.).  
*Lamna Manitobensis* (Whiteaves. N. sp. loose).  
*Enchodus Shumardii* (Leidy).  
*Cladocyclus occidentalis* (Leidy).

#### Origin of Shale

Oil shales of New Brunswick and Nova Scotia were deposited under different conditions from those of Manitoba and Saskatchewan. In

the former provinces, material from which the shale beds were derived was originally deposited in the form of fine clays on the bottom of swamps and lagoons. If the theory of Stuart<sup>2</sup> is accepted, there was associated with these sediments vegetable matter converted into a pulp, as a result of maceration and microbe action in water, together with richer materials of various kinds, such as spores, and a proportion of animal matter. We may, therefore, conclude that the origin of the petroleum in the shales may be traced to fermentation and decomposition of organic matter, through microbe action. On the other hand, the shales of Manitoba and Saskatchewan were laid down in a muddy sea, and were, therefore, not subjected to the various influences that affected the shales of the Maritime Provinces.

#### PHYSICAL CHARACTERISTICS OF SHALES IN THE PROVINCES OF Manitoba and Saskatchewan.

It is evident that great areas of Cretaceous shales, with their notable content of organic matter, have been subjected to very slight disturbance. Had the shales been affected by folding or by compression, as a result of great earth movements, distillation of organic remains, due to heat and pressure, would doubtless have resulted in the formation of gas and petroleum.

Attempts to ignite fine splinters of the various shales examined, by means of a match, failed in nearly every instance. Niobrara shales, when partially dried, will, however, frequently ignite when placed on an open wood fire. Pierre shales examined ignite but rarely, and then with difficulty. Benton shales showed no evidence of being combustible. Sampling was, therefore, confined largely to Niobrara shales.

In appearance and physical characteristics, as well as in the conditions under which they were deposited, these upper Cretaceous shales are thus in marked contrast to the oil shales of Nova Scotia and New Brunswick. In these provinces, two general types of shale are recognized, viz., plain and curly. A sub-variety, usually referred to as paper shale, is apparently a weathered form of plain oil shale. It is thin-bedded or papyraceous, separating readily into thin flexible sheets of considerable surface dimensions. Plain oil shales are usually flat surfaced, showing more or less well defined lamination. On the other hand, curly shale usually occurs as massive bands, somewhat curled and contorted. It is very tough, breaks with a conchoidal fracture, and is, usually very rich in hydrocarbons.

As a rule, oil shales of Nova Scotia and New Brunswick are free from grit, and, although readily cut with a sharp knife, do not yield thin, flexible shavings as do the Scotch shales. They are strongly resistant to

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Stuart, D. R., The Chemistry of Oil Shales, Oil Shales of the Lothians, Geol. Surv., Scotland, 1912.

weathering, and fragments have remained, for many years, exposed to action of atmospheric agencies, with but little loss of their hydrocarbon content. When placed in a grate, fragments may be readily ignited, giving off an intense heat, and continuing to burn with a long yellow flame for periods of from one to three hours. Small splinters of the shale may usually be ignited by means of a match.

Oil shales of New Brunswick, Nova Scotia, and Scotland, do not contain hydrocarbons which are liquid at atmospheric temperature. Numerous wells have been drilled through the shales by various companies, but, although free petroleum has been encountered in interbedded sands and sandstones, practically none has been met with in the shales themselves.

#### WEATHERING OF SHALES

Weathering of the Cretaceous shales presents an interesting study. As a rule, the hydrocarbon content of shales examined does not prevent rapid weathering, while, on the other hand, the shales contain within themselves a variety of potent disruptive agencies. Apart from the high percentage of water present, this disintegration, due to the formation of secondary minerals, as iron and calcium sulphates, hydrated iron oxides, etc., and circulating solutions containing acids and sulphates, is very marked.

#### ASSOCIATED MINERALS

Incidental to the examination of the Cretaceous shales, the presence of certain associated minerals was noted. None of these minerals were observed in sufficient quantity to be of present economic importance, but, pending a complete chemical analysis of the shales, their somewhat complex geochemistry may be briefly alluded to.

These associated minerals fall into two classes, viz., those which, being deep seated, have not been subjected to oxidizing influences, and others, which have been deposited at, or near the surface, in the presence of circulating waters, and under the influence of other oxidizing agencies. In addition to bentonite, the principal deep seated minerals comprise pyrite, siderite, and glauconite. Owing to the length of time during which they have been forming, these minerals occur in considerable quantity and exhibit comparative purity. Surface deposits comprise marcasite, iron sulphate, limonite, gypsum (in the forms of selenite and satinspar) native sulphur, and small amounts of iron oxide. Pyrite, in the form of small cubes, sometimes occurs in narrow lenses, but is more often disseminated and invisible to the naked eye. Siderite is most abundant in the Pierre series, at times forming beds a few inches thick. Bentonite occurs as uniform, sharply defined beds, having a maximum observed thickness of 24 inches. Pyrite and marcasite, in the presence of oxidation agents, give rise to the formation of small quantities of sulphurous and sulphuric acids. It may be possible that these acids, circulating through the shales, form soluble sulphates which are subsequently leached out, leaving bentonite as a residual product. To the oxidation of iron sulphides may also be attributed the nauseating sulphurous odour which, at times, emanates from

shale banks, and has frequently been construed as indicating the presence of natural gas.

Absorption of oxygen, assisted by oxidation of iron sulphides, is responsible for slow spontaneous combustion in progress in many of the banks of shale. At one locality a column of thin blue smoke was observed rising from a smouldering bank, the talus at the foot of the bank being cemented together by various sulphates. Moreover, traces of free petroleum, released as a result of recognized chemical reactions, as well as small amounts of colloidal iron hydroxide, frequently accumulate to form a scum, which, at times, has been mistaken for a true petroleum seepage.

#### SAMPLING AND ANALYSES

In securing samples overburden was removed and trenches cut from top to bottom of exposed sections. The depth at which samples were taken varied from four to eight feet from the face, but it is considered that in each instance unaltered shale was procured. A number of samples were tested in the field, and satisfactory results were obtained by the use of a retorting apparatus<sup>1</sup> somewhat similar to that recommended by the United States Bureau of Mines. Only the petroleum content was determined. In the case of certain other samples, subsequently tested in the laboratory, the yield of ammonium sulphate was also determined. Nineteen representative samples were procured from Steeprock river, Birch river, Favel river (east and west branches), Sinclair river, North Duck river, Sclater river, North Pine river, Vermilion river and Ochre river, in the province of Manitoba.

Certain of these samples showed merely a trace of hydrocarbons, hence reference to them is omitted in the following summary.<sup>2</sup>

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<sup>1</sup> Reports of Investigations, U.S. Bureau of Mines, Serial No. 2229.

<sup>2</sup> Determinations by Chemical Laboratories, Mines Branch, Department of Mines.

## ANALYSES

Locality	Imperial gals. crude petroleum per ton <sup>1</sup>	Spr. gr. of crude pet- roleum at 60° F.	Imperial gals. water per ton	Pounds ammonium sulphate per ton
Birch River, Sec. 31, Tp. 33, R. 26, W. 1st.	0	....	42.7	....
Favel River, Sec. 30, Tp. 35, R. 25, W. 1st.	6.2	0.972	12.1	....
Sec. 26, Tp. 35, R. 26, W. 1st.	6.8	0.984	7.0	....
" " " " " "	5.9	0.965	15.2	....
Sclater river, (2 samples), Sec. 15, Tp. 34, R. 23, W. 1st	4.8-7.5	0.966-0.968	9.2-18.7	....
Pine river, Sec. 6, Tp. 33, R. 22, W. 1st	3.3	0.969	4.5	....
Vermilion river, (2 samples), Sec. 12, Tp. 24, R. 20, W. 1st	1.1-5.1	0.952	8.1-22.0	....
Ochre river, (2 samples), Sec. 29, Tp. 22, R. 17 W. 1st	4.0-5.3	0.955	14.6-15.2	....

<sup>1</sup>Calculations based on ton of 2,000 pounds.

The sulphur content of the crude petroleum recovered from six representative samples of shale varied from 5.3 per cent to 7.7 per cent, with an average for the six samples of 6.5 per cent. A sample of crude petroleum distilled from shale secured on Ochre river, Manitoba, (Sec. 29, Tp. 22, R. 17, W. of 1st) was fractionated with the following result:-

<u>Distillation range</u>	<u>Barom. 766.0 mm.</u>
1st drop .....	79 degrees C.
10 p.c. ....	149 " C.
20 p.c. ....	170 " C.
30 p.c. ....	221 " C.
40 p.c. ....	254 " C.
50 p.c. ....	281 " C.
60 p.c. ....	301 " C.
70 p.c. ....	336 " C.
80 p.c. ....	350 " C.
90 p.c. ....	361 " C.
92 p.c. dry point (cracking occurred).	"

The gas yield from 13 representative samples of shale was also determined, and varied from 410 to 1,130 cubic feet per ton, with an average yield of 695 cubic feet. Analyses of two samples of gas recovered from shales secured on Man river, Sask., (Tp. 50, R. 5, W. of 2nd) are as follows.

	Sample No. 1825 p.c.		Sample No. 1831 p.c.	
Carbon dioxide .....	32.6	.....	23.3	
Oxygen .....	2.1	.....	3.1	
Ethylene .....	3.7	.....	3.8	
Carbon monoxide .....	2.4	.....	1.9	
Methane .....	28.7	.....	30.6	
Hydrogen .....	9.1	.....	7.5	
Nitrogen .....	21.4	.....	29.8	
Inflammable gas .....	43.9	.....	43.8	
Calorific value, gross .....	383	.....	297	B.Th.U per
				cu. ft.
Calorific value, net .....	346	.....	359	"

#### CONCLUSION

It is, therefore, obvious that shales examined during the past season in the provinces of Manitoba and Saskatchewan are of little present economic importance as a possible source of petroleum or of ammonium sulphate. Should conditions at any time warrant commercial development, open cut mining could be undertaken in many areas under favourable conditions. Over very considerable areas the shales examined are covered by a comparatively light overburden, consisting chiefly of boulder clays and gravel, which could be readily removed by hydraulic methods.



Dept of Mines Canada Summary Report  
1921

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Market Prices — The market prices for alkalis are constantly varying. The following figures, as reported in the Oil, Paint, and Drug Reports, New York, give the New York market prices for the years 1911 to date —

	Aug 14 1914	Jul 1 1915	Jan 1 1917	Jun 1 1918	* 1919	* 1920	* 1921
	\$	\$	\$	\$	\$	\$	\$
Salt cake ground—	11 00	11 00		30 00	12 00	17 00	17 00
bbl per ton	to 13 00	13 00		to 31 00	to 30 00	to 30 00	to 28 00
Clayber's salt—cwt	0 65	0 60	0 60	0 90	1 00	1 15	1 00
	to 0 75	to 0 75	to 0 75	to 1 00	to 2 25	to 2 65	to 1 75
Lpsom salt U S P—	Not quoted prior to 1918			5 62½	2 75	2 30	2 20
cwt				to 3 90	to 6 25	to 5 30	to 2 75
Lpsom salt tech—	1 00	1 75	1 75	3 7½	1 50	1 75	1 10
cwt	to 1 10	to 2 00	to 1 85	to 3 50	to 3 37½	to 3 50	to 1 75

\*High and low figures for year

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CRETACEOUS SHALES OF MANITOBA AND SASKATCHEWAN,  
AS A POSSIBLE SOURCE OF CRUDE PETROLEUM

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INTRODUCTORY

During recent years attention has been directed to reported occurrences of oil shale in the provinces of Manitoba and Saskatchewan. The shales are exposed along the escarpment of the Pembina, Riding, Duck, and Porcupine mountains, which border the lake plain and Red River valley of Manitoba. They are also found in the escarpment of the Pasquia hills, a northern extension of the same series. These hills, dignified by the name of mountains, constitute the erosion escarpment of the Cretaceous beds which form the first prairie step. The eastern edge is indented by drainage valleys of varying importance, which separate the hill features into groups. The Pasquia hills, and Porcupine, Duck, and Riding mountains, occupy an area which is bounded toward the north and northwest by the Carrot river, and toward the east by waterways, which include Moose lake, Cedar lake and lake Winnipegosis. Broad, low-lying, slightly undulating, lacustral plains, which formed the bed of glacial Lake Agassiz, stretch away from the various waterways of the lower slopes of the hills. These lower slopes are marked by a series of old lake beaches, and rise by easy gradients, through some five or six hundred feet, to the more abrupt escarpment of the main ridge.

The whole area is well watered by numerous small streams few of which have a width greater than 60 feet. In descending from the table lands, these streams, for the most part flow with rapid current along boulder-strewn channels, deeply entrenched in precipitous valleys and ravines, where active erosion and landslides are much in evidence. On reaching the lower slopes of the hills the current slackens, and many excellent geological sections are exposed in cut banks at concave bends

through the low-lying alluvial lands, the banks, as a rule, are low, and the channels of the meandering type.

Outcrops of shales examined on Bush river, Fivel river, Selater river, Pine river, Vermilion river and Ochre river, are all near the Winnipeg-Prince Albert branch of the Canadian National Railway, and may be easily reached by highway roads. Outcrops examined on the Tee river—a branch of Pasqua river, Man river, Cracking river, and Papikwan river in the Pasqua hills, are somewhat remote from rail transportation.

Toward the east and northeast, the slopes of the Pasqua hills are marked by somewhat sharp gradients. In passing westward, however, the slopes become more gentle, and rock exposures along the various streams less frequent. The elevation of the summit has not been accurately determined, but apparently ranges from 2,000 to 2,300 feet above sea level datum. The area throughout is densely wooded. Apart from a number of comparatively limited areas reserved as timber berths, the forest growth consists, principally, of large poplar and birch. Much of the land is of excellent quality, and, when cleared, should prove well adapted to agricultural development.

Outcrops of shale along streams which drain the northeastern slopes of the hills (as on Tee river and Man river) can best be reached from the Pas. From Mountain cabin on Carrot river, some 60 miles W S W from the Pas, a good summer trail, approximately 2½ miles in length, leads south to Tee river. From Camp No. 6 on Carrot river, some 90 miles from the Pas, a fair bush road, approximately 17 miles in length, leads to Man river.

Papikwan and Cracking rivers can best be reached from McDonald's siding, 2½ miles west of Mistatim station, on the Canadian National Railway. From McDonald's siding, a fair wagon road, some 24 miles in length, leads to Connell cabin. From Connell cabin to Papikwan cabin, a distance of approximately 12 miles, a fair pack trail is available, but between Papikwan cabin and the shale outcrops at the forks of Cracking river, the trail is wet and difficult. Other trails indicated on the accompanying map are, for the most part, poor, and in many instances, difficult to follow.

#### GEOLOGY

The general geology of the area was worked out many years ago by Tyrrell, Dowling, and McInnes, and the reports then issued<sup>1</sup> remain authoritative. The general geological features are simple, and may be very briefly summarized as follows—

The Pasqua hills, and Porcupine, Duck, and Riding mountains, are built up of Cretaceous sediments, resting unconformably on limestones of Paleozoic age. Apart from type fossils, which are found at many localities, the principal subdivisions of the Cretaceous, within the area under consideration, may frequently be recognized by lithological characteristics.

<sup>1</sup> Report on Northwest and Manitoba with portions of the adjacent Provinces of Saskatchewan, J. B. Tyrrell and D. B. Dowling, Geol. Surv. of Canada, 189. The Basins of Nelson and Churchill's River, Wm. McInnes, Geol. Surv. of Canada, Memoir 1913.

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Cretaceous	Montana	{ Pierre (upper) Odunah shales (lower) Millwood shales	400 feet 600
	Colorado	{ (Boyer shale) Niobrara (Morden shale) (Assiniboia shale)	170-210
		{ Benton shale Dakota sandstone	180
Devonian	Unconformity		

The detailed geological structure has not been accurately worked out, although numerous exposed sections, together with correlated elevations, indicate that the general dip of strata of Cretaceous age is almost horizontal. Between Winnipegosis and the well drilled by the Manitoba Oil Company, 11 miles southwest of Dauphin, the dip of Upper Paleozoic limestones is apparently about four feet to the mile in a southwesterly direction. South of Swan River valley, Cretaceous sediments also dip at a very low angle toward the southwest. North of this point, the strata flatten out and finally dip toward the north. The same general horizontal structure apparently typifies strata underlying the Pasquia hills. Minor local folding is observed, but this in itself can scarcely be considered as of economic importance. The general physical characteristics of members of the Cretaceous system may be briefly summarized as follows—

**Pierre**—Odunah series. Dark fissile shales usually poor in fossils and weathering to light grey fissile flakes. Often rusty along joint planes.

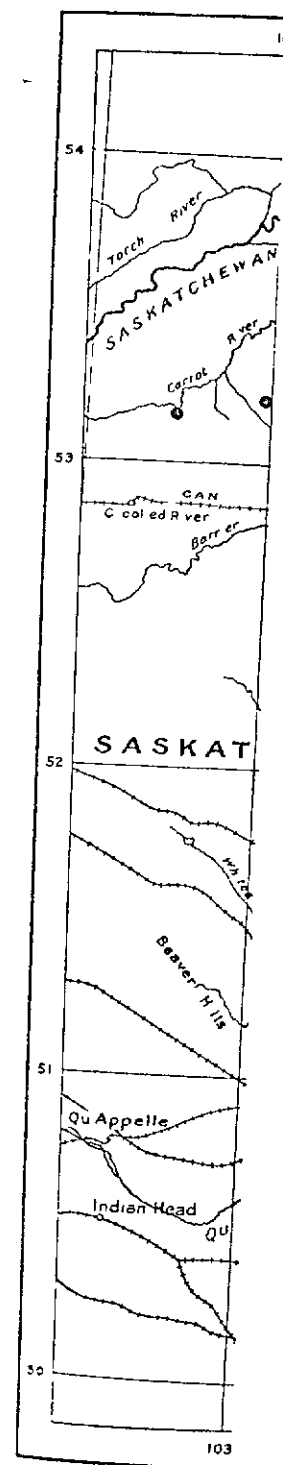
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**Niobrara**—Dark grey clay shales frequently mottled and interbedded with heavy bands of hard calcareous shale usually highly fossiliferous, and almost black in colour. Shale weathers to light grey colour. Comparatively weather resistant.

**Benton**—Very dark, soft non-calcareous somewhat carbonaceous, evenly bedded shale poor in fossils. Weathers to small thin flakes, which rapidly disintegrate into dark plastic clay.

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With the exception of the upper and the lower members of the Cretaceous system the sediments exposed within the area under consideration thus consist chiefly of rather soft greenish grey, clay shales more or less darkened owing to the presence of hydrocarbons and iron sulphides. Moreover, many of these shales when freshly broken emit a marked odour of petroleum, which, on exposure, rapidly passes off. When disintegrated by



Map showing sampled

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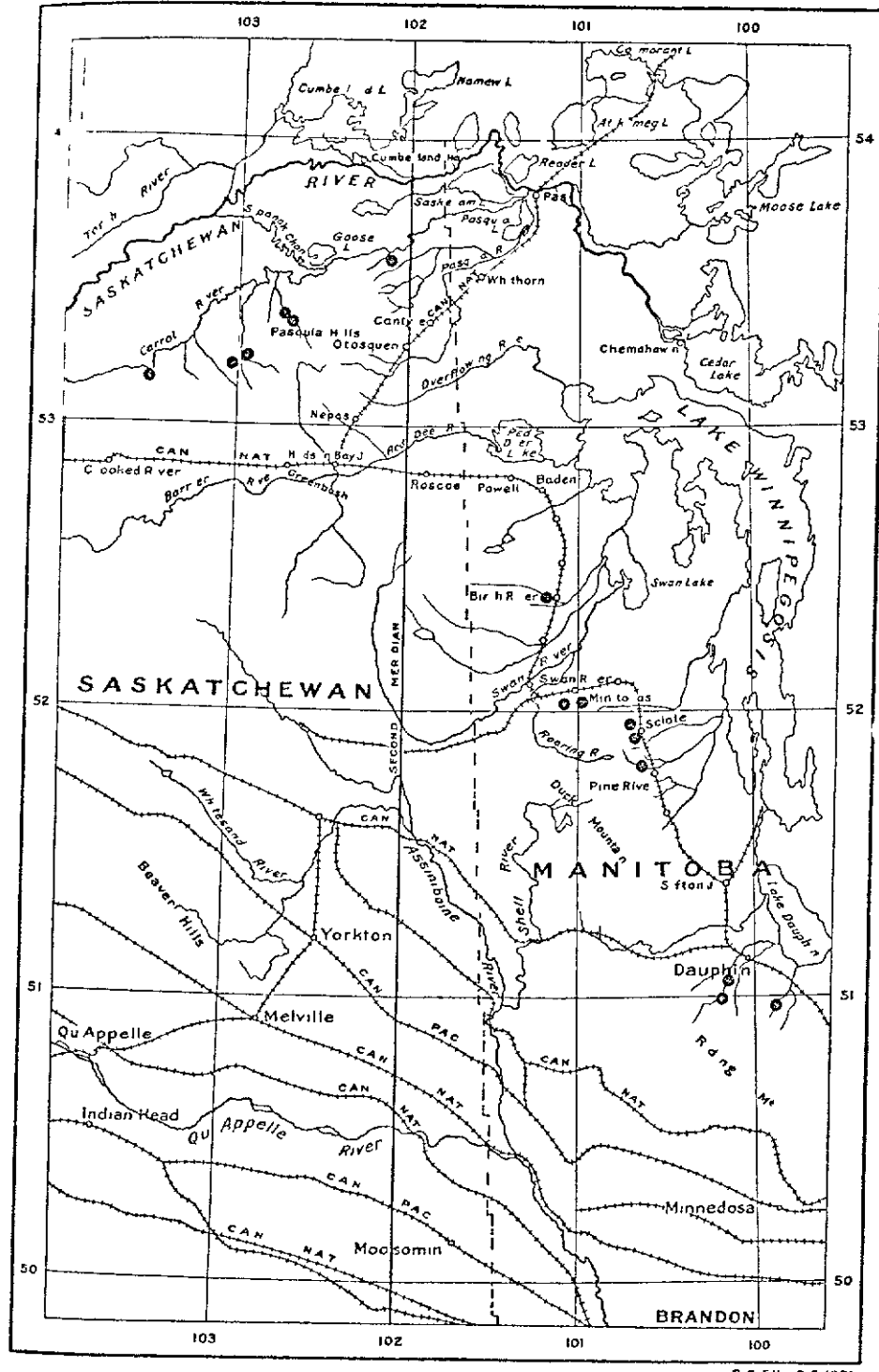
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Map showing position of Outcrops of Cretaceous Shales  
sampled during year 1921, in Manitoba and Saskatchewan

Scale of Miles  
20 10 0 20 40 60 80

FIG 2

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It is evident that great areas of Cretaceous shales, with their notable content of organic matter, have been subjected to very slight disturbance. Had the shales been affected by folding or by compression, as a result of great earth movements, distillation of organic remains, due to heat and pressure, would doubtless have resulted in the formation of gas and petroleum.

<sup>1</sup> Geol. Surv. Report on North Western Can., Manitoba, J. B. Lyell, 1897.  
<sup>2</sup> Stewart, D. J., The Chemistry of Oil Shales. Oil Shales of the Lothians, Geol. Surv. Scotland, 1917.

Attempts to ignite fine splinters of the various shales examined, by means of a match failed in nearly every instance. Niobrara shales when partially dried, will however, frequently ignite when placed on an open wood fire. Pierre shales examined ignited but rarely, and then with difficulty. Benton shales showed no evidence of being combustible. Sampling was, therefore, confined largely to Niobrara shales.

In appearance and physical characteristics, as well as in the conditions under which they were deposited, these upper Cretaceous shales are thus in marked contrast to the oil shales of Nova Scotia and New Brunswick. In these provinces, two general types of shale are recognized, viz. plain and curly. A sub-variety usually referred to as paper shale, is apparently a weathered form of plain oil shale. It is thin-bedded, or papery, and separates readily into thin, flexible sheets of considerable surface dimensions. Plain oil shales are usually flat surfaced, showing more or less well defined lamination. On the other hand, curly shale usually occurs as massive beds, somewhat curled and contorted. It is very tough, breaks with a conchoidal fracture, and is usually very rich in hydrocarbons.

As a rule, oil shales of Nova Scotia and New Brunswick are free from grit, and, although readily cut with a sharp knife, do not yield thin, flexible shavings as do the Scotch shales. They are strongly resistant to weathering, and fragments have remained, for many years, exposed to action of atmospheric agencies, with but little loss of their hydrocarbon content. When placed in a grate, fragments may be readily ignited, giving off an intense heat, and continuing to burn with a long yellow flame for periods of from one to three hours. Small splinters of the shale may usually be ignited by means of a match.

Oil shales of New Brunswick, Nova Scotia, and Scotland, do not contain hydrocarbons which are liquid at atmospheric temperature. Numerous wells have been drilled through the shales by various companies but, although free petroleum has been encountered in interbedded sands and sandstones, practically none has been met with in the shales themselves.

#### WEATHERING OF SHALES

Weathering of the Cretaceous shales presents an interesting study. As a rule, the hydrocarbon content of shales examined does not prevent rapid weathering, while, on the other hand, the shales contain within themselves a variety of potent disruptive agencies. Apart from the high percentage of water present this disintegration, due to the formation of secondary minerals as iron and calcium sulphates, hydrated iron oxides, etc., and circulating solutions containing acids and sulphates, is very marked.

#### ASSOCIATED MINERALS

Incidental to the examination of the Cretaceous shales the presence of certain associated minerals was noted. None of these minerals were observed in sufficient quantity to be of present economic importance, but, pending a complete chemical analysis of the shales, their somewhat complex geochemistry may be briefly alluded to.

These associated minerals fall into two classes, viz. those which, being deep seated, have not been subjected to oxidizing influences, and others, which have been deposited at, or near the surface, in the presence of circulating waters, and under the influence of other oxidizing agencies. In

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In addition to bentonite the principal deep seated minerals comprise pyrite, siderite and glauconite. Owing to the length of time during which they have been forming, these minerals occur in considerable quantity and exhibit comparative purity. Surface deposits comprise muscovite, iron sulphate, limonite, gypsum (in the forms of selenite and satin spar), native sulphur, and small amounts of iron oxide. Pyrite, in the form of small nodules sometimes occurs in narrow lenses, but is more often disseminated and invisible to the naked eye. Siderite is most abundant in the Pierre series, at times forming beds a few inches thick. Bentonite occurs as uniform, sharply defined beds, having a maximum observed thickness of 24 inches. Pyrite and marcasite, in the presence of oxidation agents give rise to the formation of small quantities of sulphurous and sulphuric acids. It may be possible that these acids, circulating through the shales, form soluble sulphates which are subsequently leached out leaving bentonite as a residual product. To the oxidation of iron sulphides may also be attributed the nauseating sulphurous odour which, at times emanates from shale banks, and has frequently been construed as indicating the presence of natural gas.

Absorption of oxygen, assisted by oxidation of iron sulphides, is responsible for slow spontaneous combustion in progress in many of the banks of shale. At one locality a column of thin blue smoke was observed rising from a smouldering bank, the talus at the foot of the bank being cemented together by various sulphates. Moreover, traces of free petroleum, released as a result of recognized chemical reactions, as well as small amounts of colloidal iron hydroxide, frequently accumulate to form a scum, which, at times, has been mistaken for a true petroleum seepage.

#### SAMPLING AND ANALYSES

In securing samples overburden was removed and trenches cut from top to bottom of exposed sections. The depth at which samples were taken varied from four to eight feet from the face, but it is considered that in each instance unaltered shale was procured. A number of samples were tested in the field, and satisfactory results were obtained by the use of a retorting apparatus somewhat similar to that recommended by the United States Bureau of Mines. Only the petroleum content was determined. In the case of certain other samples, subsequently tested in the laboratory the yield of ammonium sulphate was also determined. Twenty-two representative samples were procured on the northern branch of the Pis river, Man river, Craking river, Papikwan river, Jordan river, and Carrot river, in the province of Saskatchewan, and nineteen samples from Steeprock river, Birch river, Fivel river (east and west branches), Simlar river, North Duck river, Selater river, North Pine river, Vermilion river, and Ochre river, in the province of Manitoba.

Certain of these samples showed merely a trace of hydrocarbons, hence reference to them is omitted in the following summary.

<sup>1</sup> Reports of Investigations U.S. Bureau of Mines. Serial No. 229.

<sup>2</sup> Determinations by Chemical Laboratories, Mine Branch, Department of Mines.

Analyses

Locality	Imperial gals crude petroleum per ton <sup>1</sup>	Spr gr of crude petroleum at 60 f	Imperial gals water per ton	Pounds ammonium sulphate per ton
Tee river Sask (2 samples) Tp 03 R 1 W of 2nd	3 1-5 3	0 972-0 981	12 0-16 0	
Man river Sask (5 samples) Tp 00 R 5 W of 2nd	0 0-9 4	0 912-0 964		3 1-8 7
Cracking river Sask (6 samples) Tp 49 R 7 W of 2nd	7 0-12 8	0 919-0 970	11 6-23 0	2 6-4 0
Jordan river Sask (float) Sec 6 Tp 48 R 10 W of 2nd	10 9	0 964		3 0
Carrot river Sask (2 samples) Sec 25 Tp 45 R 11 W of 2nd	4 9-6 0	0 903-0 909		1 7-2 8
Birch river Man Sec 31 Tp 39 R 26 W of 1st	0		42 7	
Favel river Man Sec 30 Tp 05 R 25 W of 1st	6 2	0 972	12 1	
Sec 26 Tp 3, R 26 W of 1st	6 8	0 984	7 0	
Selater river Man (2 samples) Sec 15 Tp 34 P 23 W of 1st	0 9	0 960	15 2	
Fine river Man Sec 6 Tp 33 R 22 W of 1st	4 8-7 0	0 906-0 968	9 2-13 7	
Vermilion river Man (2 samples) Sec 12 Tp 21 R 20 W of 1st	3 3	0 969	4 5	
Ochre river Man (2 samples) Sec 29 Tp 22 R 17 W of 1st	1 1-0 1	0 952	8 1-22 0	
	4 0-5 3	0 905	14 6-10 2	

<sup>1</sup>Calculations based on ton of 2 600 pounds

The sulphur content of the crude petroleum recovered from six representative samples of shale varied from 5.3 per cent to 7.7 per cent, with an average for the six samples of 6.5 per cent. A sample of crude petroleum distilled from shale secured on Ochre river, Man, (Sec 29, Tp 22, R 17, W of 1st) was fractionated with the following result —

Distillate on range	Per cent	Pressure 766.0 mm
1st drop		79 C
10 p.c.		149 C
20		170 C
30		221 C
40 "		234 C
50		281 C
60		301 C
70		336 C
80		350 C
90		361 C
92 dry ppt (cracking occurred)		

The gas yield from 13 representative samples of shale was also determined, and varied from 410 to 1,130 cubic feet per ton, with an average yield of 695 cubic feet. Analyses of two samples of gas recovered from shale secured on Man river, Sask (Tp 50, R 5 W of 2nd) are as follows —

Carbon  
Oxygen  
Hydrogen  
Methane  
Nitrogen  
Inflam  
Caloric

It is, therefore, in the province economic importance, open to sulphate, under favorable conditions covered by a clay and gravel

(S)

Occurrences within the Dominion. Nevertheless, with the discovery of petroleum and gas in 1862 and again in 1862 to recent local shale discovered pools of oil and gas of nearly six hundred value of C. This is the first time it has appeared to the west of the province, and consequently, much of the oil and gas in the province may be definitely determined. The writer has been in New Brunswick and has been in contact with the geological information.



	Sample No 1825	Sample No 1831
	p c	p c
Carbon dioxide	32.6	23.3
Oxygen	2.1	3.1
Ethylene	3.7	3.8
Carbon monoxide	2.4	1.9
Methane	28.7	30.6
Hydrogen	9.1	7.5
Nitrogen	21.4	24.8
Inflammable gas	43.9	43.8
Calorific value gross	383	297 B l h U per cu ft
Calorific value net	346	359

CONCLUSION

It is, therefore, obvious that shales examined during the past season in the provinces of Manitoba and Saskatchewan are of little present economic importance as a possible source of petroleum or of ammonium sulphate. Should conditions at any time warrant commercial development, open cut mining could be undertaken in many areas under favourable conditions. Over very considerable areas the shales examined are covered by a comparatively light overburden, consisting chiefly of boulder clays and gravel, which could be readily removed by hydraulic methods.

VII

OIL SHALES OF CANADA

S C ELLS

(SUMMARY AND REVIEW OF AVAILABLE INFORMATION)

Occurrences of petroliferous shales (commonly termed oil shales), within the Dominion of Canada, have for many years been known to exist. Nevertheless with the exception of a comparatively brief period, Canada has had to depend largely on foreign sources for its supplies of crude petroleum and petroleum products. In 1859, near Collingwood, Ontario, and again in 1862 near Baltimore, New Brunswick, attempts were made to retort local shales. In each instance these attempts were abandoned owing to the rapidly increasing production of well petroleum from the newly discovered pools of western Ontario and Pennsylvania. Now, after a lapse of nearly sixty years, attention is once more directed to the commercial value of Canadian shales as a possible source of crude petroleum.

Thus it is that only within very recent years, changing economic conditions appeared to warrant detailed study of existing shale bodies in Canada. Consequently, much work still remains to be done, not only in the field but in the laboratory, before the economic importance of such areas can be definitely determined. Meanwhile such information as is available, may be credited almost wholly to the Geological Survey of Canada. The writer is personally familiar with the oil shales of Nova Scotia, New Brunswick, Gaspe peninsula, northern Ontario, Manitoba, and Saskatchewan. Reference to occurrences outside of these areas is based on information secured indirectly.

Imperial gals water per ton	Pounds ammonium sulphate per ton
12.5-16.0	3.1-8.7
11.6-28.0	2.6-4.5
	3.0
	1.7-2.8
42.7	
12.1	
7.0	
15.2	
9.2-18.7	
4.5	
8.1-22.0	
14.6-15.2	

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- P nom 766 0 mm
- 79 C
- 149 C
- 170 C
- 221 C
- 234 C
- 281 C
- 301 C
- 350 C
- 350 C
- 361 C

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 Williams<sup>7</sup> indicate a  
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 of crude petroleum  
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 chemical laboratory

These showed an  
 8 Imperial gallons  
 sulphite per ton of  
 mited by Williams<sup>8</sup>

level, sedimentary  
 plan extends south  
 basin is undisturbed  
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an escarpment of Pro-Cambrian rocks. The area is traversed by a  
 number of large streams, notably the Abitibi, Mattagami and Missinaibi.  
 Among these Paleozoic sections, including strata of Ordovician, Silurian,  
 and Devonian age, are exposed.

The possibility of discovering well petroleum in Paleozoic rocks of  
 the James Bay coastal plain, has recently drawn attention to certain  
 Cretaceous shales which outcrop on the Abitibi and Mattagami rivers.  
 These shales, which belong to the Ohio formation, are well exposed at the  
 Long Rapids on the Abitibi river, and to a limited extent above the Long  
 Rapids on the Mattagami river. Sections exposed along the Abitibi are  
 marked by a series of low folds, the general strike of which is approx-  
 imately east and west. The maximum thickness of shale observed at any  
 point was approximately 55 feet.

In places the shales are covered by a comparatively light overburden of  
 boulder clay and other unconsolidated materials. The construction of the  
 proposed extension of the Timiskaming and Northern Ontario railway  
 would bring the deposits within reach of rail transportation.

In 1911<sup>1</sup> these shales were examined by the writer, and samples sub-  
 sequently tested in the laboratory showed a content of petroleum ranging  
 from 7 to 16 Imperial gallons per long ton. The maximum yield of am-  
 monium sulphate was equivalent to 16 pounds per long ton of shale.  
 Partial analyses of three other samples, collected by Dr. M. Y. Williams,<sup>2</sup>  
 indicated a yield of from 3.5 to 12 Imperial gallons (4.2 to 14.4 U.S.  
 gallons) crude petroleum per ton of shale. The calculated yield of am-  
 monium sulphate, based on the nitrogen content, was equivalent to from  
 18.8 to 38.6 pounds per ton. The amount recoverable in actual commer-  
 cial practice would be considerably less.

### Provinces of Manitoba and Saskatchewan

NOTE.—During the field season of 1921 the writer undertook a reconnaissance with a view of determining the  
 probable economic importance of the Cretaceous shales of the Esquimaux hills, Porcupine, Duck and Riding mountains.  
 A complete report embodying the results of this work is in course of preparation.

During recent years, attention has been directed to reported occur-  
 rences of oil shales of Cretaceous age in the provinces of Manitoba and  
 Saskatchewan. Forty-one samples of the shales, representative of a wide  
 area, were collected by the writer during the field season of 1921 and were  
 subsequently tested in the laboratory. The maximum yield of crude  
 petroleum from any sample was 10.9 Imperial gallons (13.1 U.S. gallons),  
 while the maximum yield of ammonium sulphate was equivalent to 3 pounds  
 per ton of shale. The specific gravity of the crude petroleum varies from  
 .944 to .984. All shales examined carry a high percentage of water, the  
 average content of 15 samples being equivalent to 15.4 Imperial gallons  
 (18.4 U.S. gallons) per ton.

It appears, therefore, that the shales examined in the provinces of  
 Manitoba and Saskatchewan are of little present economic importance,  
 as a possible source of petroleum or ammonium sulphate. Should con-  
 ditions at any time warrant commercial development, open cut mining

<sup>1</sup> Report on James Bay Surveys, 1911, S. C. Mills.

<sup>2</sup> Coal Surv. Can. Summary, Sept. 1919, Part C.

<sup>3</sup> Mines Branch, Dept. of Mines, Memorandum Series No. 3, 1921.

could be undertaken in many areas under favourable conditions. Over very considerable areas, shales examined are covered by a comparatively light overburden, consisting chiefly of boulder clays and gravel, which could be readily removed by hydraulic methods.

### Province of British Columbia

The occurrence of oil shales has been reported<sup>1</sup> near Harper's camp in the Cariboo district, near Lytton, and along Calder creek, a tributary of Flathead river. Detailed exploration of these occurrences has not been undertaken, but from present information it appears that the maximum yield of crude petroleum from any sample tested does not exceed 7 Imperial gallons per ton.

On Graham island, the most northerly of the Queen Charlotte group, viscous hydrocarbons have a wide distribution throughout sedimentary and intrusive rocks of Cenozoic and Mesozoic age. Semifluid bitumen is seen as thin films along bedding planes and joint surfaces, as veinlets in various fractured rocks, and in amygdules of certain basalts, as at Tian point. This condition was observed by the writer in 1905, and has subsequently been fully described by J. D. Mackenzie in a report<sup>2</sup> dealing with the geology of Graham island.

The presence of traces of bitumen over a wide area on Graham island was, at one time, interpreted as a possible indication of petroleum pools. Mackenzie considers that the bitumen originates in the Maude formation, a series of dark coloured, fine-grained, thinly laminated and highly fossiliferous argillites, of Lower Jurassic age. He considers that the possibility of discovering commercial pools of petroleum, associated with sediments of Graham island, may be regarded as remote, but suggests the possibility that oil shale bands of commercial value may be found associated with the Maude formation.

### Provisional District of Mackenzie

From time to time, during the past 30 years, the occurrence of oil shale has been reported<sup>3</sup> along the Mackenzie river between Fort Norman and Fort Good Hope. These shales are associated with rocks of Devonian age, but little information is as yet available regarding their thickness, extent, and value as a possible source of petroleum and various by-products. The outcrops occur some 1,500 miles to the north of the city of Edmonton, the nearest large centre of population, and apart from other considerations, their geographical position thus renders them of little present economic importance.

<sup>1</sup>Ann. Rept. of Min. of Mines B.C. 1903 p. 24.  
Ann. Rept. of Min. of Mines B.C. 1904 pp. 23-24.

Can. Geol. Surv. Mem. 88, 1916.

<sup>2</sup>McConnell, R. G. An Exploration in the Yukon and Mackenzie basins, N.W.T. Geol. Surv. Can. Ann. Rept. new series, vol. IV, pt. D, 1888-90 (1900) p. 31.

Kunde, E. M. and Bosworth, T. O. Oil-bearing Rocks of Lower Mackenzie Valley. Geol. Surv. Can. Surv. 1920 Pt. B.

Among the specimens of the SS. Arctic in the beach of Melville landing readily with same class and production, New Brunswick, Newfound

The results of Branch showed 64 gallons of crude oil

In referring to America many writers have attended recognized factors in Scotch oil shale working operating costs, increase from established fields unproductive oil fields by declining market other than oil shale finance only

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