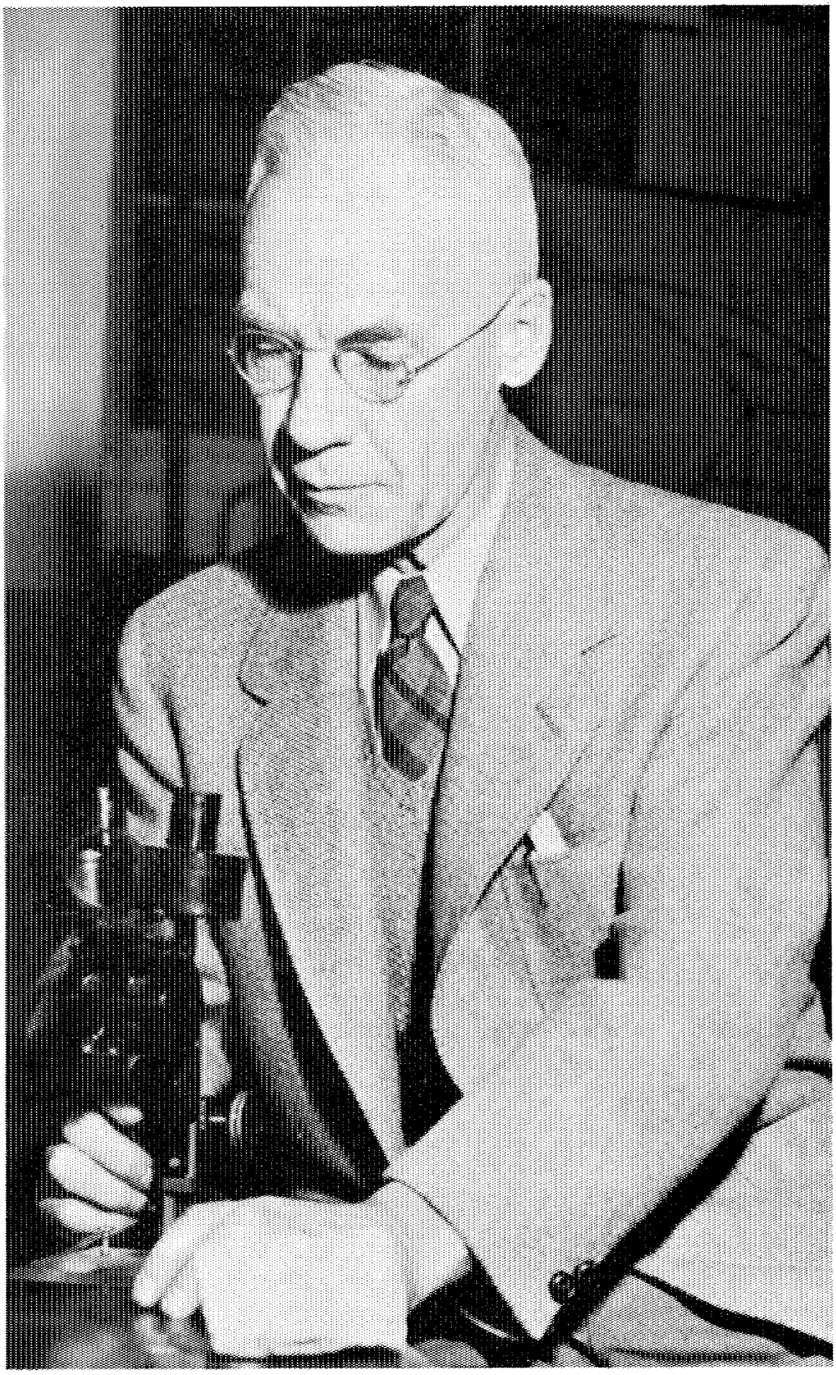

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presented to
K. A. Clark
on the 75th anniversary of his birthday.

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M. A. Carrigy

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FOREWORD

It is a real personal pleasure to extend greetings and best wishes to Dr. Clark on the occasion of his 75th birthday. I should like also to congratulate the Research Council of Alberta on the happy thought of making this presentation.

To Albertans and to many beyond our borders, any reference to Athabasca Oil Sands immediately brings to mind the name of K. A. Clark. The recent applications by a number of oil companies for permission to produce large volumes of oil from the oil sands are the tangible results of the many years of persistent effort by a dedicated scientist. From the original test tube work, through the many pilot plant stages, Dr. Clark's interest and devotion to a single purpose seem never to have wavered.

My own association with Dr. Clark has been a long and pleasant one, and because of this I am sure that the younger students and scientists who have worked with him over the years will remember Dr. Clark's kindly humour and goodwill toward his fellow men probably longer than the details of his many scientific papers.

In the papers of this Volume are many facts and figures concerning the Athabasca Oil Sands. Each one is a tribute to the pioneer work of Dr. K. A. Clark.

E. C. MANNING

Premier of the Province of Alberta

PREFACE

In September, 1962, Research Council of Alberta staff decided that the most fitting tribute they could pay to the pioneer research work of Dr. K. A. Clark on the 75th anniversary of his birthday would be to present him with a volume of papers, written by fellow scientists, on the subject of the Athabasca Oil Sands. To ensure that the volume would be a worthy tribute, research workers in this field of endeavour in universities, government, and industry were contacted and asked to contribute papers; the response to this request has been very gratifying.

The preparation of this volume has been made enjoyable by the excellence of the contributions and by the efforts of many individuals and institutions. It is a pleasure to acknowledge the support given me during the planning stage by the members of the Advisory Committees, and by the Director of the Research Council, E. J. Wiggins. At the editorial level I have been fortunate in being able to call upon G. W. Hodgson, B. Hitchon, R. Green, and L. B. Halferdahl for assistance in preparing the manuscripts for the printer. Redrafting of some of the maps and figures was capably handled by S. J. Groot and his staff.

To provide a forum for discussion of the contributions contained in this volume the Edmonton Geological Society, in co-operation with the Alberta Society of Petroleum Geologists and the Research Council of Alberta, undertook to sponsor the second Conference on the Athabasca Oil Sands held in the Northern Alberta Jubilee Auditorium, Edmonton, Alberta, on October 30th and 31st, 1963. Although manuscripts of several of the papers presented at the conference were not available, this volume does contain the substance of seventeen of the other papers presented and, as such, fulfils the same function as the proceedings of the first Athabasca Oil Sands Conference held in Edmonton in September, 1951.

M. A. Carrigy

Research Council of Alberta
October, 1963

KARL ADOLF CLARK

This volume is dedicated to Karl Clark on the 75th anniversary of his birthday as a token of gratitude for the 43 years he has devoted to the search for a method of producing large quantities of oil, at a competitive price, from the Athabasca oil sands.

Karl Clark came to Alberta in September, 1920 to accept a position with the then newly formed Scientific and Industrial Research Council where he began his long and distinguished career of research into methods of oil-sand separation. Dr. Clark and S. M. Blair built their first hot-water separation plant in the basement of the University powerhouse in 1923. In 1924 a larger plant was erected at the Dunvegan Railway Yards in northwest Edmonton and was operated there until 1929 when it was moved to the north bank of the Clearwater River near Waterways. At the Clearwater River site the plant was operated in partnership with the Mines Branch, Ottawa. Mines Branch staff supervised the digging and delivered the oil sand to the separation plant. The oil produced was delivered to Edmonton where it was used for experimental road paving. This plant offered Clark and his associates their first real opportunity to study and assess the problems that would have to be faced by a commercial venture. It was probably at this time that Dr. Clark saw clearly the tremendous difficulties involved in transferring a very simple process from the laboratory bench to the vast multi-million dollar industrial complex necessary to produce a marketable oil product. Then came the great depression and Dr. Clark along with other Research Council staff were given teaching duties at the University of Alberta. In 1936 Dr. Clark as granted leave of absence to work on a waterflooding project in Trinidad; while there he was offered the post of Professor of Metallurgy at the University of Alberta and he served in that capacity from 1938 until his retirement in 1954. He was appointed head of the Department of Mining and Metallurgy in 1947. After the failure of the wartime venture of Abasand Oil Limited, the government of Alberta decided to build a demonstration plant on the Athabasca River at Bitumount using the hot-water process developed by Dr. Clark. This plant was operated successfully in 1948 and 1949 and stimulated the major oil companies into recognizing the Athabasca oil sands as a potential source of oil for the commercial market.

In 1954 Dr. Clark might have retired to a life of quiet leisure; instead he chose to continue his research and to devote all his time to solving some of the many problems blocking development of the oil sands. He has maintained his office and laboratory space at the Research Council, where hundreds of visitors have sought his advice.

He has impressed all of them with his sincerity and his impartial judgement. Also, during this time he has acted as a consultant and advisor to industry and government, his only concern being that all undertakings and commitments must contribute to the ultimate goal of commercial development.

In August of 1954 Dr. Clark was granted life membership by the Association of Professional Engineers of Alberta. His value to the nation was officially recognized in 1955 when he was the unanimous choice for the Gold Medal of the Professional Institute of the Public Service of Canada.

Nearly 400 papers have been published on all aspects of the Athabasca oil sands and it is of interest to note that two men, K. A. Clark and S. C. Ells have written or contributed to one-fifth of these papers. The appended list of papers written by Dr. Clark indicates the wide variety of oil-sand topics on which he has worked and published.

During his lifetime Dr. Clark has travelled widely. He has met many people and made many friends all of whom will wish him many years of good health and success in all his future experiments.

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CONTENTS

DESCRIPTIONS AND RESERVE ESTIMATES OF THE OIL SANDS OF ALBERTA

J. R. Pow*, G. H. Fairbanks* and W. J. Zamora*

ABSTRACT

This paper has been taken from parts of a more comprehensive and detailed report published by the Alberta Oil and Gas Conservation Board that embodied a study involving the nonconfidential data associated with over 1200 oil-sands evaluation holes and 600 wells. The purpose of this paper is to identify the oil-sands deposits of the Province and to present the Conservation Board's estimate of the reserves that they contain.

The oil-sands deposits are classified into three main groups distinguished from each other by the stratigraphic unit and the area in which they occur. The Athabasca deposit occurs almost entirely within the Wabiskaw-McMurray unit and is located in the northeastern part of Alberta; the Bluesky-Gething deposits exist in the Bluesky and Gething Formations and are situated in the northwestern part of the Province; the Grand Rapids deposits occupy portions of the Grand Rapids Formation and are located in the central part of northern Alberta.

The evaluated portion of the Athabasca deposit occupies about five and one-half million acres and is buried by zero to 2000 feet of overburden. The evaluated portions of the other two groups of deposits together cover over two million acres in area and are buried by between 300 and 2600 feet of overburden.

Reserves have been estimated for those oil sands that, in the opinion of the Board, warrant an evaluation on the basis of the magnitude of the reserves and the adequacy of the data. The majority of the reserves in place were estimated by deriving oil-pay values for each hole or well, plotting the values on maps, connecting the values by isopachs and measuring the reserve volume by means of a planimeter. Intervals having a weight saturation of less than two per cent were excluded from the estimate.

Recovery factors were selected by the Board to convert reserves in place to recoverable reserves of raw oil-sand oil and recoverable reserves of upgraded synthetic crude oil.

The following estimates are listed in terms of reserves in place, recoverable reserves of raw oil-sand oil and recoverable reserves of upgraded synthetic crude oil, respectively, for the deposit or group of deposits designated: Athabasca deposit: 626, 369 and 267 billion barrels; Bluesky-Gething deposits: 51, 28 and 21 billion barrels; Grand Rapids deposits: 33, 18 and 13 billion barrels. The Athabasca reserves are further classified into overburden, oil-saturation and drilling-definition categories. The Bluesky-Gething and Grand Rapids reserves are classified into the drilling-definition categories only.

The Oil and Gas Conservation Board believes that its estimate of the grand total of oil-sands reserves is in reasonable agreement with the actual total reserves in existence within the area evaluated, but it believes that a considerable amount of additional evaluation drilling is needed to establish, with confidence, the magnitude of the reserves within certain widespread portions of these areas. Furthermore, it expects that future evaluation drilling undertaken beyond the evaluated areas will warrant additions to its present reserve estimate.

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*Alberta Oil and Gas Conservation Board, Calgary

INTRODUCTION

This paper has been taken from parts of a more comprehensive and detailed report recently published by the Alberta Oil and Gas Conservation Board.

In the first section of this paper, the oil-sands deposits of Alberta are described with reference to their location, stratigraphic position and depth of burial. In the second section, the Oil and Gas Conservation Board classifies the oil-sands reserves, briefly outlines the methods and recovery factors used to estimate the reserves, presents its reserve estimates and comments on the reliability of its estimates.

The report of the Oil and Gas Conservation Board from which this paper was taken was based mainly on an oil-sands study completed by its staff, although a number of oil-sands publications and several submissions made at public hearings were considered when preparing certain parts of the report. The study covered an area extending across the Province between townships 75 and 105 and it involved the examination of the logs and core analyses available for over 1200 evaluation holes and the logs and sample cuttings taken from approximately 600 wells. The majority of the evaluation-hole data used for this study were supplied to the Conservation Board during the past five or six years pursuant to regulations issued under The Mines and Minerals Act, and all data used for the study have been released for public examination in accordance with a policy established on November 24, 1952, by the Department of Mines and Minerals. The well data were supplied to the Conservation Board and subsequently released in compliance with the Drilling and Production Regulations issued under The Oil and Gas Conservation Act.

The oil sands referred to in this report are primarily distinguished from the other oil-bearing sands of the Province by the type of crude oil that they contain. In The Oil and Gas Conservation Act, the oil sands are defined as those having a "highly viscous crude hydrocarbon material not recoverable in its natural state through a well by ordinary production methods." The oil-sands oil is of a naphthene base, is black in colour and contains a characteristically high percentage of sulphur, nitrogen and trace metals. Relative to the conventionally produced crude oils of the Province, it is heavy, having a gravity that varies considerably but which, over the Athabasca deposit, averages about 10.5 degrees A.P.I. at 60°F. The gravity of the oil in the Peace River area is similar to that of the Athabasca area, but several minor deposits located some 100 miles northeast of the town of Peace River appear to have a lighter-gravity oil and, therefore, they may not qualify under the oil-sands definition of The Oil and Gas Conservation Act.

DESCRIPTIONS OF THE DEPOSITS

IDENTIFICATION

The Oil and Gas Conservation Board has evaluated the oil sands deposits shown on figure 1. These areas total approximately 8,000,000 acres and, therefore, underlie almost one twentieth of the entire Province of Alberta.

Between the shaded areas are extensive areas that may contain additional oil-sands reserves. Data collected to date reveal the widespread existence of oil staining, low oil-saturation and the local occurrence of medium and high saturation within these intervening areas, but the data are considered to be too inexact and incomplete to permit a detailed reserve evaluation of these areas.

The oil sands of Alberta occur within three distinct stratigraphic units of Early Cretaceous age. Each unit is shown on figure 2, a geological section that extends from a few miles west of the town of Peace River to several miles southeast of the town of Fort McMurray, a distance of about 280 miles. The section intersects the three largest oil-sands deposits of the Province. In the eastern part of the section, the Athabasca deposit is seen to occupy a major part of the Wabiskaw-McMurray stratigraphic unit. In the western part of the section, the Peace River deposit is noted to occur within the stratigraphically equivalent Bluesky-Gething unit. The Wabasca deposit overlies the western edge of the Athabasca deposit and occupies two standstone members of the higher Grand Rapids Formation. Other minor deposits, located north of the section, occur in the Bluesky-Gething and Grand Rapids units.

The evaluated oil-sands deposits are separately designated by name and formation in figure 3. This map corresponds to figure 1 in that it shows only those oil-sands areas that have been evaluated by the Oil and Gas Conservation Board. No reference is made on this map to the leaner, more localized or undelineated oil sands that may exist beyond the evaluated areas.

The enormous Athabasca deposit of the Wabiskaw-McMurray unit is shown in figure 3. The evaluated portion of this deposit occupies an area in excess of five and three-quarter million acres and it contains about 88 per cent of the total evaluated oil-sands reserves of the Province.

The second most important group of deposits, from the standpoint of reserves, is outlined in the western part of figure 3. They have been collectively named the Bluesky-Gething deposits after the stratigraphic unit in which they occur. They constitute the Peace River deposit and the smaller Buffalo Head Hills and Loon River deposits. The evaluated portion of the Peace River deposit occupies an area in excess of one million acres, and the latter two deposits occupy at

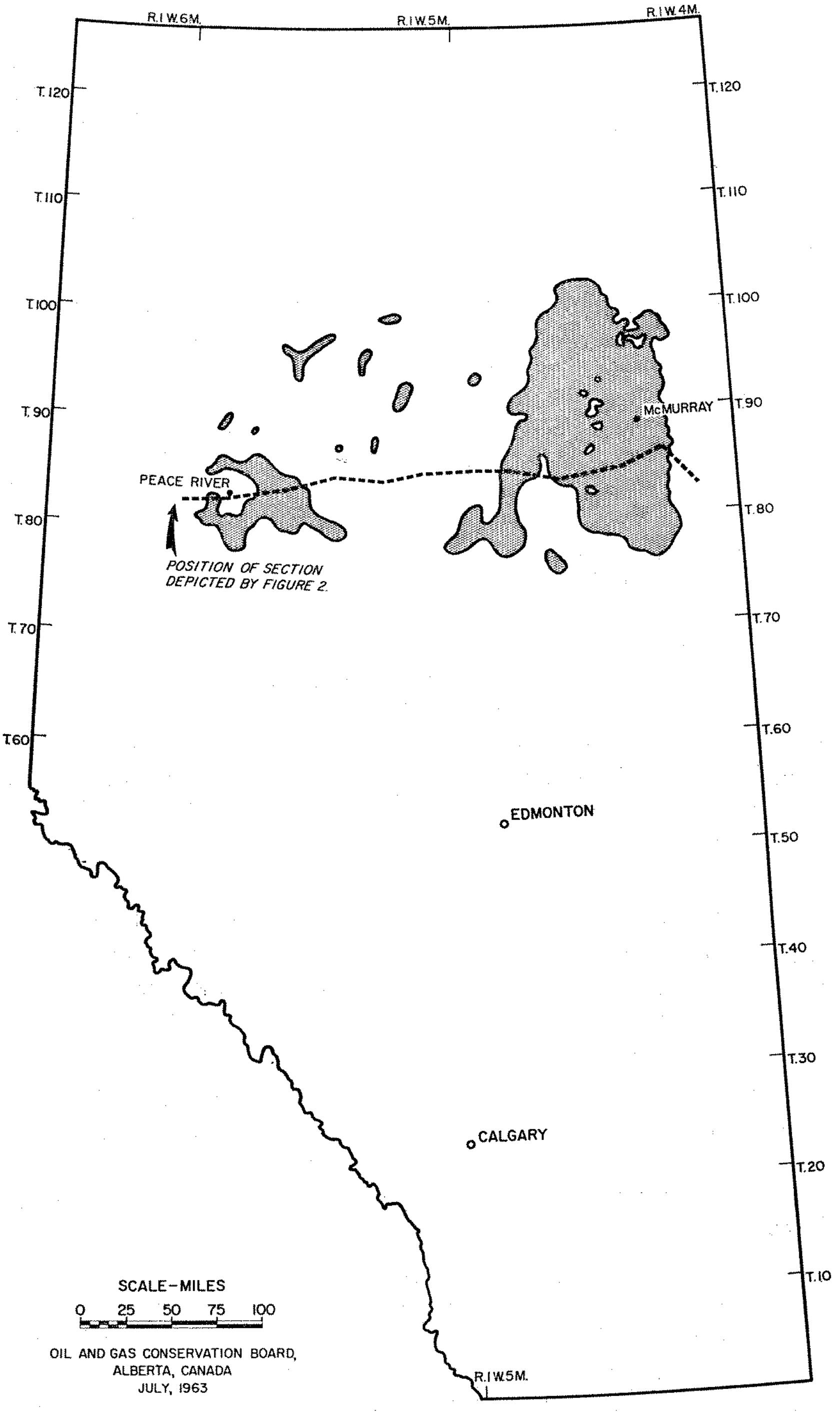

FIGURE 1. *Location of evaluated oil-sands reserves in Alberta.*

least 150,000 acres. The Bluesky-Gething deposits contain about seven per cent of the total evaluated oil-sands reserves of the Province.

The remaining deposits, located in the central part of figure 3, are collectively referred to as the Grand Rapids deposits, being so named after the formation in which they occur. They constitute the large Wabasca deposit together with the less significant Pelican and Bad Rapids deposits. The evaluated portion of these deposits occupies a total of about one million acres and contains approximately five per cent of the presently known oil-sands reserves.

DEPTH OF BURIAL

Except for the localized outcrop areas associated with the Athabasca River and its tributaries, the evaluated oil-sands areas of Alberta are covered by overburden.

The overburden generally consists of a thin layer of soil mantle and glacial drift together with the strata that occur between the drift and the oil sands. These formations, consisting mainly of soft sandstones, siltstones and shales, are shown and named in figure 2. It is evident from figure 2 that variations in the thickness of the overburden are caused by variations both in the structure or dip of the underlying oil sands and in the surface topography.

The thickness and configuration of the overburden covering the evaluated oil-sands areas is briefly described below for each of the three main groups of deposits.

Athabasca Deposit

The Athabasca deposit is covered by less than 100 feet of overburden in the vicinity of that part of the Athabasca River north of township 90. From that area the overburden abruptly thickens, eastward to 600 feet, northward to 2,000 feet and westward to a maximum of 1,600 feet. Towards the south, and particularly the southwest, the rate of overburden thickening is more gradual but maximum thicknesses of 1,500 and 700 feet, respectively, are attained in those directions.

Bluesky-Gething Deposits

The overburden covering the evaluated portion of the Peace River deposit increases in a westward direction from 1,500 to 2,300 feet and in a southward direction from 1,500 to 2,600 feet. The larger Buffalo Head Hills deposit is buried by between 1,100 and 2,100 feet of overburden and the small Buffalo Head Hills and Loon River deposits are covered by overburden that averages between 800 and 1,600 feet in thickness.

Grand Rapids Deposits

The overburden covering the large Wabasca deposit thickens in a southward direction from 300 to 1,100 feet. The Bad Rapids deposits are buried by between 500 and 1,100 feet of overburden, and the

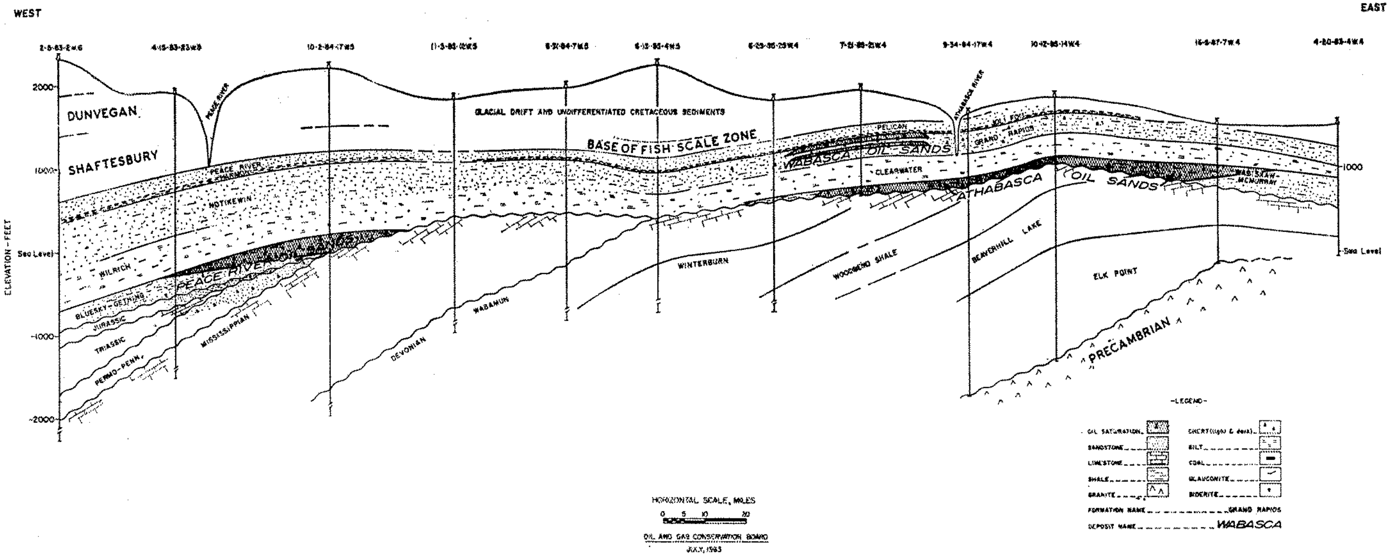

FIGURE 2. Schematic geological cross section showing oil-sand deposits and associated formations between the western boundary of the Peace River deposit and the eastern boundary of the Athabasca deposit (see Figure 1 for location of cross section).

Pelican deposits are covered by between 1,000 and 1,400 feet of overburden with an approximate minimum of 600 feet in the valley of the Athabasca River.

RESERVES

The Oil and Gas Conservation Board has estimated the reserves of those oil sands that, in its opinion, warrant an evaluation on the basis of the magnitude of the reserves and the adequacy of the data. The areas for which the reserves have been estimated are outlined on figure 3.

In this section, the Conservation Board describes the reserves-classification system adopted for this paper, summarizes its evaluation methods, presents an abbreviated tabulation of the oil-sands reserves and discusses the reliability of the estimates.

CLASSIFICATION

For the purpose of this paper, the reserves of the Athabasca, Bluesky-Gething and Grand Rapids deposits are classified in the manner shown by figure 4. A more detailed classification of the reserves is provided in the Conservation Board's report, in which eight overburden categories are employed and in which the reserves of each overburden category are subdivided into the saturation and drilling-definition categories.

The classification scheme shown by figure 4 is based upon three factors: the depth to which the reserves are buried, the extent to which the host sand is saturated and the degree to which the reserves have been defined by drilling. Each factor and the categories related to it are outlined below.

Depth of Burial

The reserves of the Athabasca deposit are grouped into the following three overburden categories, the limits of which were arbitrarily selected: zero to 100 feet, 100 to 250 feet, and greater than 250 feet. The reserves of Bluesky-Gething and Grand Rapids deposits are covered by more than 250 feet of overburden and, therefore, qualify under the third category only.

The reserves of the Athabasca deposit were subdivided into the three overburden categories by the use of an overburden isopach map representing the interval from surface to the highest occurrence of oil sand having an oil saturation greater than 10 per cent by weight. For this purpose, the highest occurrence of the 10 per cent sand was chosen as the base of the overburden because it generally represented the beginning of an interval of continuous saturation, whereas the highest occurrences of leaner sands were more often noted to be isolated from the main oil-sands development. However, such higher sands of lesser saturation were considered when estimating the reserves.

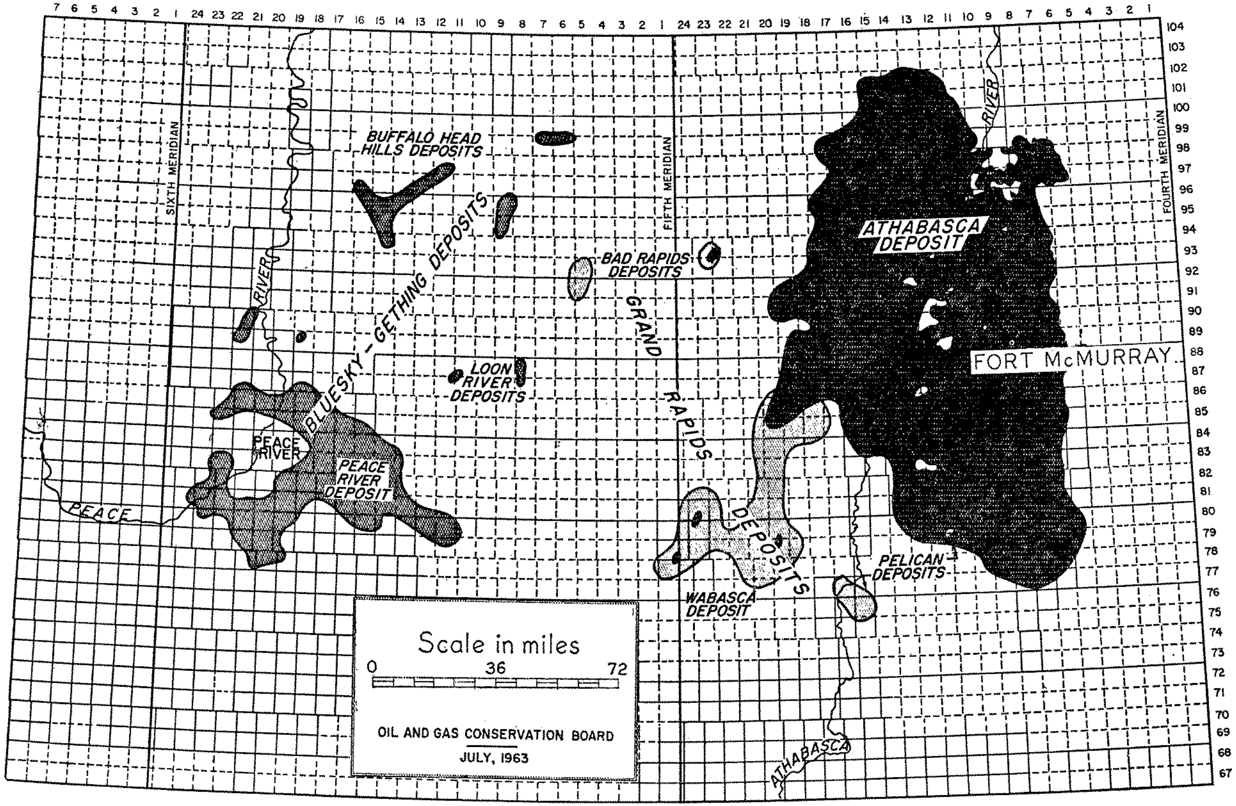

FIGURE 3. *Oil sands of northern Alberta evaluated by the Oil and Gas Conservation Board, 1963.*

If these overburden categories had been established on the basis of an isopach map representing the interval from surface to the highest occurrence of a leaner sand, the zero to 100-foot overburden area would have been slightly increased, the 100 to 250-foot overburden area would have remained about the same and the thicker overburden area would have been negligibly reduced from the area used for this study.

Oil Saturation

A classification based on the degree of saturation has been applied only to the reserves of the Athabasca deposit. It has not been applied to the reserves associated with the Bluesky-Gething or Grand Rapids deposits because the data available to the Board at the time of the study were considered to be inadequate.

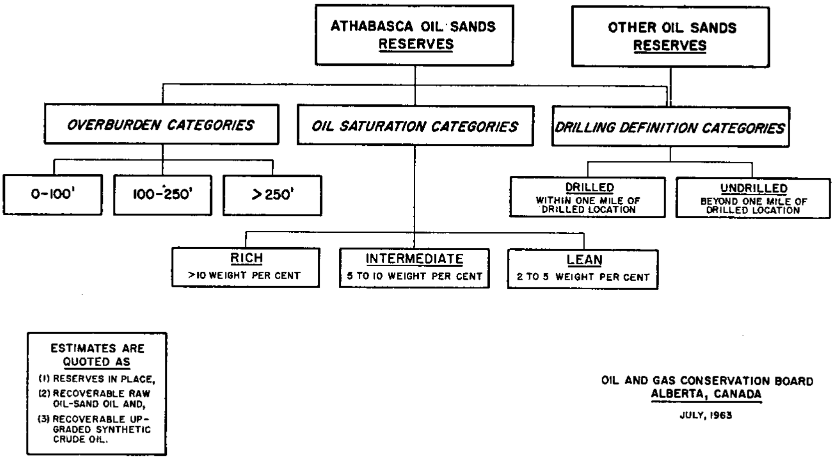

FIGURE 4. Classification of oil-sands reserves.

The following three categories have been used to classify the Athabasca reserves: (1) rich-sand category, comprising those sands having an oil saturation greater than 10 weight per cent (19.2 volume per cent); (2) intermediate-sand category, comprising the sands having an oil saturation of between 5 and 10 weight per cent (9.9 and 19.2 volume per cent), and (3) lean-sand category, representing the sands having a saturation of between 2 and 5 weight per cent (4 and 9.9 volume per cent). The limits of these three categories were selected arbitrarily.

Since the reserves estimates of the Bluesky-Gething and Grand Rapids deposits were based on a visual estimate of saturation, no definite minimum saturation cut-off can be cited as a basis for the estimates. However, when the pay thicknesses were determined, attempts were made to exclude material that appeared to be below the lean-sand category.

Definition by Drilling

The reserves of each deposit have been further subdivided into the drilled and undrilled categories.

The drilled reserves are defined as these reserves situated within 1 mile of the evaluation hole or well on which they are based. For holes or wells separated by more than 2 miles, the area of the drilled reserves assigned to each drilled location was that of a circle having a radius of 1 mile. In cases where the holes or wells are separated by less than 2 miles or where unproductive acreage obviously occurred within 1 mile of the hole or well, the areas of the drilled reserves were adjusted accordingly.

The undrilled reserves constitute those reserves located beyond 1 mile from drilled locations. They are equal to the difference between the total reserves, as evaluated by isopachs, and the drilled reserves.

ESTIMATION METHODS

Certain methods have been developed by the Oil and Gas Conservation Board to estimate the reserves of the oil sands. These methods take into account the following factors peculiar to the oil sands or their evaluation data: (1) the spacing of the holes or wells at which the data were taken is haphazard and generally wide, (2) the core analyses usually express oil saturation in terms of a weight percentage rather than a volume percentage, (3) for certain widespread areas, the only records available to the Board to determine oil saturation consisted of sample cuttings and logs, (4) many electrical logs were suitable only for qualitative, rather than quantitative, determinations, and (5) the recovery of the oil-sand oil is dependent upon either mining or artificial stimulation techniques and not upon the natural drive applicable to conventional fields.

The following summary is with reference to the general approach taken to estimate the oil-sands reserves. Additional detail concerning the estimation methods and the reserve factors used for the study is supplied in the Conservation Board report from which this paper was taken.

In the case of the rich and the intermediate sands of the Athabasca deposit, the "oil-feet"* were separately derived from core analyses and logs, plotted on separate maps, and the values were connected by isopachs to provide maps representing the volume of the rich and intermediate oil-sands reserves in place. The overburden map was superimposed on each oil-pay isopach map, and the reserve volumes were determined by planimeter for each overburden category. Drilled reserves were calculated by multiplying the area situated within 1 mile of each drilled location by the appropriate pay values.

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*"Oil-Feet" is defined as the product of oil-sand thickness and volume fraction oil saturation

To estimate the reserves contained by the lean sands of the Athabasca deposit, some 480 core analyses were first processed to determine the ratio of core-analysis oil pay contained by the lean oil-sands category to the core-analysis oil pay contained by the intermediate category. The reserves contained in the lean sands under a particular overburden category were then calculated by multiplying this ratio by the total reserves of the intermediate sands belonging to the same overburden category. Owing to the adoption of this short-cut method, no need arose to either calculate individual pay thicknesses or saturations or to prepare a reserve map for the lean oil sands.

Owing to the lack of core analyses available for the Bluesky-Gething and Grand Rapids deposits, the aforementioned estimation methods could not be applied to these deposits. Instead, the thickness of the oil sand was estimated from well logs, and the saturation of the sand was estimated from the appearance of the sample cuttings. Oil-foot values were calculated from the two factors, plotted on maps and connected by isopachs. The reserves were not classified according to the saturation of the sand, due to the inadequacy of the data available for the study.

The final stage of the reserve calculations involved the conversion of reserves in place to recoverable reserves. For this purpose, the Board, having regard to evidence submitted to it at public oil-sands hearings, selected a set of recovery factors which are outlined in the following section of this paper.

RECOVERY FACTORS

For the purpose of this paper, the Conservation Board has employed the recovery factors of table 1 to convert the reserves in place to recoverable reserves.

In selecting the recovery factors for the reserves associated with the zero to 100-foot overburden category, the Conservation Board considered the separate evidence submitted by Great Canadian Oil Sands Ltd. and the Cities Service Athabasca group at public hearings

TABLE 1. ESTIMATED RECOVERY FACTORS
(expressed as a percentage of the oil-sand oil in place)*

Range of overburden thickness (feet)	Recovery of raw oil-sand oil (generally between 9 and 13° API at 60°F) (per cent)	Recovery of upgraded synthetic crude oil (30 to 37° API at 60°F) (per cent)
0-100	85	60
100-250	70	50
250 and greater	55	40

*Based on the Oil and Gas Conservation Board cut-off of two per cent oil saturation by weight held during 1960 and 1962. Both applications pertained to mining and plant-processing schemes and they applied to reserves that, for the most part, occurred beneath zero to 100 feet of overburden.

In choosing the recovery factors listed for the reserves covered by more than 250 feet of overburden, the Conservation Board considered evidence submitted during 1963 by the Shell Oil Company of Canada Ltd. with reference to its application to recover oil-sands oil by injecting steam or hot aqueous solutions, or both, into the oil sands.

The recovery factors used for this study with reference to the 100 to 250-foot overburden category represent an arithmetical mean between those employed for the thinner and thicker overburden categories and are based on the assumption that these reserves will be recovered in part by mining and in part by *in-situ* processes.

TABULATION OF RESERVES

The Alberta Oil and Gas Conservation Board has estimated that the oil sands of northern Alberta contain over 700 billion* barrels of oil in place. Of this total, over 400 billion barrels are considered as recoverable raw oil-sand oil, from which about 300 billion barrels of upgraded synthetic crude oil are expected to be produced. It is obvious that this reserve is enormous for, by comparison, only 18.4 billion barrels of conventional oil, in place, or 5.1 billion barrels of conventional recoverable oil have been found to date in the Province (Oil and Gas Conservation Board, 1962).

TABLE 2. EVALUATED RESERVES OF THE ATHABASCA DEPOSIT, CLASSIFIED BY OVERBURDEN CATEGORIES

Overburden (Feet)	Reserves in place (billion bbls.)	Recoverable raw oil-sands oil (billion bbls.)	Recoverable upgraded synthetic crude oil (billion bbls.)
0-100	45.2	38.4	27.2
100-250	75.3	52.7	37.7
Greater than 250	505.4	278.0	202.0
Total	**625.9**	**369.1**	**266.9**

The reserve estimates are listed (Tables 2 to 6) in accordance with the simplified system of classification employed for this paper. A more detailed subdivision of the estimates is contained in a report recently published by the Oil and Gas Conservation Board.

In the reserve tabulation, the estimates are presented in terms of billions of barrels and they have been rounded to the nearest one tenth of a billion barrels.

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*Billion, one thousand million. Ed.

TABLE 3. EVALUATED RESERVES OF THE ATHABASCA DEPOSIT, CLASSIFIED BY SATURATION CATEGORIES

Saturation category	Reserves in place (billion bbls.)	Recoverable raw oil-sands oil (billion bbls.)	Recoverable upgraded synthetic crude oil (billion bbls.)
Rich sands (greater than 10% weight saturation)	440.8	260.5	188.3
Intermediate sands (5 to 10% weight saturation)	145.7	85.5	61.9
Lean sands (2 to 5% weight saturation)	39.4	23.1	16.7
Total (greater than 2% weight saturation)	**625.9**	**369.1**	**266.9**

TABLE 4. EVALUATED RESERVES OF THE ATHABASCA DEPOSIT, CLASSIFIED BY DRILLING DEFINITION

	Reserves in place (billion bbls.)	Recoverable raw oil-sands oil (billion bbls.)	Recoverable upgraded synthetic crude oil (billion bbls.)
Drilled	140.1	92.8	66.5
Undrilled	485.8	276.3	200.4
Total	**625.9**	**369.1**	**266.9**

TABLE 5. EVALUATED RESERVES OF THE BLUESKY-GETHING DEPOSITS*

	Reserves in place (billion bbls.)	Recoverable raw oil-sands oil (billion bbls.)	Recoverable upgraded synthetic crude oil (billion bbls.)
Drilled	4.7	2.6	1.9
Undrilled	46.8	25.7	18.7
Total	**51.5**	**28.3**	**20.6**

*The reserves of the Peace River deposit comprise about 98 per cent of the total evaluated Bluesky-Gething reserves.

RELIABILITY OF THE ESTIMATES

The Alberta Oil and Gas Conservation Board believes that its estimate of the total oil sands reserves for the combined deposits is in reasonable agreement with the actual reserves in existence within the areas evaluated. However, it believes that a considerable amount of additional evaluation-drilling is needed to establish, with confidence,

TABLE 6. EVALUATED RESERVES OF THE GRAND RAPIDS DEPOSITS*

	Reserves in place (billion bbls.)	Recoverable raw oil-sands oil (billion bbls.)	Recoverable upgraded synthetic crude oil (billion bbls.)
Drilled	2.0	1.1	0.8
Undrilled	31.4	17.2	12.5
Total	**33.4**	**18.3**	**13.3**

*The reserves of the Wabasca deposit comprise about 91 per cent of the total evaluated Grand Rapids reserves.

the magnitude of the reserves within certain widespread portions of the area classified as undrilled in this report. Such drilling would not only permit a more accurate estimate of the total oil sands reserves, but it would enable a more reliable geographic identification of the undrilled reserves, which constitute almost 80 per cent of the reserves of the Athabasca deposit and over 90 per cent of the reserves of the other deposits.

The Conservation Board expects that additional oil sands reserves will be delineated when evaluation drilling is conducted beyond the areas evaluated for the purpose of this report. The possibility of such a reserve growth is particularly applicable to the Bluesky-Gething and Grand Rapids deposits.

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GEOLOGICAL CONTROLS ON THE DISTRIBUTION OF ATHABASCA OIL SAND RESERVES

G. A. STEWART*

ABSTRACT

The Athabasca oil sands are the world's largest known reserve of oil. Five hundred and sixty billion barrels of oil are contained within an area of approximately 13,000 square miles. Pre-Cretaceous topography developed on the eroded Devonian surface controls in part the distribution of Athabasca oil sand reserves. Both structural features and irregularities resulting from the processes of erosion contribute to the configuration of the pre-Cretaceous landscape.

INTRODUCTION

The Athabasca oil sands contain the world's largest known reserve of oil. Since the dawn of this century men have speculated about the quantity of oil contained in this reservoir. However, it is just recently that enough data have become available to estimate the oil reserves realistically. Data from 1,570 exploratory wells within an area of 13,000 square miles indicate reserves of approximately 560 billion barrels of oil.

HISTORY OF EXPLORATION

From the time of the original work of Bell (1884), and McConnell (1891), until S. C. Ells' (1926) summary report, thirty-four exploratory wells had been drilled, principally along the banks of the Athabasca River. The largest number of these early wells were drilled by A. von Hammerstein and Athabasca Oils Limited. During the same period Ells (1926) extensively surveyed the outcrops along the lower Athabasca River and its major tributaries.

Further studies were carried on from 1924 to 1942 by the Scientific and Industrial Research Council of Alberta, under the guidance of Dr. K. A. Clark. Several companies, including Abasand Oils Limited and the Consolidated Mining and Smelting Company of Canada, were also working at this time. The many samples from outcrops and shallow drill-holes were insufficient to provide a basis for valid reserve estimates.

In 1942, Federal authorities initiated the first concerted attempt to appraise oil reserves in the Athabasca area. At the conclusion of the government program in 1947, 291 test holes had been drilled, and three important oil-sand deposits outlined, namely the Mildred Lake, Steepbank, and Horse River deposits.

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*Imperial Oil Limited, Calgary

Since 1947, oil companies have drilled a large number of exploratory test wells within an area of approximately 13,000 square miles. The first major drilling effort during this period was conducted between 1952 and 1954 by a group of oil companies known as the Athabasca Oil Sands Project. Approximately 100 wells outlined several more rich oil-sand deposits in addition to those previously discovered by the Federal Government. After 1954, thirty oil companies drilled approximately 1,300 exploratory test holes which have contributed significantly to the knowledge of oil reserves in the Athabasca area.

GENERAL GEOLOGY

In the northeastern corner of Alberta, adjacent to the Canadian Shield, formations of Cretaceous age outcrop (Fig. 1). The earliest Cretaceous deposits in this area, the McMurray Formation, lie unconformably on an eroded Devonian surface. The McMurray Formation, with an average thickness of 175 feet, is comprised essentially of sands and silts impregnated with heavy, viscous oil, and these portions of the formation are commonly referred to as Athabasca oil sands. Marine sands and shales of the Clearwater Formation, which vary in thickness from 250 to 300 feet, overlie the McMurray Formation. Grand Rapids sandstones averaging 280 feet in thickness overlie the Clearwater Formation. The remainder of the Lower Cretaceous section, which attains a maximum thickness of 1000 feet, is represented by dark, marine shales of the La Biche Formation. The total Cretaceous section in this area is approximately 2000 feet thick.

Irregular pre-Cretaceous topography, developed on the eroded Devonian surface, plays a major role in controlling the thickness and areal extent of the overlying McMurray Formation. McMurray sands and silts tend to fill in Devonian limestone valleys and thin over knobs and ridges (Fig. 2, cross section A-A′). This general levelling-out effect by the McMurray Formation is somewhat obscured by local post-Cretaceous folding (Fig. 2, cross section B-B′). Furthermore, the distribution of oil contained within the McMurray Formation is, in part, controlled by this ancient topography.

PRE-CRETACEOUS TOPOGRAPHY

The major factors controlling the configuration of the surface of the pre-Cretaceous unconformity are the Devonian, Nisku (D-2)-Grosmont topographic high, leaching of Elk Point evaporites, and deep-seated structures involving Precambrian rocks. Post-Cretaceous deformation tends to complicate the interpretation of these controlling factors.

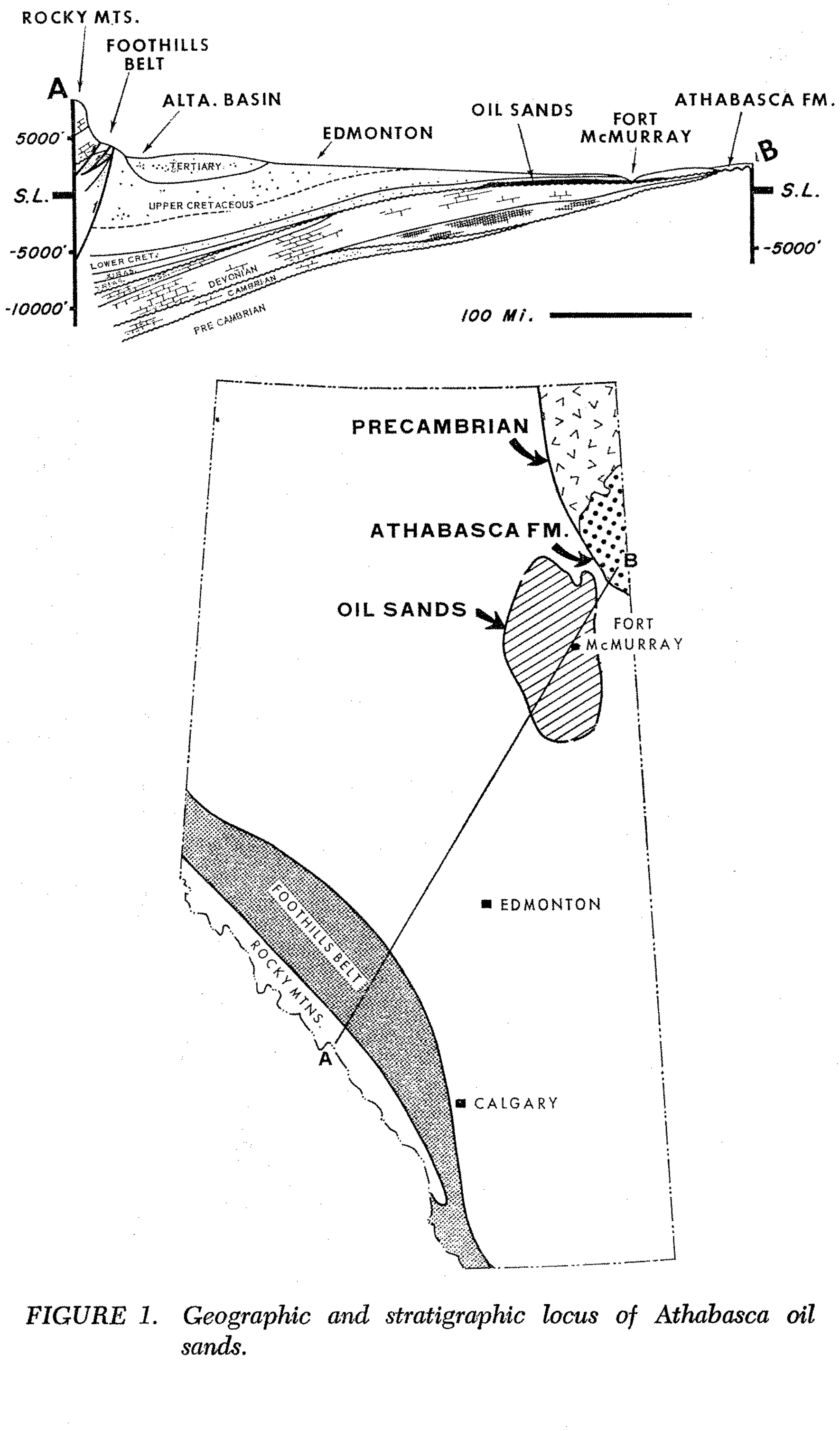

FIGURE 1. Geographic and stratigraphic locus of Athabasca oil sands.

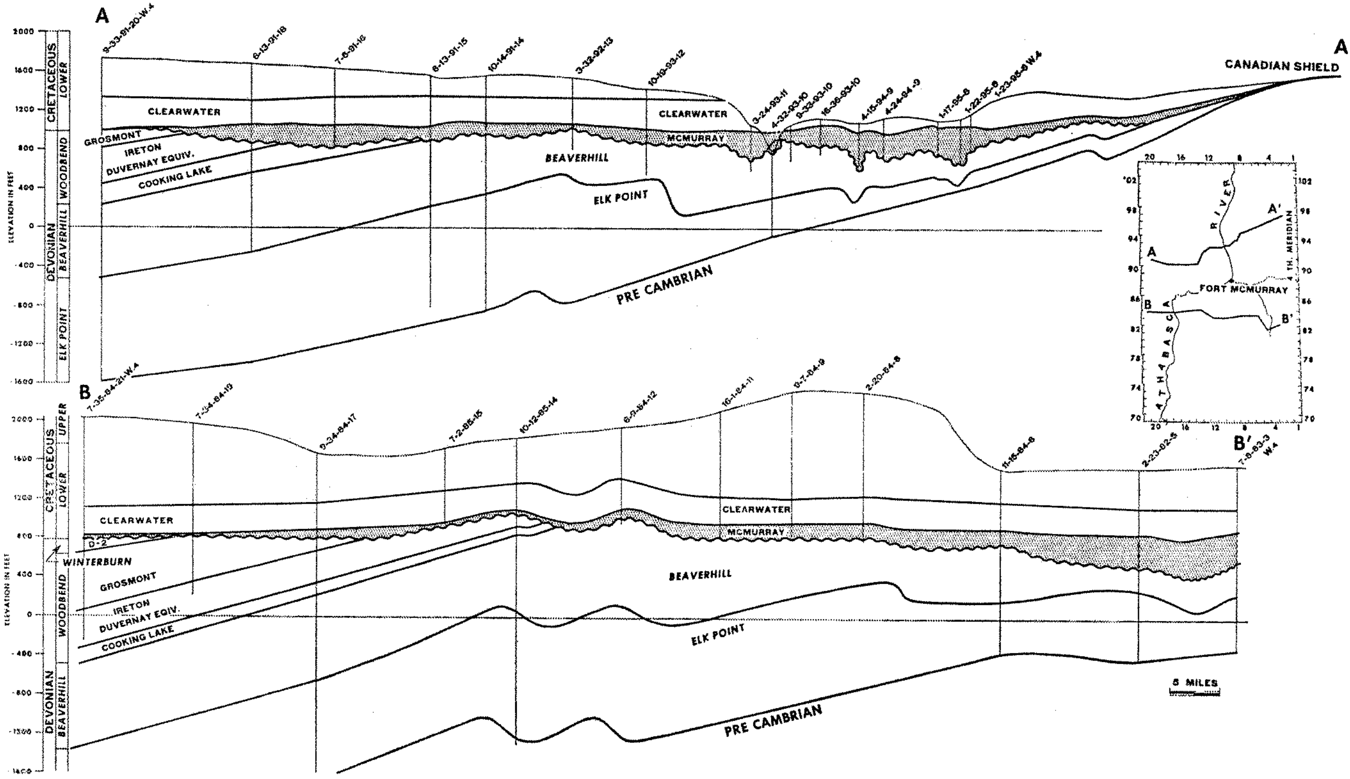

FIGURE 2. *East-west geologic sections showing variations in thickness of the McMurray Formation north and south of Fort McMurray.*

NISKU-GROSMONT TOPOGRAPHIC HIGH

Differential erosion of the Devonian subcrop left a resistant ridge of Nisku-Grosmont carbonates across the western part of the area (Fig. 4). This ridge was a highland during McMurray time, as evidenced by the absence of McMurray strata in this western area (Fig. 3).

ELK POINT LEACHING

The western edge of leaching of Elk Point evaporites is shown on figure 4. East of this boundary removal of the salt by solution has caused a general down-warping. This depression is of major importance as a substantial portion of Athabasca oil reserves are concentrated within it (Fig. 6). In the northeastern part of the area, leaching has been more severe, resulting in the development of a karst topography (Fig. 2, cross section A-A′; Fig. 3).

DEEP-SEATED STRUCTURES

Deep-seated structures, apparently the result of block faulting in the Precambrian basement, reflect as ridges on the unconformity surface. Steep aeromagnetic gradients which coincide with these ridges tend to substantiate this idea. The most predominant ridge on the unconformity surface is a long, arcuate feature, running in a northerly direction through the middle of the area. The McMurray Formation thins markedly over this ridge, being only 20 feet thick at its summit (Fig. 3). The sands overlying this ridge contain relatively small amounts of oil as compared to the major concentrations found in the basins (Fig. 6). Several similar ridges are present throughout the Athabasca area. The most notable of these are near Fort MacKay (Tp. 94, R. 10), in the Firebag River area (Tp. 97, R. 7), and at Cottonwood Creek (Tp. 89, R. 5) on the Clearwater River. Although direct evidence is lacking, these ridges are probably caused by faulting of underlying Precambrian rocks. In general, the McMurray Formation becomes thinner and contains relatively small quantities of oil over these ridges.

POST-CRETACEOUS STRUCTURE

Post-Cretaceous deformation also contributes to the configuration of the unconformity surface. Such deformation complicates structural studies of pre-Cretaceous topography. The Bitumount Basin (Tp. 96, R. 10) is one example of such activity. At this location post-Cretaceous leaching of Elk Point evaporites has caused a collapse of the overlying McMurray and Clearwater Formations of the order of 200 feet (Fig. 5). Another possible example of post-Cretaceous movement is found near Telegraph Creek (Tp. 84, R. 12) where the tops of the McMurray and Clearwater Formations are anomalously high by approximately 150 feet (Carrigy and Zamora, 1960). This anomaly appears to be related to post-Cretaceous activity of Precambrian rocks (Fig. 2, cross section B-B′; Fig. 5).

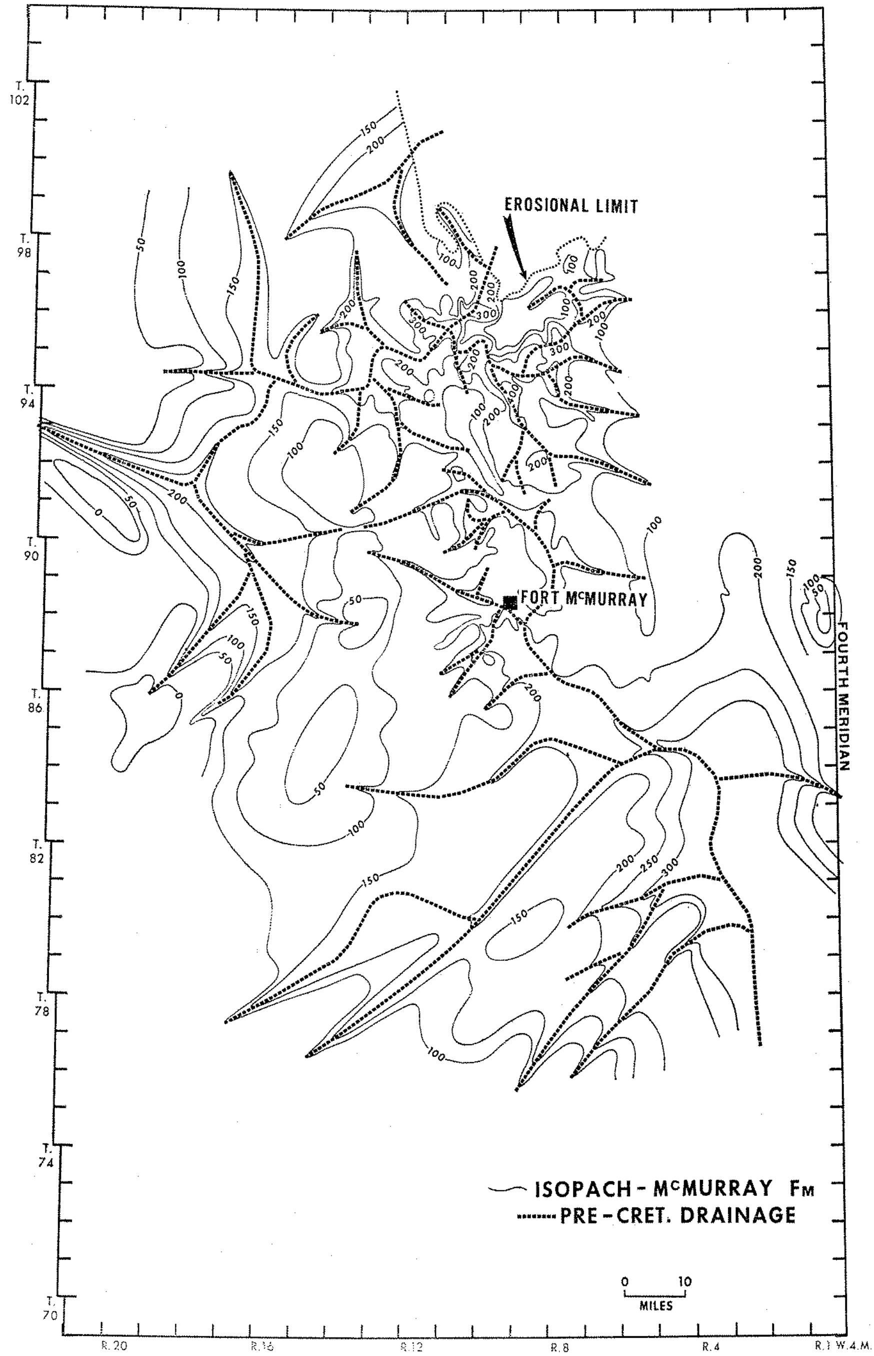

FIGURE 3. *Isopachs of the McMurray Formation and inferred drainage pattern in pre-Cretaceous time.*

On a regional scale, post-Cretaceous tilting has caused the strata to dip to the southwest, further complicating the unconformity surface.

PRE-CRETACEOUS EROSION

A major break in the depositional sequence of the Athabasca area occurred between Devonian and Early Cretaceous times. The area was subjected to several periods of subaerial erosion and weathering as shown by the residual, calcareous shales preserved beneath the base of the McMurray Formation. The result of this extended weathering and erosion was a landscape characterized by wide river valleys, and low, rounded hills. An attempt has been made to reconstruct the drainage system of this area as it existed immediately before the deposition of the McMurray Formation (Fig. 3).

OIL-SAND RESERVES

CONTROL

Comparison between the topography of the unconformity surface and the distribution of Athabasca oil sands reveals that river valleys developed by the ancient drainage system shown on figure 6 partly control the distribution of oil reserves. Three major concentrations of reserves are found in the Athabasca area. The first, in the Elk Point depression, contains a large proportion of Athabasca oil reserves. The second, in the western part of the area north of the Athabasca River (Fig. 6), contains reserves in a basinal area previously occupied by a large mature river. The third major development of oil sands, in the southern part of the area, is associated with a broad flood plain. Rivers originating in the Devonian, Nisku-Grosmont highlands to the west flowed eastward across this plain into the Elk Point depression.

Within the ancient river valleys, thick sections of McMurray sediments accumulated. These sections indicate that a favourable environment existed for the deposition of sands. Consequently, thick reservoir sections predominate in these ancient valleys.

EXTENT

The areal extent of the McMurray oil sands is fairly well known (Fig. 6). To the west the McMurray Formation pinches out against the Nisku-Grosmont high. To the northwest McMurray reservoir sands grade into dark marine shales. Recent glacial erosion marks the limit of McMurray oil sands in a north and northeasterly direction. The limit of the field to the east is not as clearly defined, although drilling in this area indicates a relatively high surface causing a marked thinning of the McMurray Formation. This thinning is particularly evident along the Firebag River. Outcrop surveys along the Clearwater River found McMurray sands to be devoid of oil east of Cottonwood Creek. The southeasterly boundary of the field is outlined by a fairly well defined oil-water contact. The extent of oil

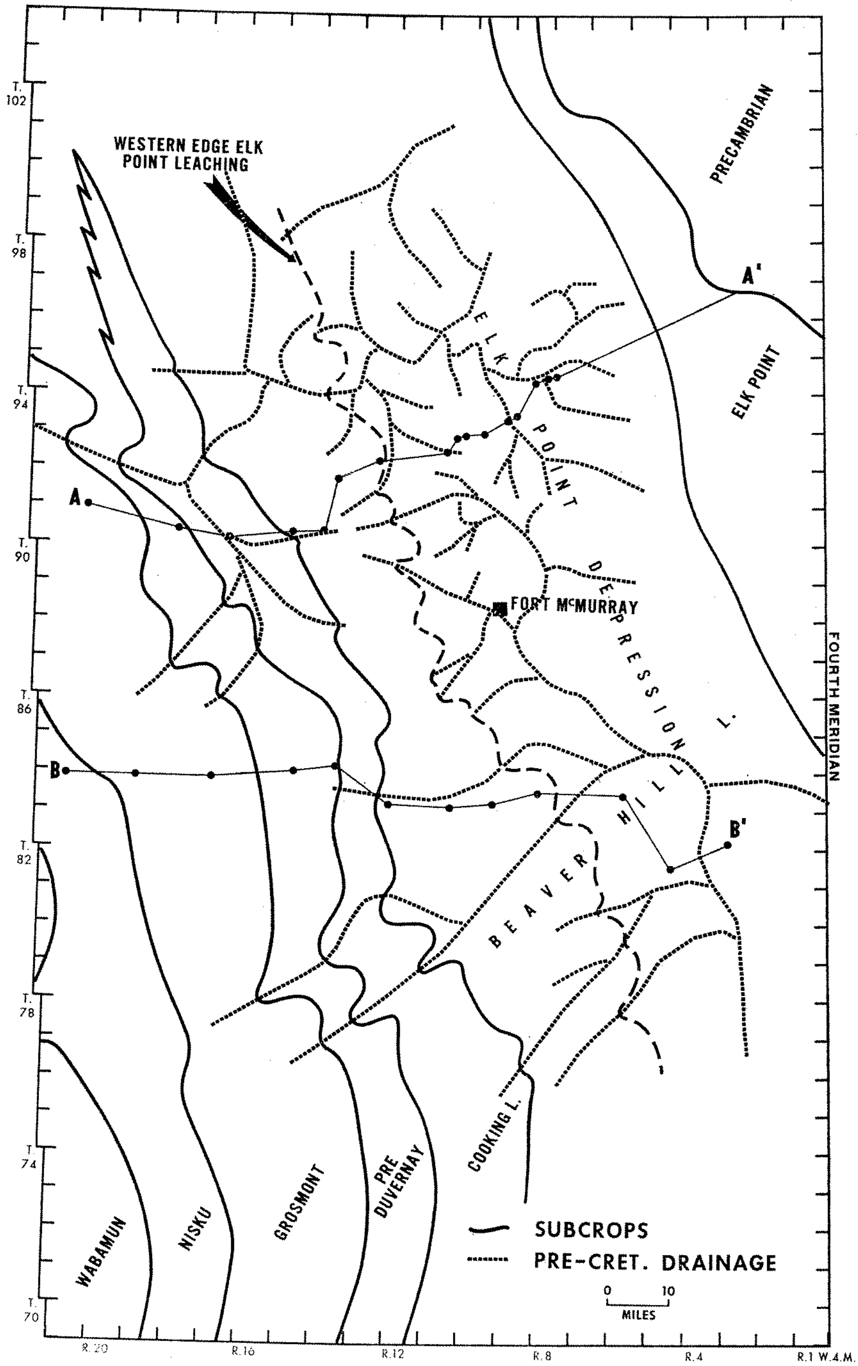

FIGURE 4. *Subcrops of older strata at the pre-Cretaceous unconformity.*

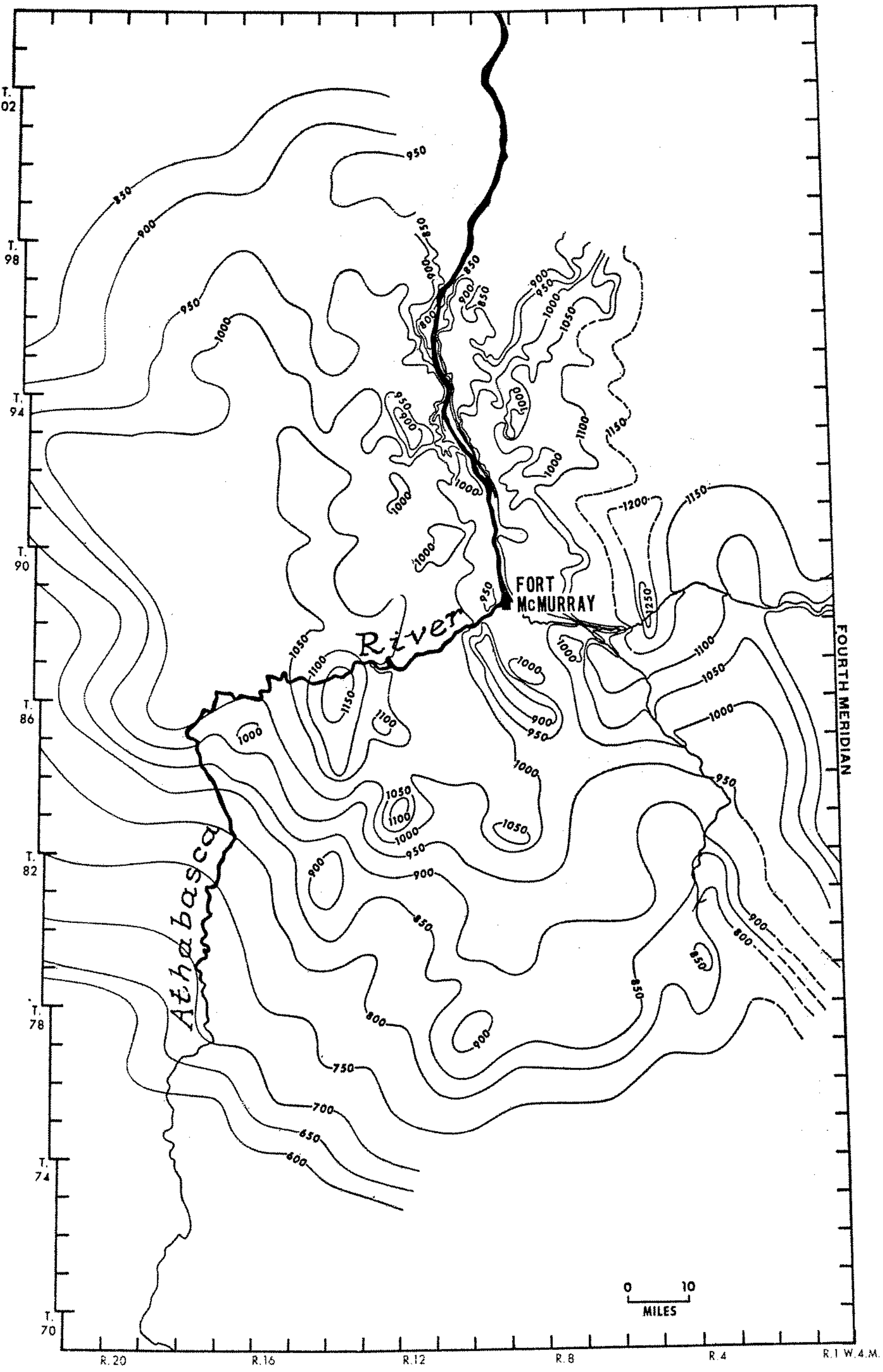

FIGURE 5. Structure contours on the upper surface of the Athabasca oil sands.

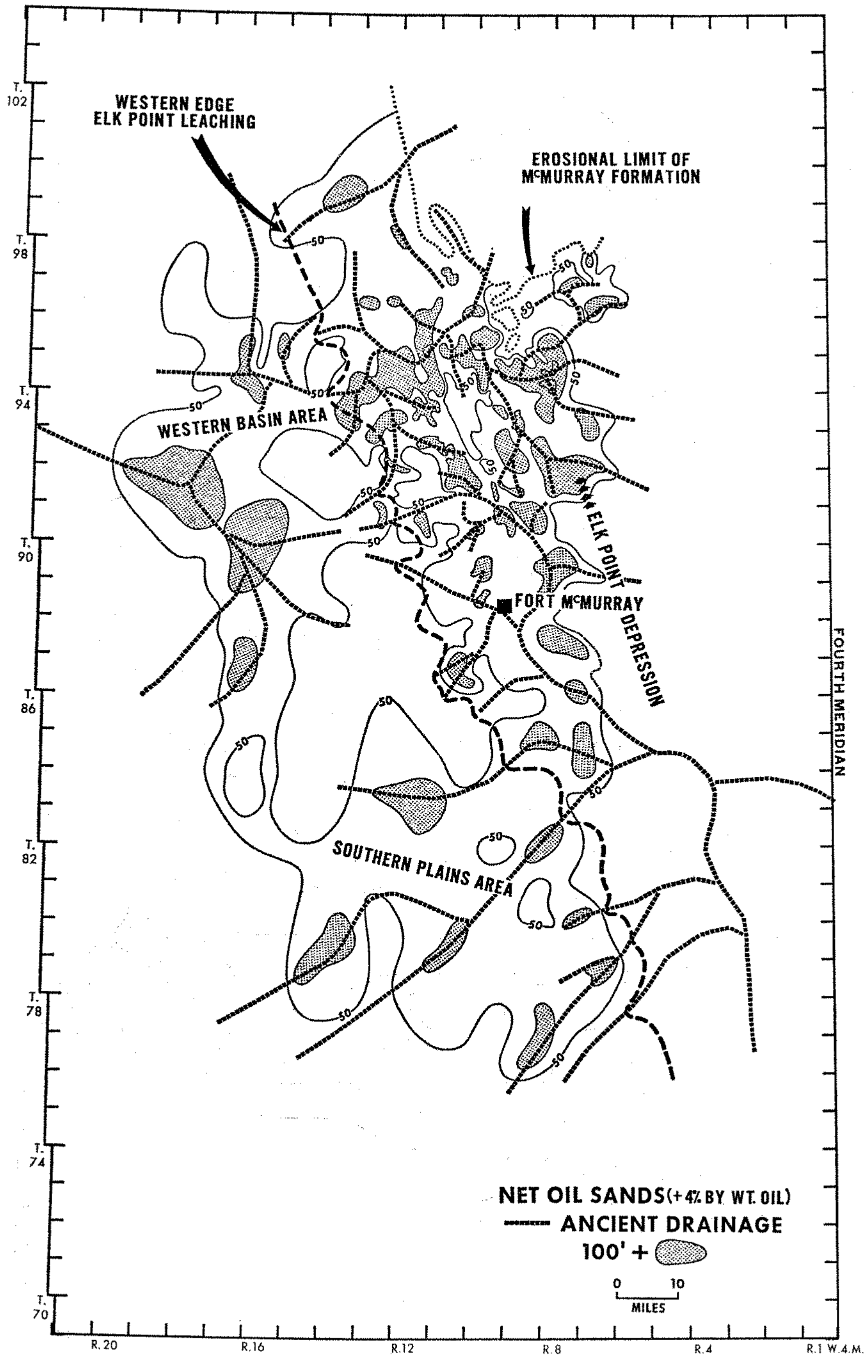

FIGURE 6. Relation of oil-sand bodies, greater than 100 feet in thickness and containing more than 4 per cent by weight of oil, to pre-Cretaceous drainage channels.

sands to the south is more poorly defined, although a definite thinning occurs in this direction, and the section grades to a more shaly sequence.

QUANTITY

The in-place reserves of the Athabasca area have been calculated at 560 billion barrels of oil. This calculation is based on data from core analyses and electrical logs from 1,570 exploratory wells in an area encompassing 13,000 square miles. An analysis of these reserves indicates the following subdivisions:

(1) Reserves in porous, reservoir sands are 440 billion barrels of oil. This category is designated as rich oil sands containing greater than 10 per cent oil by weight. These reservoir sands are comprised essentially of well-sorted, fine- to medium-sized quartz grains with an average porosity of approximately 35 per cent. "Most samples of Athabasca oil sand containing more than 10 per cent of oil by weight have median diameters within the range of those sandstone reservoir rocks from which oil has been produced in commercial quantities" by conventional methods (Carrigy, 1962, p. 312).

(2) Reserves in argillaceous sands and siltstones are 120 billion barrels of oil. This group is designated as fair and poor oil sands containing between 4 and 10 per cent oil by weight.

(3) Oil reserves in sediments containing less than 4 per cent oil by weight are considered to be insignificant. This group is characterized by silty and sandy shales.

(4) Zones within the McMurray Formation that are devoid of oil include: gas and water sands, barren shales, hard siliceous sandstones, thin coal seams, and siderite-cemented lenses.

Although approximately 80 per cent of the oil reserves are contained in the porous reservoir sands (rich oil sands containing greater than 10 per cent oil by weight), these sands comprise only 40 per cent of the total volume of McMurray sediments.

RECOVERY

Attempts to recover oil from the Athabasca oil sands can be divided into surface recovery by means of strip mining, and subsurface recovery by various *in-situ* techniques, such as burning, steam, and hot-water flooding, or by the use of nuclear devices. Current geological and economic knowledge of mining and processing Athabasca oil sands indicates that approximately 14 billion barrels of oil could be recovered by open-pit mining. This estimate is based on the following basic premises:

(1) Oil sands considered as plant feed must contain greater than 6 per cent oil by weight.

(2) Total reject consists of all oil sands with less than 6 per cent by weight of oil and of a thickness greater than 10 feet, regardless of their position in the section.

(3) The ratio of total reject to plant feed must not exceed one to one.

(4) Commercial deposits of oil sand must contain sufficient reserves to sustain a plant producing 100,000 barrels a day for a minimum of twenty years. At the present stage of development no estimate is available for recoveries to be expected from subsurface *in-situ* methods.

CONCLUSIONS

(1) The Athabasca oil sands within an area of 13,000 square miles in northern Alberta contain approximately 560 billion barrels of oil.

(2) Pre-Cretaceous topography developed on the eroded Devonian surface controls the distribution of the McMurray Formation and in part the oil contained within it.

(3) Structures resulting from leaching of Elk Point evaporites, block faulting of Precambrian rocks, and several periods of subaerial erosion contributed to the development of the pre-Cretaceous landscape.

(4) Exploratory drilling has outlined the probable extent of the McMurray oil sands. Deposits of rich oil sand are concentrated in basinal areas. In fact, 80 per cent of the oil reserves are found in only 40 per cent of the total volume of McMurray sediments.

(5) Current knowledge of mining and processing Athabasca oil sand suggests that approximately 14 billion barrels of oil could be recovered by open-pit mining.

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A NOTE ON THE COMPARISON OF McMURRAY AND MELVILLE ISLAND OIL SANDS

J. C. SPROULE* AND G. V. LLOYD*

ABSTRACT

Oil-impregnated sands were discovered in the Canadian Arctic Archipelago near Marie Bay, Melville Island, in August 1962 by G. V. Lloyd of J. C. Sproule and Associates Ltd. The areal extent of the oil sands is imperfectly known, but it now seems certain that the oil impregnation in the Bjorne Formation of Triassic age is local and controlled by structure. This oil could probably be produced from wells, if it occurs below the permafrost zone which is 1,600 feet thick.

A detailed comparison between the McMurray oil sands of Alberta and the oil sands discovered on Melville Island, N.W.T., is not practical at this time. Knowledge of the McMurray oil sands goes back to 1788, when they were first recorded by the explorer Peter Pond, whereas our knowledge of the Arctic Islands sands dates only from August, 1962, when one of the field geological parties of J. C. Sproule and Associates Ltd., under the supervision of G. V. Lloyd, encountered two widely separated outcrops of oil-soaked sands in the Triassic Bjorne Formation, near Marie Bay, Melville Island. At the time of this discovery, the weather was poor so that only a cursory investigation of the outcrop material was possible. Less than one hundred pounds of samples were taken, and plans to return later in the season were foiled by inclement weather. Field parties returned this current season (1963) to re-study the outcrops, still, however, on a reconnaissance basis. The detailed results of these later studies are not yet known, but selected preliminary data forwarded from the field are included in this paper.

Only an abbreviated comparison, containing the principal presently known related facts are presented here, since details of the Arctic Islands sands are imperfectly known and since much of the data is as yet restricted to oil-company clients. This can be taken care of most efficiently in the form of a table, Thus:

	McMurray Oil Sands	Melville Island Sands
Date of discovery	1788	August, 1962
Age of sands	Early Cretaceous	Triassic
Nature of sands	Cross-bedded, deltaic and shoreline	Cross-bedded, deltaic and shoreline
Thickness of oil impregnation	0 to 200 feet	0 to 85 feet

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*J. C. Sproule and Associates Ltd., Calgary

	McMurray Oil Sands	Melville Island Sands
Areal distribution	Greater than 10,000 square miles	Unknown
Degree of impregnation (% by volume)	0-25	0-32
Overburden (feet)	0-2,000 plus	0-2,000 plus (probably)
Stratigraphic relationships	Overlain conformably by other lower Cretaceous strata; underlain unconformably by Paleozoic rocks	Overlain conformably or disconformably by Jurassic strata; underlain unconformably by Paleozoic rocks
Structural relationships	Richer sands tend to be in synclines	Richer sands tend to be in structurally high areas or closely related to faulting
Origin of oil	Probably Paleozoic	Probably Paleozoic
Mining conditions	Relatively small area having low overburden; large area of overburden prohibitive for mining	Relatively large areas of low overburden; thick overburden areal extent unknown
	Several mining or other recovery methods believed practical	Permafrost to 1,600 feet; mining methods uncertain, probably can be produced from drilled holes below a depth of about 1,600 feet
Sulphur content (%)	4.5 (approx.)	Variable: 0.97 to 2.24
Base	Asphalt	Asphalt
Gravity (degrees API)	Less than 10	Greater than 10

Figure 1 constitutes a diagrammatic cross section showing the geological relationships of the oil sands to adjacent geological formations and inset maps showing the known distribution of the Marie Bay sands.

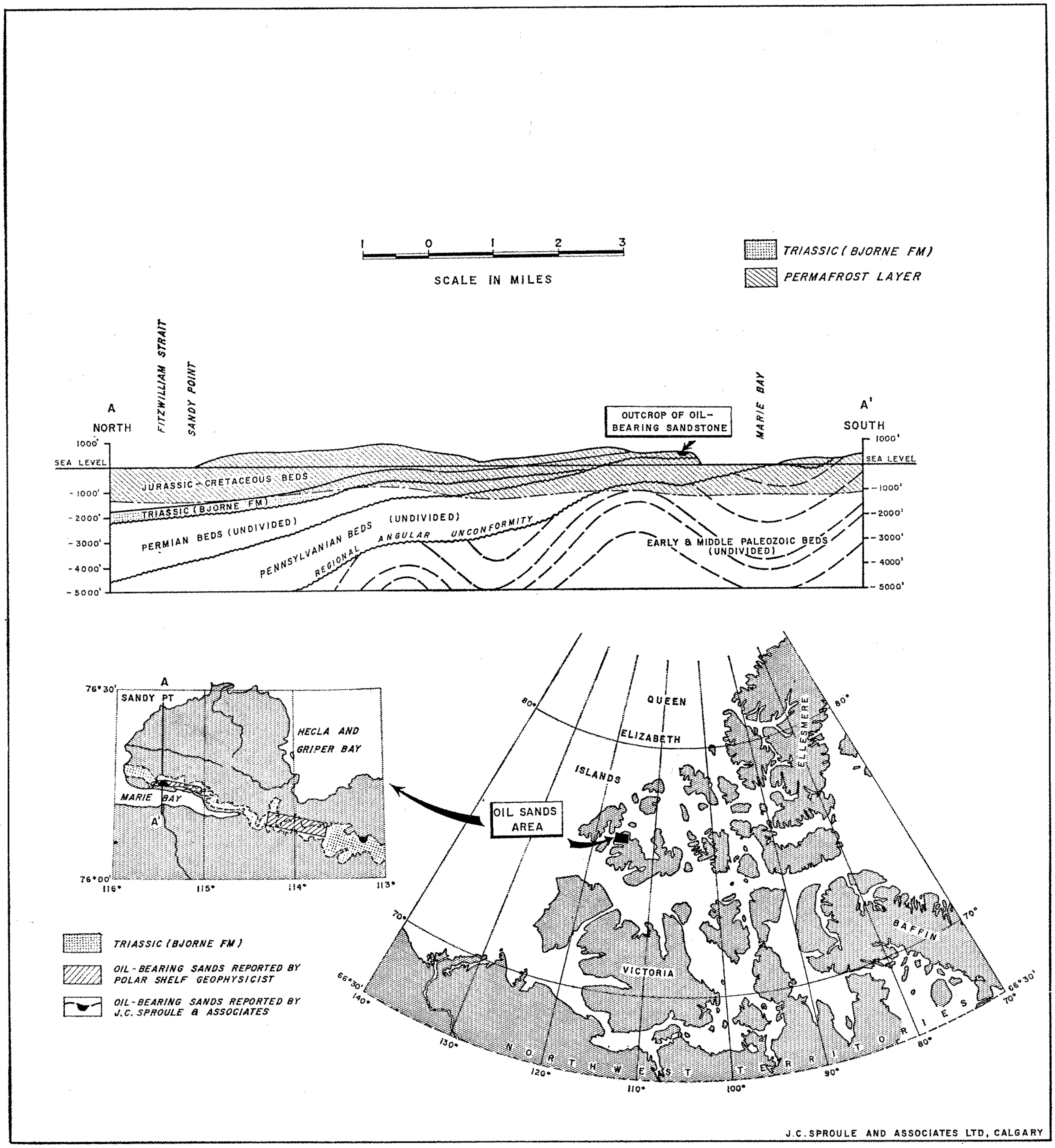

FIGURE 1. *Location of the Arctic oil sands and generalized cross section, Marie Bay, northwestern Melville Island, Northwest Territories showing the relationship of the bituminous sands to the underlying strata.*

Two analyses of oil sands have been made of the Marie Bay oil sands, one by the Testing Laboratories, Mines Branch, Department of Mines and Technical Surveys, Ottawa, and the other by research laboratories of a major oil company. Although the results were most interesting, it is considered that the details should not be generally circulated until larger, fresher samples have been analyzed and until quantities sufficient for oil-separation purposes have been tested.

With respect to the areal extent of the Marie Bay sands, our only information last year was of two isolated occurrences 60 miles apart, along an outcrop strip about 80 miles in length. A geophysicist with the Polar Continental Shelf Project has added to this, on an uncertain basis, an intermediate outcrop about 9 miles in length. Our 1963 operations have cast considerable doubt on the existence of oil sands over much of the area of the Bjorne Sandstone outcrop. Although we do not have the full details, it appears certain that much at least of the Marie Bay oil impregnation is local and controlled by structural features.

From the practical standpoint of possible oil recovery, it appears probable that oil can be produced by holes drilled to depths beneath the permafrost, which is reported to extend to about 1,600 feet in Dome *et al.* Winter Harbour well, on the south shore of Melville Island. We believe such subsurface recovery to be practical because of the relationship, along the outcrop, between the occurrence of oil sands and the structural features. Whether or not it will also become practical to mine the oil sands affected by permafrost remains to be seen.

PALEOGEOMORPHOLOGY OF THE BURIED DEVONIAN LANDSCAPE IN NORTHEASTERN ALBERTA

RUDOLF MARTIN* AND FRANK G. S. JAMIN*

ABSTRACT

The paleotopography of the Athabasca Bituminous Sands Area and adjacent areas shows a number of interesting features which are best explained in terms of paleogeomorphology. This comparatively new branch of subsurface geology deals with buried landscapes as a geomorphological problem.

The area mapped includes townships 61 to 105 between the Fourth and Fifth Meridians. The Precambrian Shield is in the northeast corner and is overlain by Middle and Upper Devonian rocks. These in turn are covered, with a marked unconformity, by the Lower Cretaceous McMurray Formation.

The dip of the Cretaceous strata is 5 to 7 feet per mile to the southwest, increasing to about 25 feet per mile beyond a hinge line in the southwestern part of the area. Beneath the Cretaceous, the Devonian strata dip at an additional 15 feet per mile to the west-southwest, so that successively younger beds subcrop below the unconformity toward the west.

A contour map of the present buried Devonian surface, not corrected for the minor amount of post-Cretaceous tilt, shows a landscape which is by no means a peneplain, but with slopes having gradients as steep as 360 feet per mile. Two major strike ridges trend south-southeast and correspond to the erosion-resistant carbonates of the Grosmont Formation and the Beaverhill Lake Group. Fragmentary ridges are formed by several other hard layers.

One of several, mostly northeast-striking faults in the area has caused a major displacement in the two main ridges and has influenced two valley trends.

Solution of salt from the Elk Point Group in the eastern part of the area has caused the collapse of the overlying Beaverhill Lake Group, resulting in an uneven topography with considerable warping of the surface and including two major closed depressions.

Two main systems of subsequent valleys drained the area northward and southward, respectively, from a *cuesta bridge* in the centre of the area. These are situated to the east of the main ridges and have many obsequent and resequent tributaries. The spacing of these valleys lends itself to interpretation in quantitative geomorphological terms, and this could lead to the discovery of additional buried valleys and hills.

INTRODUCTION

The surface of the Devonian beneath the cover of the Cretaceous McMurray Formation in the Athabasca Bituminous Sands Area has long been known to show considerable relief (Hume 1949, p. 10). It was also recognized at an early stage of the Federal Government's drilling operations in the area that the thickness of the McMurray is related inversely to the elevation of the Devonian surface: "the McMurray Formation is less thick over limestone knobs and ridges than it is over limestone valleys" (Hume 1949, p. 10). It was therefore

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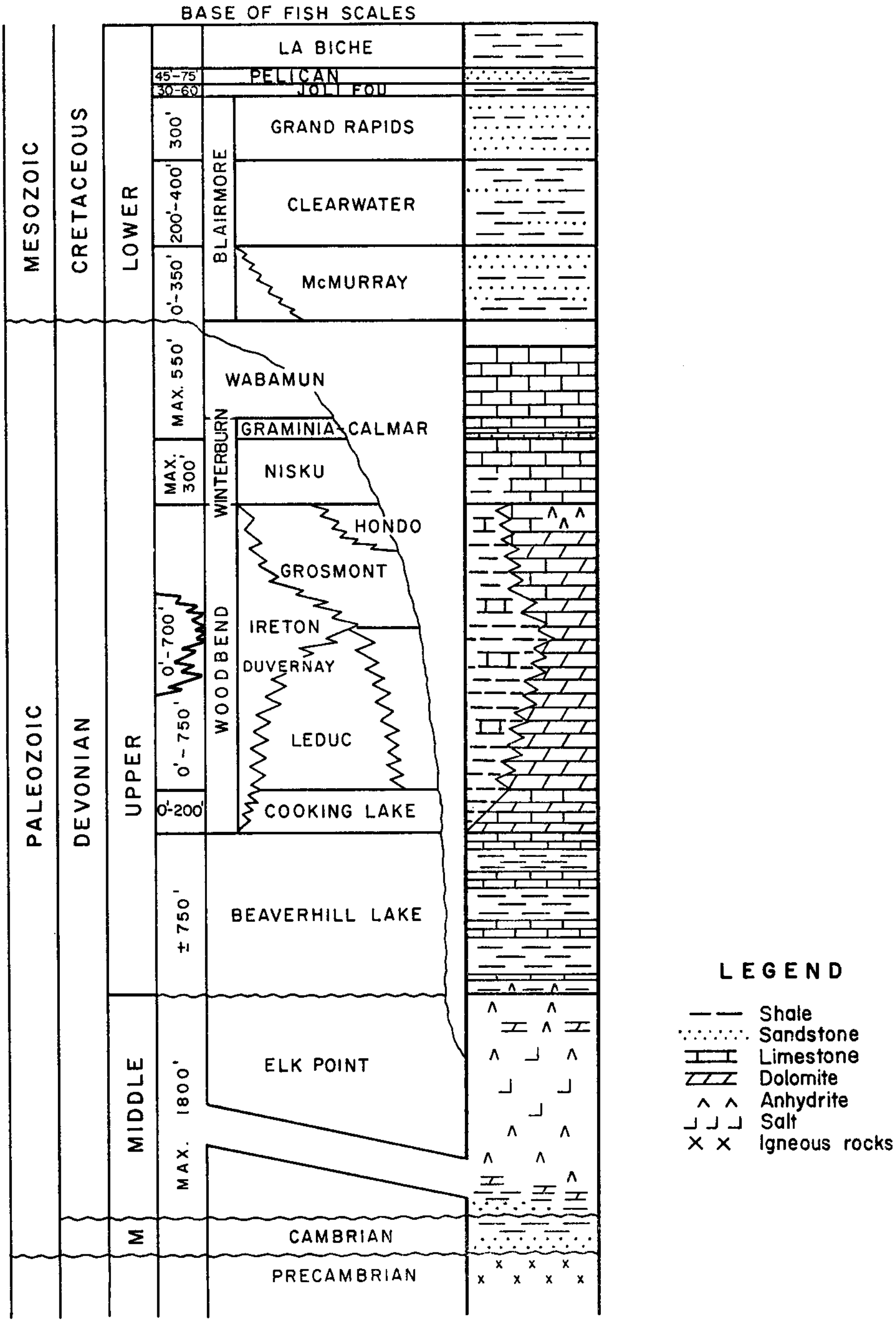

FIGURE 1. Simplified stratigraphic column in northeastern Alberta.

concluded that the McMurray had been laid down over a very irregular Devonian landscape which had been formed by pre-Cretaceous erosion. It also appeared that part of this irregularity was due to pre-Cretaceous structural movements affecting the Devonian rocks. Hume (1949), Kidd (1951), and others, concluded that both erosion and folding had taken part in shaping the pre-Cretaceous surface.

The authors have studied the paleotopography of the Devonian surface in the Athabasca Bituminous Sands Area and adjacent parts of Alberta as a problem in paleogeomorphology. This comparatively new branch of subsurface geology deals with buried landscapes such as this as a geomorphological problem (Martin, 1960). As a first step, the top of the Devonian erosional surface was established for all wells, stratigraphic tests, core holes, and other borings, in the area from townships 61 to 105, inclusive, between the Fourth and Fifth Meridians. The basic data were supplied by the Research Council of Alberta, the Oil and Gas Conservation Board, Canadian Fina Oil Limited, and others, and these were supplemented by studies of electrical and other logs combined with the available lithological data. In addition, the elevations of all outcrops of Devonian strata within the area were determined from the detailed maps published by S. C. Ells (1926).

The results of this work were embodied in a map on a scale of one inch to four miles (Martin & Associates, 1963) and in numerous working maps of local areas drafted on a considerably larger scale. This was complemented by four cross sections, drawn so as to include the majority of the wells which had reached the Precambrian or at least the Elk Point rocks in the area. In addition, a contour map of the Precambrian surface was prepared in order to establish structural relationships.

STRATIGRAPHY

The stratigraphic sequence present in the Athabasca area is summed up in figure 1. It consists of three main units: the Precambrian basement, the Devonian strata (locally underlain by Cambrian), and the Cretaceous strata. Glacial drift covers part of the area, especially in the north.

The Precambrian Shield is exposed in the northeast corner of the map area. It consists of granites and gneisses and is overlain unconformably by the Methy Formation of the Middle Devonian Elk Point Group (Tremblay, 1961). In the subsurface, the Precambrian consists predominantly of calc-alkali granites. Although Cambrian rocks are cited from several of the wells which were drilled into the Precambrian (Sherwin, 1962), the authors have made no special effort to separate them from Elk Point strata since their identification has no special bearing on the problem under consideration.

The McMurray Formation and locally younger formations of the Lower Cretaceous Blairmore Group overlie unconformably Devonian rocks, which dip about 15 feet more per mile to the west-southwest than do the Cretaceous strata. As a result, successively younger Devonian beds subcrop below the unconformity toward the west, and are succeeded by Mississippian strata outside the map area. The Devonian strata have a maximum thickness of about 4300 feet and comprise Middle Devonian clastics and evaporites and Upper Devonian shales and carbonates. The Middle Devonian Elk Point Group reaches a thickness of about 1800 feet, and the Upper Devonian rocks a maximum of approximately 2600 feet. The latter are subdivided into the Beaverhill Lake, Woodbend, Winterburn, and Wabamun Groups.

The Beaverhill Lake Group (or Waterways Formation) is about 700 feet thick and consists of alternating shales and carbonates, which are laterally very consistent in character. The Woodbend Group consists of shales and biostromal and biohermal reefs, and is subject to strong changes in facies. Carbonates of the lowermost formation of this group, the Cooking Lake, were not deposited in many parts of the area and thus vary in thickness from zero to 200 feet. This formation locally forms the platform on which Leduc Formation reefs have been built. A major Leduc reef build-up is present in the southwestern part of the area and trends just east of north. The Woodbend shale section, which comprises the Duvernay and Ireton Formations, varies in thickness from zero (where the Leduc reef continues upward into the Grosmont reef) to 750 feet. The overlying Grosmont Formation, which includes the Hondo evaporites at the top, is up to 600 feet thick. Above the Woodbend lie the Nisku carbonates of the Winterburn Group, with a maximum thickness of 300 feet. The Winterburn Group further includes the Graminia and Calmar Formations, which have been combined with the overlying Wabamun on the cross sections accompanying this study. The Wabamun Group is present only in the southwestern part of the area and has a maximum thickness of approximately 550 feet.

The Lower Cretaceous Blairmore or Mannville Group, which has been described in detail by Carrigy (1959), comprises the McMurray, Clearwater, and Grand Rapids Formations. The conglomerates, quartz sands, silts, and shales of the McMurray have a thickness of zero to 350 feet and fill the valleys of the Devonian surface. They are absent from the major ridges such as the Grosmont cuesta, and are sparse in the valleys to the west of it. Their best development is no doubt in the subcrop area of the Beaverhill Lake Group. The McMurray is overlain by the shales of the Clearwater Formation, which are 200 to 400 feet thick. A glauconitic sandstone, the Wabiskaw Member, with a maximum thickness of 20 feet, lies at the base of the Clearwater and facilitates the correlation of the top of the underlying McMurray Formation. The Grand Rapids Formation consists of "pepper-and-salt" sandstones and is more than 300 feet thick. The boundary between the Clearwater and Grand Rapids Formations is transitional in some places and is not a time line.

The Grand Rapids Formation is overlain by the Joli Fou Shale and the Pelican Sandstone. These have not been shown on the cross sections because of their moderate thickness. The Joli Fou Shale is 30 to 60 feet thick, the Pelican Sandstone 45 to 75 feet. The Pelican, which is the equivalent of the Viking Sandstone, is in turn overlain by a shale section locally known as the La Biche Formation.

STRUCTURE

REGIONAL TILTING TO THE SOUTHWEST

Tilting of the order of 15 feet per mile took place prior to the deposition of the Cretaceous sediments. Post-Cretaceous tilting was about 5 to 7 feet per mile throughout most of the map area, but was close to zero in the northeastern portion. In the southwest corner, however, it increases to about 25 feet per mile beyond a hinge-line which runs from about Tp. 73, R. 26 to about Tp. 61, R. 14, W. 4th Mer. The present dip of the pre-Cretaceous formations is therefore 20 to 25 feet per mile within most of the area, and 40 feet per mile in the southwest.

LOCAL WARPING

Kidd (1951, p. 33) described the Devonian as, "undulat[ing] markedly on a relatively small scale, but . . . not folded in the sense that the structures have any discernible and consistent strike . . . The rocks are warped into small quaquaversal domes with flank dips ranging from 1° to a maximum of 20° or more in rare instances. . . . The breadth of the domes vary from a few hundred feet to half a mile or more. The amplitudes of the domes or undulations also vary but they apparently range up to approximately 250 feet." Kidd (p. 33) goes on to say, "erosional truncation of the undulating structure in the Devonian rocks took place in pre-McMurray time." Hume (1949, p. 10), however, cites "some evidence, although by no means conclusive, to support the contention that there may be some local post-Cretaceous folding" in the Mildred-Ruth Lakes area. Some post-Cretaceous downwarping is evident from the cross sections, as in Tp. 82, R. 4, W. 4th Mer. (section D-D′ Fig. 4). There is a general impresion of structural lowering affecting the northeastern portion of all cross sections. It is evident from the sections that this goes hand in hand with a tremendous thinning of the evaporite section of the Elk Point Group, caused by the gradual removal of the salt beds by solution in the proximity of the outcrop. Most of the resulting subsidence evidently took place in pre-Cretaceous times, but the effect appears to have continued to the present.

FAULTING

Any faulting observed in the McMurray Formation probably constitutes a rejuvenation of pre-existing, deeper-seated faults. Sproule (1938) and Kidd (1951) have described a fault affecting the McMurray which crosses the Clearwater River in Tp. 89, R. 3, W. 4th Mer. The strike of this fault is not given by either author, and the present writers have not succeeded in tracing it on the buried Devonian surface. Many additional faults affect only the Paleozoic and the basement, and most of these appear to strike northeast. One of these has had a greater effect on the pre-Cretaceous landscape than all the others and is mentioned in the next section.

PALEOTOPOGRAPHY

Probably the best way to gain insight into the nature of the relief of the buried Devonian surface would be to construct an isopach map of the overlying basal Cretaceous McMurray Formation. Such a map, its thickness values slightly expanded to take care of post-McMurray compaction, would provide a cast of the Devonian surface as it was immediately prior to the deposition of the Cretaceous. This would have the advantage of automatically eliminating the influence of all post-McMurray structural movements. Unfortunately, however, the uppermost McMurray strata have been removed by erosion all along the Athabasca and Clearwater valleys and along most of their tributaries within the map area. They have furthermore been removed by (pre-)Pleistocene erosion throughout much of the northern part of the area. Finally, the depositional character of the McMurray sands makes it difficult to establish a marker at its top which can be considered a time line, except for the Wabiskaw glauconitic sandstone. An isopach map of the McMurray Formation could therefore not be constructed without prior reconstruction of its top and detailed correlation work, and the assumptions inherent in such a reconstruction and correlation would eliminate many of the advantages of the isopach map.

Since most of the area has been subject to only minor post-Cretaceous tilt, with the exception of the southwest corner, the authors decided to map the Devonian surface by means of contours simply showing its present attitude (Fig. 2). The nature of the pre-Cretaceous relief which is immediately evident from this map is in sharp contrast to the concepts of W. M. Davis and others, which are still being cited in several textbooks on geomorphology today. According to these concepts, an area such as this, having been exposed to subaerial erosion for a period of 170 million years or so, embracing at least the Pennsylvanian, Permian, Triassic and Jurassic Periods, should have been worn down to a smooth peneplain with slopes measured in inches instead of in feet per mile. Instead, this buried landscape is highly dissected with slopes of as much as 360 feet per mile in many

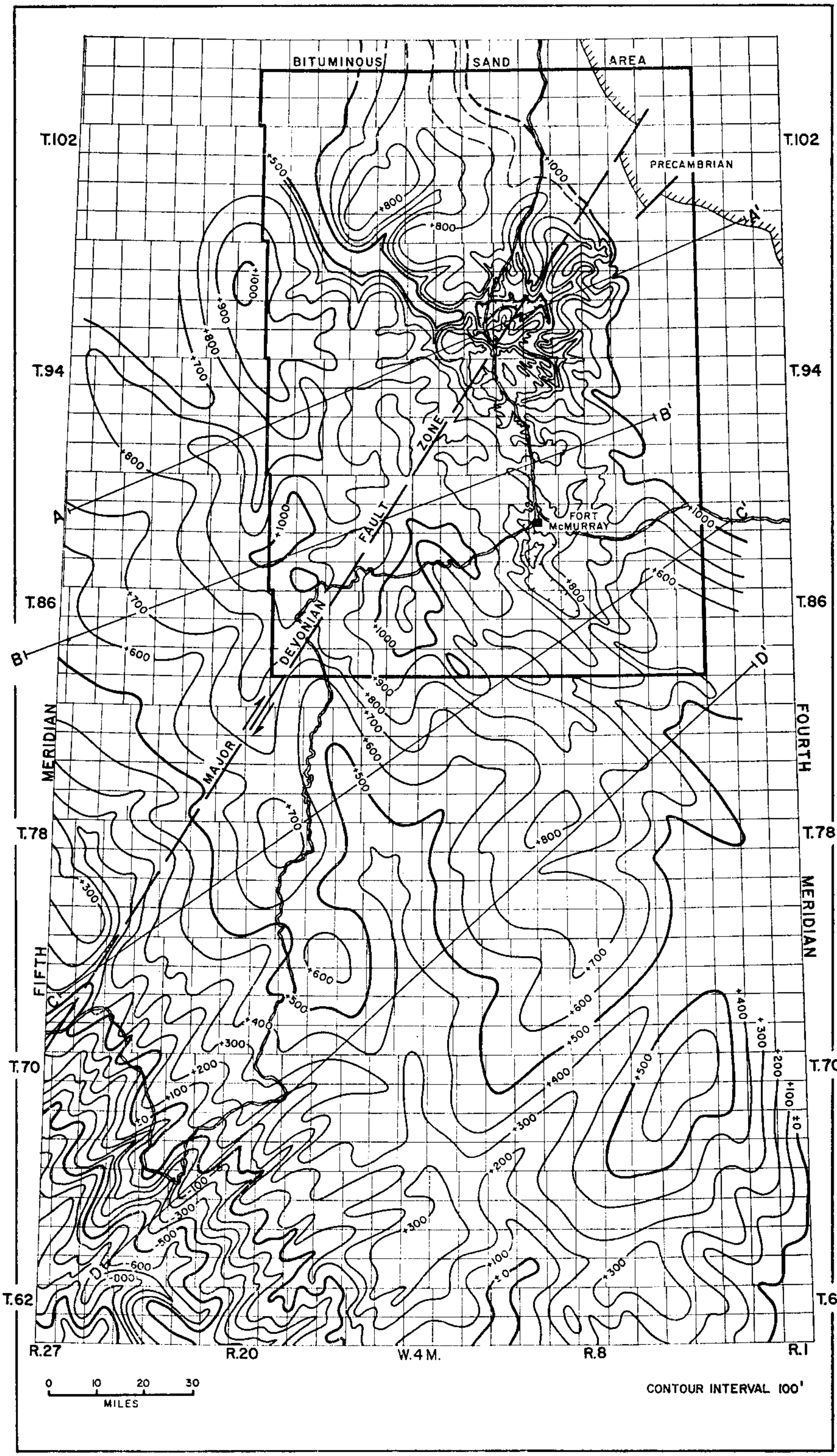

FIGURE 2. *Contour map of the erosional surface of the Devonian rocks in northeastern Alberta.*

places, specially in the area of Tps. 94 to 97, Rs. 9 to 11, W. 4th Mer. Most lesser slopes on the map are probably steeper in reality, but lack sufficiently dense subsurface control. These observations, and those made by the first writer (Martin, 1960) and others on buried landscapes elsewhere, lend support to the theory of W. Penck and of many modern geomorphologists. According to this concept, a slope retains its original angle while responding to the forces of erosion by gradually retreating parallel to itself. The theory of slope retreat is adequate to explain the buried Devonian landscape in the Athabasca Bituminous Sands Area in geomorphological terms.

The map (Fig. 2) shows that the pre-Cretaceous landscape included two major strike ridges, formed by the erosion-resistant carbonates of the Grosmont and Beaverhill Lake units. Both trend in a south-southeasterly direction across the map area. They are interrupted and offset by a major fault which strikes north-northeast and has displaced the northern part of both ridges toward the east. This fault also cuts off a major scarp formed by the Wabamun Group in the Marten Hills area.

Fragmentary ridges corresponding to other erosion-resistant beds are more readily recognized on the cross sections (Figs. 3, 4) than on the map. Most of those which correspond to the Wabamun, Nisku, and Hondo strata form subsidiary scarps on the west flank of the Grosmont ridge, where they lose their identity as a result of the increasing superimposed southwestward tilt near the hinge line. The three escarpments in Wabamun Group strata are interrupted by valleys trending normal to their strike direction. The same can be said for the single Nisku escarpment. The Hondo evaporites form a local scarp on cross section B-B′ (Fig. 3) which has not been traced to other cross sections.

All of these escarpments correspond to the subcrop edges of erosion-resistant beds where they reached their maximum height in the now buried pre-Cretaceous landscape. The highest scarp appears to have been that of the Grosmont which is a true cuesta parallel to the contemporary basin rim. Locally, a secondary escarpment below the Grosmont corresponds to two carbonate beds below its base (Fig. 3), but as a rule east of the Grosmont cuesta is an extensive lowland corresponding to the shales of the Ireton and Duvernay Formations of the Woodbend Group. On the east side of this lowland the Cooking Lake carbonates form a small escarpment, which locally still supports some Woodbend shale. Reefs in the Cooking Lake Formation may result in two escarpments instead of one (Fig. 3).

The Beaverhill Lake Group ridge, in contrast to that of the Grosmont, is not an escarpment or cuesta, but corresponds primarily to an irregular fold, also parallel to the basin rim, which resulted from the downwarping of the Beaverhill Lake strata toward the east due to the removal of salt from the underlying Elk Point Group by solution. This is most evident from the cross sections (Figs. 3, 4), which indicate a stepwise thinning of the Elk Point toward the outcrop. The Beaverhill Lake Group is warped over the successive steps of the collapsing Elk Point. It would be wrong, however, to conclude that the Devonian erosion surface in the eastern part of the map area coincides with the top of the Beaverhill Lake Group. For one thing, the Beaverhill Lake strata do form three escarpments, which correspond to different resistant limestone beds; on the map, these cannot readily be distinguished from the steps caused by the Elk Point collapse, but they are recognizable on the cross sections.

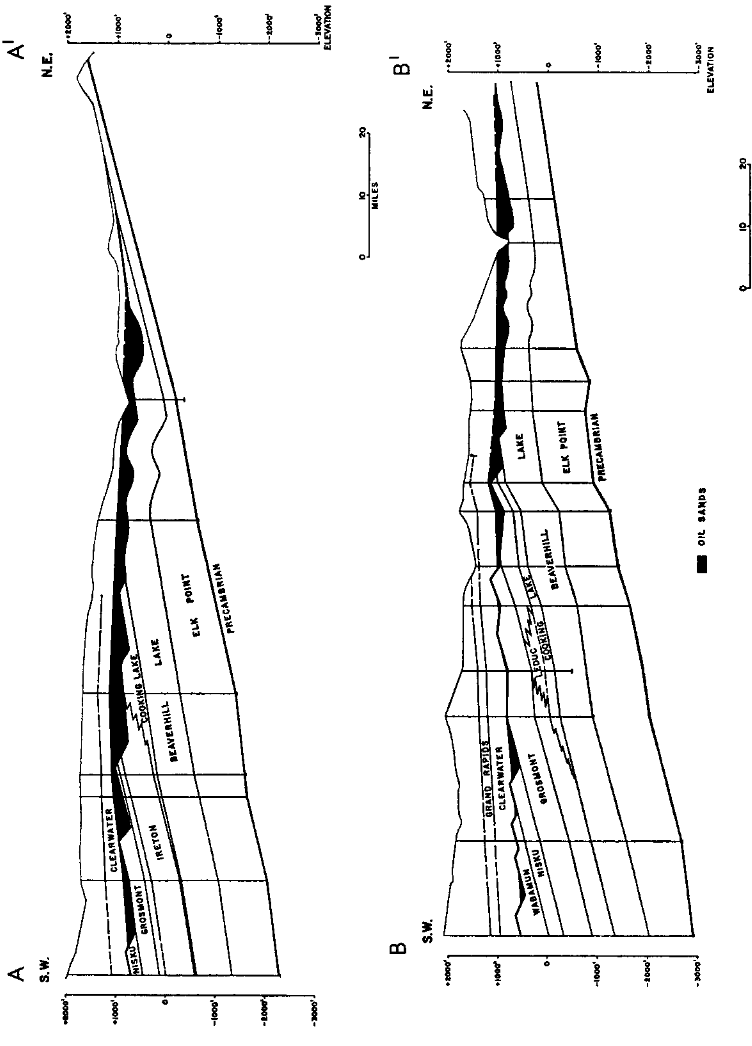

FIGURE 3. *Geological cross sections A-A′ and B-B′ showing the relation of the McMurray oil sands to the paleotopography.*

In the second place, running water, which does, after all, shape most landscapes, will flow down the slopes of erosion-resistant layers only where these serve its purpose locally. Elsewhere, hydrophysical laws dictate the formation of additional valleys independent of the underlying geology (Horton, 1945). Two major systems of subsequent rivers, parallel to the two major ridges, drained the area north and southward, respectively, from a cuesta bridge in townships 89 to 91. One system followed the Woodbend shale lowland, which was mentioned earlier, and the other followed the major collapse valley to the east of the Beaverhill Lake Group ridge. This corresponds in part to the "general synclinal depression" of Kidd (1951, p. 37), which

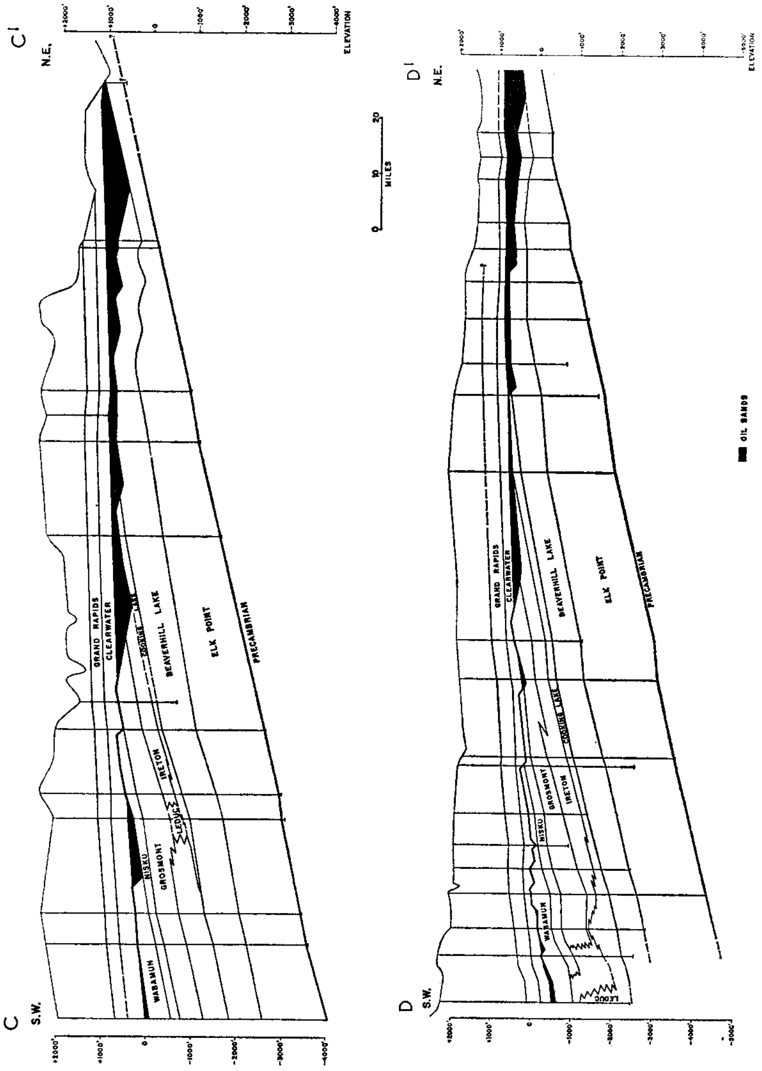

FIGURE 4. *Geological cross sections C-C' and D-D' showing the relation of the McMurray oil sands to the paleotopography.*

"parallels the Athabasca [River] for several miles north of McMurray." Into these strike valleys flowed obsequent streams, which cut the east slopes of the two ridges, and resequent streams, which flowed down their respective dip slopes to the southwest. The obsequent valleys are narrow and appear to be spaced an average of eight miles apart on the east side of the Beaverhill Lake Group ridge, as shown by the area of densest subsurface control. The resequent valleys, on the other hand, appear to be considerably wider and average about 29 miles apart, except near the hinge line in the southwest, where their spacing also averages 8 miles. The ratio of subsequent spacing to obsequent-resequent spacing, which is equivalent to Horton's (1945) stream-length ratio, is 44 to 8 miles, or 5.5, which compares with a ratio of 4.9 for the Mississippian buried landscape of southeastern Saskatchewan. By applying Horton's principles of quantitative geomorphology, the approximate position of the major consequent streams which drain the map area may be established. They should be spaced about 5.5 times 44 miles apart; they may thus be expected to be about half that distance, or 120 miles north and south, respectively, of the cuesta bridge mentioned earlier. The northern consequent valley would thus lie in the northwestern corner of the map area (Fig. 2), whereas the southern one would correspond to the deepest valley in that part of the area between Lac La Biche (Tp. 67, R. 13) and Cold Lake (Tp. 64, R. 1).

There is some evidence that certain portions of the Elk Point collapse area formed closed paleotopographical depressions, especially around Fort McMurray and Bitumount. The Oil and Gas Conservation Board map (1963) shows three such depressions south of Fort McMurray, which the authors have combined into one on their map. Kidd (1951, p. 37) described the McMurray Formation as being "sharply folded" into a "pronounced depression, the Bitumount Basin." It is possible that an isopach map of the McMurray strata of these two areas, if it could be constructed, would show that these depressions were not closed in McMurray time, but developed later as a result of continued subsidence. Instead of having been deposited in "irregular, sometimes interconnecting depressions" (Kidd, 1951, p. 33), the McMurray sands appear to have been laid down in flowing water, which would tend to indicate the existence of a properly connected drainage system at that time.

Several valley trends, specially those with very steep sides, suggest the influence of faults or fracture systems on the pre-Cretaceous topography. A northeasterly trend appears to be most frequent among valleys suspected of belonging to this type. The major northeast-trending cross-fault which was mentioned earlier appears to have been instrumental in the formation of at least two valleys which seem to deviate from the general trend.

The foregoing constitutes but a brief introduction to what could become a major new approach to the prediction of the presence and location of additional Cretaceous sand-filled valleys and Devonian buried hills in this area.

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PETROLOGY OF COARSE-GRAINED SANDS IN THE LOWER PART OF THE McMURRAY FORMATION*

M. A. CARRIGY**

ABSTRACT

Coarse-grained sands and gravels at the base of the oil-impregnated McMurray Formation are river-channel deposits, which have significantly different proportions of nonopaque heavy minerals from the overlying finer-grained sands, but appear to be equally favourable petroleum reservoir rocks.

INTRODUCTION

The variability of the grain size of the sediments of the McMurray Formation was recognized 70 years ago by McConnell during the course of a geological survey of the Lower Athabasca River area. He wrote (McConnell, 1893, p. 32), "The tar sands must have consisted originally of almost unconsolidated sands and soft sandstone, ranging in texture from a fine silt to a coarse grit. . . ." As outcrops of the coarser-grained sediment are not common, the significance of this coarse sand facies within the McMurray Formation was not recognized. Hume (1947) believed the coarse-grained sands and conglomerates in the Mildred-Ruth Lakes oil-sand deposit to indicate a near-shore environment of deposition. Carrigy (1959) showed that there are well-defined but irregularly shaped bodies of coarse-grained sand in the McMurray Formation, and that mechanical analyses of these sediments yield characteristic particle-size distribution curves. He also mapped the distribution of these coarse-grained sands in the Fort MacKay area and showed that they were related to the topography at the unconformity with the underlying Paleozoic limestones. In general, he found the coarse-grained sands (where present) to be at the base of the formation, the fine-grained, well-sorted sands to be in the middle, and the fine-grained sands and silts to be in the upper part of the formation.

To determine whether the observed textural differences in the McMurray Formation sediments involved concomitant changes in mineral composition, nine coarse-grained sand samples, spaced as widely as possible and covering as great a stratigraphic interval as permissible, were collected and analysed. In addition, the data on the Mildred-Ruth Lakes deposit contained in volume 2 of Canada Mines Branch Report 826 were re-analysed with two objectives in mind: (1) to draw an isopach map of the coarse-grained lithofacies and (2) to determine whether the degree of oil impregnation within these bodies differed from that of the overlying finer-grained oil sands.

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*Contribution No. 220

**Research Council of Alberta, Edmonton

DESCRIPTIONS OF SAMPLES

The sampling locations are given in table 1. One outcrop sample and eight samples from four wells were analysed. All nine samples are oil impregnated and are from the McMurray Formation.

TABLE 1. LOCATIONS OF NINE COARSE-GRAINED SANDS FROM THE MCMURRAY FORMATION

Sample Number	Location, west of the 4th Meridian			Depth below surface	Remarks
	Sec.	Tp.	R.	(in feet)	
1	NW ¼, 31	92	9	—	Pebble conglomerate: base of outcrop on Steepbank River
2	NE ¼, 27	92	10	157-158	Great Canadian Oil
3	NE ¼, 27	92	10	160- 61	Sands Ltd., Hole E.
4	SE ¼, 14	93	10	128-134	Canada Cities Service Petroleum Corporation Well 2-14-93-10
5	34	93	10	89- 95	Canada Cities Service
6	34	93	10	97-105	Petroleum Corporation
7	34	93	10	105-107	Well 4-34-93-10
8	2	97	11	255-260	Richfield Oil Corporation
9	2	97	11	305-310	Well 16-2

PARTICLE SIZE

The nine samples are examples of coarse-grained sediments which fall within the Class I group of Carrigy (1959). Such sediments are defined by having a maximum grain-size larger than 1.0 mm, median diameters greater than 0.13 mm, and more than 80 per cent of the sample by weight larger than 0.074 mm.

Histograms of the particle-size distributions of these samples are shown in figure 1. The modal size varies from the very coarse sand grade (1-2 mm) in sample 3 to the fine-sand grade (¼-⅛ mm) in samples 1 and 4. Both samples 1 and 4 have minor secondary modes in the medium-sand grade size (½-1 mm), but it is probable that these small secondary modes are due to sampling deficiencies and have no genetic significance. Sample 1 is from an oil-impregnated pebble conglomerate at the base of a cliff on the Steepbank River in Tp. 92, R. 9, and sample 4 is from a well in Tp. 93, R. 10.

MINERAL COMPOSITION

The mineral fractions of the nine oil-sand samples were separated into light and heavy portions by use of a heavy liquid (S.G., 2.95), and grain mounts for the microscopic examination of each portion were prepared. The frequency percentages of the mineral constituents reported in tables 2 and 3 are based on line counts of the minerals in the grain mounts examined under the petrographic microscope.

Light Minerals (S.G.<2.95)

Quartz grains are the most abundant constituent in the nine samples examined, forming between 84.5 and 99.5 per cent of the total number of grains. Most quartzose grains are single crystals, but a few polycrystalline grains were observed in most samples. The grains show signs of abrasion, but few are well rounded or angular. Grains with authigenic quartz overgrowths are also common.

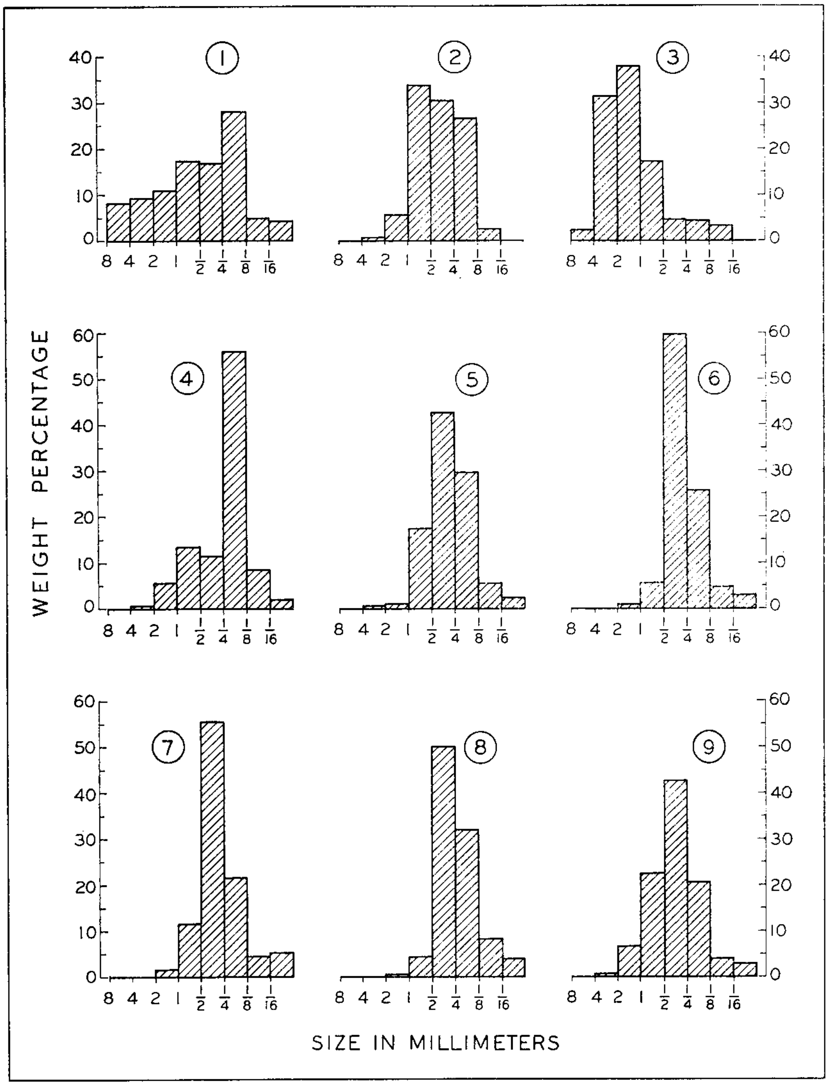

FIGURE 1. Histograms showing particle-size distributions of nine samples of coarse-grained sand from the McMurray Formation.

Potash feldspar grains are not abundant, ranging in frequency from zero to 3.5 per cent of the total number of grains. Such grains are usually in the form of angular cleavage fragments, and many grains have serrated edges suggestive of corrosion. Some of the grains exhibit a slight degree of alteration.

TABLE 2. FREQUENCY PERCENTAGES OF LIGHT MINERALS IN NINE COARSE-GRAINED SANDS FROM THE McMURRAY FORMATION

	Frequency percentages of mineral grains			
Sample Number	Quartz	Potash feldspar	Chert	Others*
1	91.0	0.5	0.5	8.0
2	84.5	—	—	13.0
3	99.0	0.5	0.5	—
4	99.5	—	0.5	—
5	91.5	3.5	2.5	2.5
6	98.0	0.5	—	1.5
7	95.5	—	0.5	4.0
8	95.5	2.5	1.0	1.0
9	98.0	0.5	0.5	1.0

*Mainly comminuted carbon and quartz grains with an opaque coating of iron oxides.

Cryptocrystalline chert grains are a ubiquitous constituent of these sediments, but in only one sample do they exceed one per cent of the total number of grains.

Heavy Minerals (S.G.>2.95)

The nonopaque heavy minerals of the McMurray Formation were described by Mellon (1956). Although the heavy mineral assemblage in the coarse-grained sediments is qualitatively the same as that described by Mellon, the relative proportions of the minerals appear to be significantly different (Table 3) from Mellon's assemblages, which were from predominantly fine-grained sediments. For example, tourmaline, which forms more than 50 per cent of the total number of grains in 14* of the oil-sand samples described by Mellon, forms only 24 per cent of the nonopaque grains in the coarse sediment; and

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*Sample No. 16 of Mellon (1956) was excluded from the computation of averages in table 3 because it is probably a coarse-grained sample from the same outcrop as sample 1 of this report. The relative proportions of nonopaque heavy minerals in this sample would not be representative because of the exclusion by Mellon of all grains retained on the 100 mesh sieve during the preparation of his grain mounts. This procedure is believed to have not effected the results in the other 14 samples described by Mellon.

Table 3. Frequency Percentages of Nonopaque Heavy Mineral Grains in Nine Coarse-grained Sands from the McMurray Formation

Mineral	Sample Number										
	1	2**	3**	4	5	6	7	8	9	Average	Average of Class II Sands* N=14
Garnet	63	32	13	23	24	27	19	22	6	25	6
Staurolite	4	18	10	9	15	18	18	9	21	14	10
Kyanite	7	22	11	23	15	33	29	26	47	24	5
	74	**72**	**34**	**55**	**54**	**78**	**66**	**57**	**74**	**63**	**21**
Tourmaline	9	14	14	38	37	15	30	41	22	24	53
Zircon	16	12	41	—	5	3	1	1	3	9	8
Chloritoid	—	1	9	1	2	2	1	1	—	2	15
Rutile	—	—	1	—	—	—	—	—	—	—	1
Apatite	—	—	—	—	—	—	—	—	—	—	1
Others	1	1	1	6	2	2	2	—	1	2	1
TOTAL	100	100	100	100	100	100	100	100	100	100	100

*After Mellon (1956).

**Insoluble residues; siderite, which was the most abundant constituent, has been dissolved in hot HCl (see p. 48).

chloritoid, which averaged 15 per cent in Mellon's samples forms only 2 per cent in the average coarse-grained sand, being absent from two of the nine samples examined.

The most notable difference in the heavy mineral frequencies, however, is in the abundance of pink garnet grains: Mellon found them to be extremely rare in his samples, whereas they are very conspicuous in all of the coarse-grained samples examined by the writer. The pink garnets are especially large in sample 1, eight out of ten grains exceeding 0.15 mm in size, with a maximum size of 0.8 mm. The proportion of large kyanite grains is also greater in the coarse-grained samples, and, although the proportions of high-grade metamorphic minerals vary from sample to sample, the combined percentages of garnet, kyanite, and staurolite are significantly greater in the coarser-grained samples than in the other McMurray sands.

In contrast, in the coarse-grained sediments zircon grains appear to maintain a uniform size regardless of the size of the other heavy minerals, and in sample 3 abundant small zircon grains are found along with large pink garnet grains.

In samples 2 and 3, from the Mildred-Ruth Lakes area, heavy minerals form 6.4 per cent and 15.8 per cent, respectively, of the total weight of the mineral matter. This unexpectedly large quantity of heavy minerals in these samples was found to be due to an abundance of small spherulites of siderite ($FeCO_3$). The optical identification of the mineral composition of the spherulites is confirmed by X-ray diffraction powder patterns. Examination of the allogenic nonopaque heavy mineral content of these samples was possible only after the siderite had been dissolved in acid.

The siderite is in the form of amber grains with a spore-like appearance. They have dark dumbell-shaped nuclei with two to three concentric growth rings at each end. Under crossed nicols they show a radiating, fibrous, spherulitic texture, with dark extinction crosses paralleled to the directions of vibration of the light. As siderite-cemented sands and siderite nodules are common throughout the McMurray Formation, the presence of siderite spherulites in these samples is not surprising. Nevertheless, the abundance of these grains in the coarse-grained sands in the Mildred-Ruth Lakes deposit was not previously suspected.

The form of the spherulites as discrete grains and cemented aggregates suggests that they are of secondary origin. The time relations between their formation and the oil impregnation of these sediments is not known.

MILDRED-RUTH LAKES DEPOSIT

A large body of coarse-grained, oil-impregnated sediment was outlined in the lower part of the McMurray Formation by the drilling operations of the Federal Government in the Mildred-Ruth Lakes area (Tp. 92, R. 10) between the years 1942 and 1947 (Fig. 2). Beneath this area reserves of 900 million barrels of oil are present with a ratio of oil sand to overburden of 2.6 to 1. Thus, an understanding of the genesis of this deposit could be of considerable value in the search for other "ore bodies" in the oil sands. Therefore, it seemed worthwhile to re-examine the data contained in volume 2 of Canada Mines Branch Report 826 to see if they were amenable to classification by the scheme proposed by Carrigy (1959).

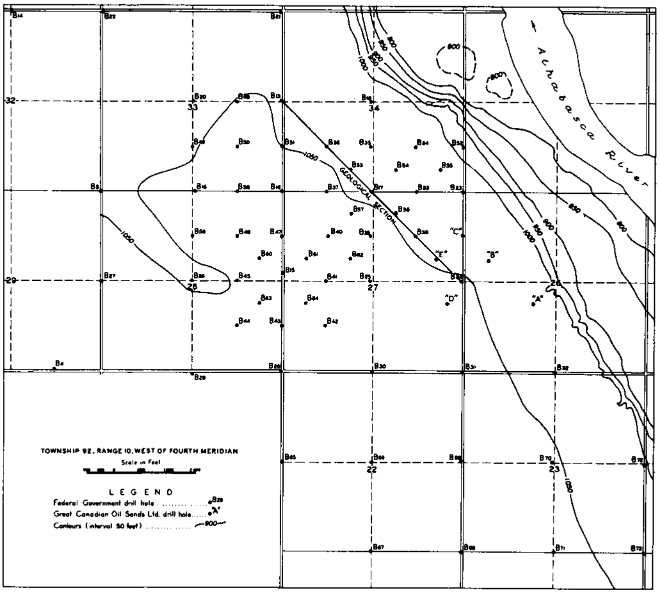

FIGURE 2. Locations of wells drilled in the Mildred-Ruth Lakes area.

When the 660 cumulative curves prepared from the results of the sieve analyses reported in volume 2 of Report 826 are grouped according to the scheme of classification proposed by Carrigy (1959), 300 samples fall into Class I, 230 samples fall into Class II, and 130 samples into Class III (Fig. 6). Stratigraphically, the coarse Class I are ovelain by the fine-grained Class II sands (Fig. 5). The thickness of the Class I sands in this area varies from 20 to 100 feet (Fig. 4), and the form of the isopachytes seems to be controlled by the presence of a northeasterly trending depression in the Paleozoic limestone (Fig. 3).

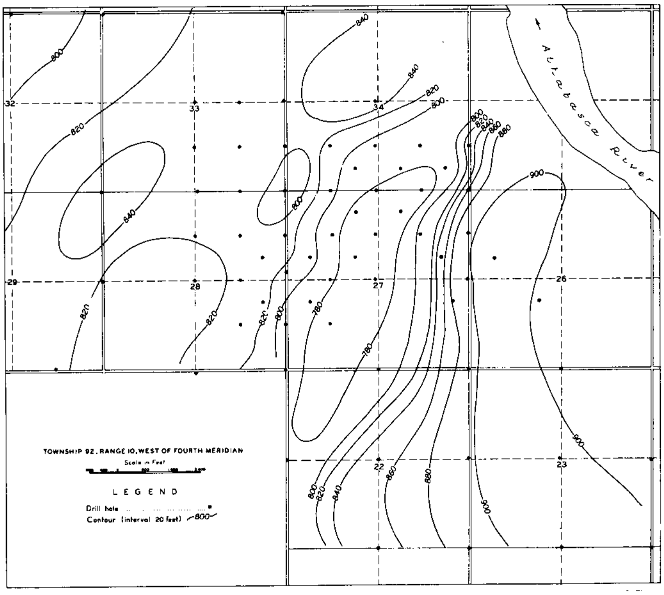

FIGURE 3. *Topography on the Paleozoic limestone in the Mildred-Ruth Lakes area.*

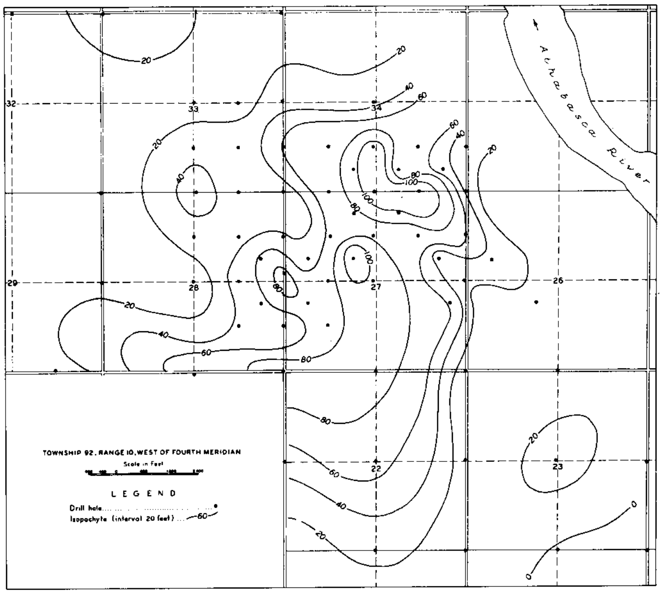

FIGURE 4. *Distribution of coarse-grained sediment in the Mildred-Ruth Lakes area.*

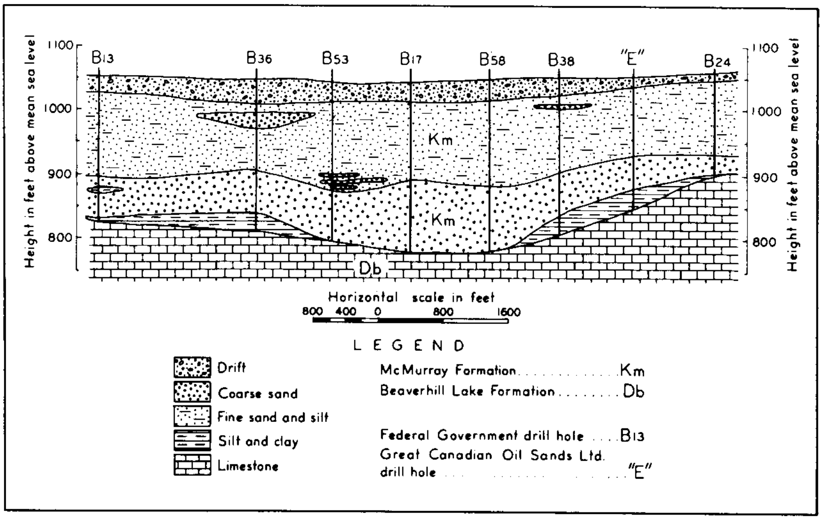

FIGURE 5. Geological cross section of the Mildred-Ruth Lakes deposit (See figure 2 for location of section).

DISTRIBUTION OF OIL

Frequency histograms have been prepared of the oil contents within each sediment class (Fig. 6). The coarse-grained Class I sands show a large range of values with a mean oil content of 12.47 per cent by weight. The fine-grained Class II sands in the same deposit have a mean oil content of 14 per cent and have a smaller range of values. The very fine grained sands and silts of Class III have a mean of 5.0 per cent and have a wide range of oil contents. Carrigy (1962) has recently examined the relationship between oil content and texture of the sediments in the McMurray Formation and has shown that the oil content of a sample can be predicted with a reasonable degree of precision if the median diameter and clay content of the sediment are known. The clay content was found to be the most important property influencing the oil content of a sediment, and thus the results reported here are compatible with the previous conclusion that the median diameter has only a minor effect on the amount of oil a sediment can hold.

ORIGIN OF THE COARSE-GRAINED LITHOFACIES

The particle-size distribution data (Fig. 1) show that most of the samples are not well sorted, the modal class commonly containing less than 50 per cent by weight of the total sample. Pettijohn (1957, p. 284) divides sands into three degrees of sorting based on the number of Wentworth grade sizes covered by the particle-size distribution; well-sorted sands are contained in less than 3 grades, fair sorting is indicated when the number of grades is from 4 to 6, and poorly sorted sands cover more than 7 grade sizes. On this basis most of the sands shown on figure 1 are poorly sorted.

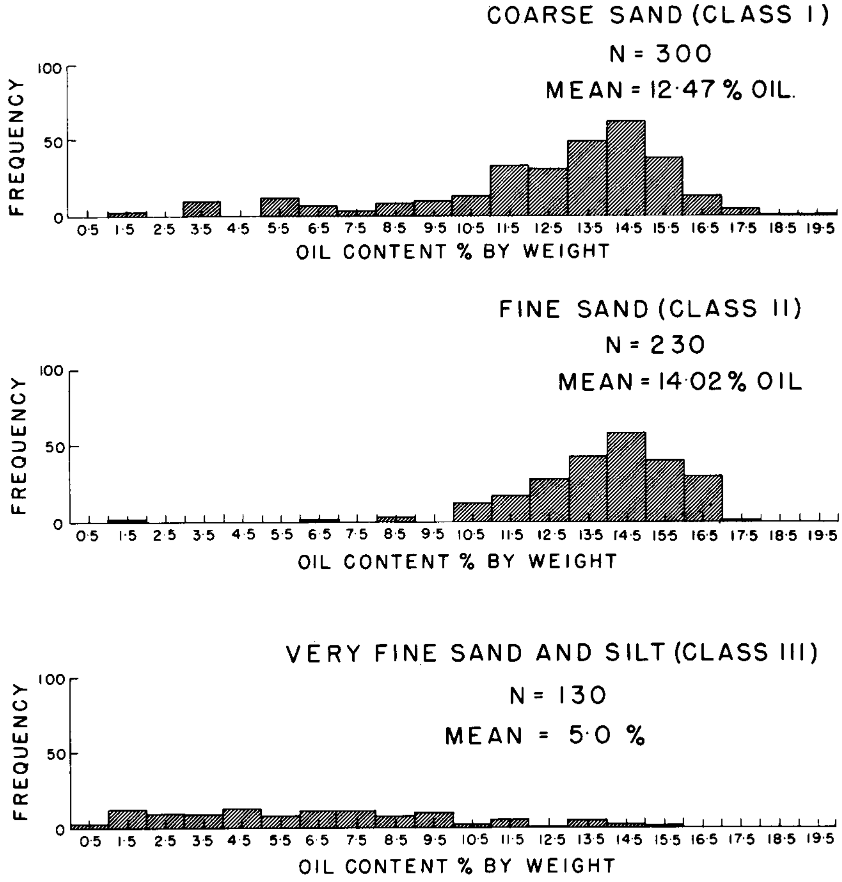

FIGURE 6. Frequency histograms showing the distribution of oil in three types of sand in the McMurray Formation.

Pettijohn (1957) also points out that both beach sands and beach gravels are well-sorted sediments. In beach gravels the modal class commonly contains 50 to 60 per cent and sometimes up to 90 per cent of the total weight of the sample. The poor sorting of the nine samples of coarse-grained sand and gravel from the McMurray Formation suggests that these sediments were deposited in a fluvial rather than a shoreline environment. Additional evidence as to the mode of deposition, deduced from the location and shape of the coarse-sand bodies, similarly points to a fluvial origin. For example, the cross section of the Mildred-Ruth Lakes deposit (Fig. 5) and the linear trend of the isopachytes parallel to the contours of the depression in the Devonian limestone (Figs. 3 and 4) all accord with the hypothesis of a fluvial environment of deposition.

The results of the mineralogical analyses (Tables 2 and 3) are of little value in determining the environmental conditions of deposition of a sediment, but can, however, be of considerable use as indicators of the source rocks.

The light-mineral fractions of the sand-size grades in both the fine-grained and coarse-grained sands are similar. In the conglomerate outcrop on the Steepbank River some pebbles of an orthoquartzite similar in aspect to the Athabasca Sandstone of northern Saskatchewan are found and may have been derived from this source.

The suite of nonopaque heavy minerals present in the coarse-grained lithofacies is similar to that found in the fine-grained sand lithofacies by Mellon (1956), who suggested a source area on a highly metamorphosed terrain probably on the adjacent Precambrian Shield. Although conglomerates in the coarse-grained lithofacies of the McMurray Formation contain orthoquartzite pebbles probably derived from the Athabasca Formation, the wide variety of heavy minerals present is in direct contrast to the impoverished suite in the Athabasca Formation, wherein tourmaline and zircon are the only nonopaque heavy minerals present in significant amounts (Fahrig, 1961). Thus, it must be concluded that the source rocks for the McMurray Formation conglomerates also included some metamorphic rocks in addition to orthoquartzitic material. In this respect the coarse-grained sands and conglomerates within the McMurray Formation were derived from a source area similar to that supplying the formation as a whole. The differences in the relative proportions of the nonopaque heavy minerals in the coarse- and fine-grained McMurray sands can be readily explained by sorting during sediment transportation.

CONCLUSIONS

(1) The coarse-grained sands and gravels at the base of the McMurray Formation are river-channel deposits derived from an area of the Precambrian Shield formerly covered in part by the Athabasca Formation and underlain by highly metamorphosed sediments.

(2) The variations in the proportions of the nonopaque allogenic heavy minerals between the fine-sand and coarse-sand facies in the McMurray Formation can be attributed to the availability of a wide range of sizes of heavy minerals in the source rocks and to their subsequent sorting during transportation to the area of deposition.

(3) The two types of clean sand, the well-sorted, fine-grained sands of Class II and the coarse-grained channel sands and pebble conglomerates of Class I, appear to be equally favourable petroleum reservoir rocks.

ACKNOWLEDGMENTS

The writer is indebted to Dr. T. P. Clarke of Great Canadian Oil Sands Ltd. for permission to publish some of the data obtained from their 1959 drilling operations on Lease 4.

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CONTINUITY OF BEDDING WITHIN THE McMURRAY FORMATION

Ralph W. Ansley* and W. G. Bierlmeier*

ABSTRACT

The data gathered from closely spaced drill-holes in the McMurray Formation on Lease 17 indicate that there is extreme variation laterally, and difficulty is experienced in tracing thin beds even at 200-foot centres. However, fairly massive beds can be traced purely on bitumen concentration and classified as good plant feed, marginal feed, or reject material for a selective mining operation.

INTRODUCTION

The lithology of the McMurray Formation is very variable. It has been stated (Clark and Blair, 1927, p. 72), "There is no system to the succession of rich and lean bituminous sand beds in sections through the sand formation. The formation is composed of a haphazard assortment of lenses of variable material. There is a general tendency, however, for the upper beds to be very changeable in nature and composed of lean material, and for the thick, massive, rich bituminous sand beds to occur toward the bottom of the formation." Although this type of information is generally sufficient for descriptive purposes, more detailed knowledge of the bedding is required before any individual deposit can be exploited economically. In an engineering appraisal of a deposit as prospective feed for an extraction plant, the value of a bed is assessed essentially on its bitumen content. If selective mining were employed, all those beds below a predetermined minimum value may be classified as undesirable feed and discarded. The purpose of the drilling and coring program described herein is to give some indication of how these poorly impregnated beds could be distributed, both areally and stratigraphically. A futher purpose was to determine the hole spacing required to plan an efficient selective-mining operation in areas where interbedding is prevalent.

This study began with the examination of an outcrop at the site of the pilot plant operated by Cities Service Athabasca, Inc., Imperial Oil Company Limited, Richfield Oil Corporation, and Royalite Oil Company Limited, at Mildred Lake, approximately 25 miles north of Fort McMurray (Fig. 1). Detailed examination of the outcrop showed that the McMurray Formation contained, in addition to the bituminous sands, thin interbeds of clay and silt, as well as lenses of barren siltstone. Figure 2 shows the stratified nature of the McMurray Formation on a face of the outcrop at Mildred Lake exposed by blasting. The thin, light beds running horizontally across the photograph are oil-barren silts and clays. Figure 3 shows the discontinuous

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*Cities Service Athabasca Inc., Edmonton

nature of thin clay beds. Some beds of a few inches in thickness can be traced for more than 100 feet on the escarpment but, as shown in the photograph, other beds cannot be traced even a few feet. Immediately below the pencil in figure 4 is an extremely hard, argillaceous siltstone lens. This lens is only 3 or 4 feet in lateral extent, but on the same outcrop a slightly thicker lens can be traced for approximately 600 feet.

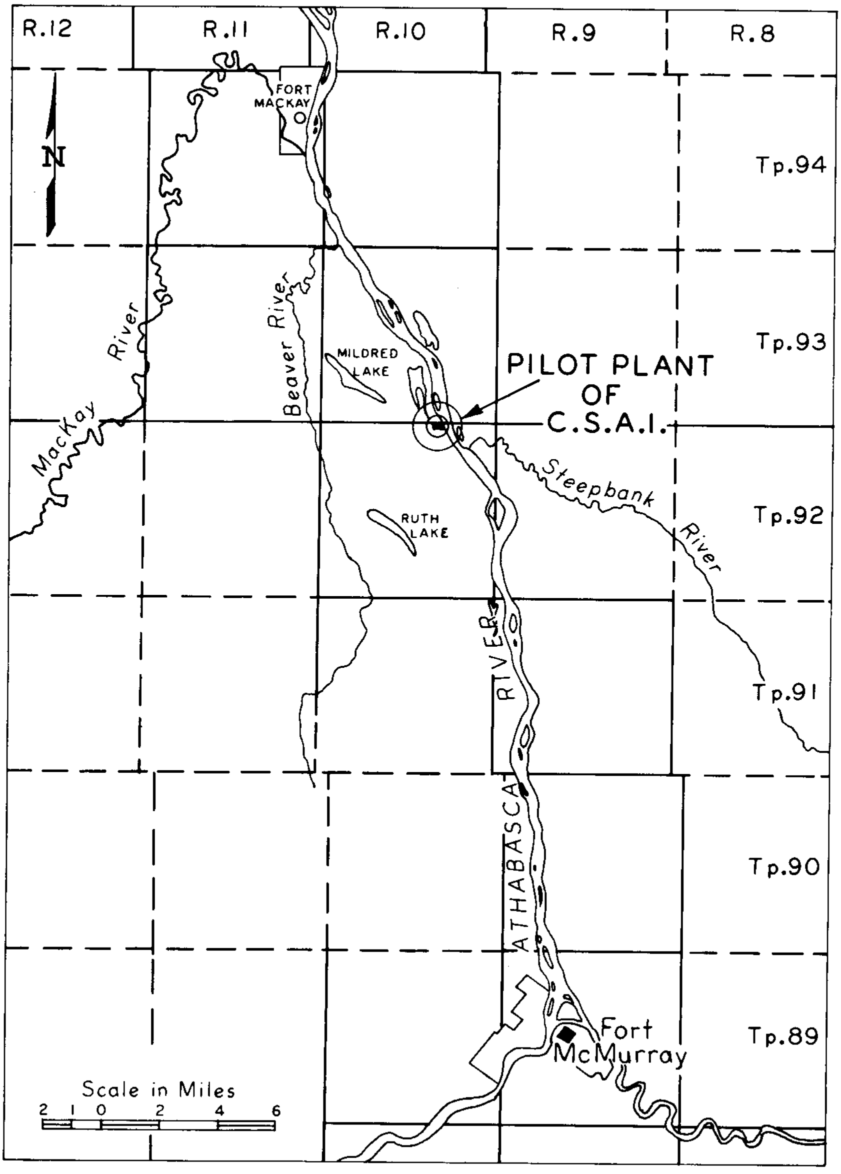

FIGURE 1. Location of pilot plant operated by Cities Service Athabasca Inc., Imperial Oil Company Limited, Richfield Oil Corporation, and Royalite Oil Company Limited.

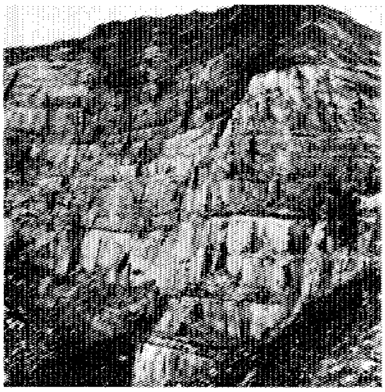

FIGURE 2. McMurray Formation: Oil-sand face exposed by blasting at Mildred Lake. This face is approximately 70 feet high.

FIGURE 3. Close-up of oil-sand face showing thin barren silt beds.

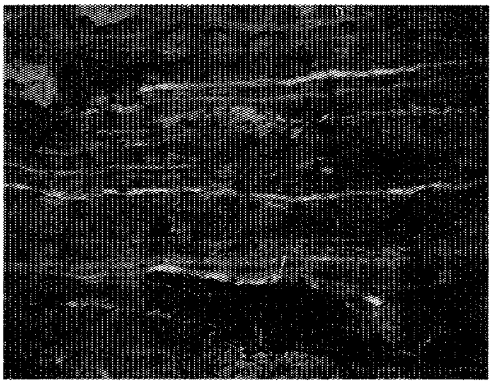

FIGURE 4. Close-up of oil-sand face showing the irregular nature of the thin silt beds. Below the pencil is a hard rock-like siltstone bed 3 to 4 feet in width.

Although the outcrop contains minor silt and clay partings, as shown in the photographs, it is essentially an oil sand of high bitumen saturation. Core holes drilled approximately one-quarter mile west of the escarpment shown in the photographs revealed interbedding of clays and silts with oil-sand lenses such that the average bitumen saturation was low over thicknesses of 10 to 50 feet.

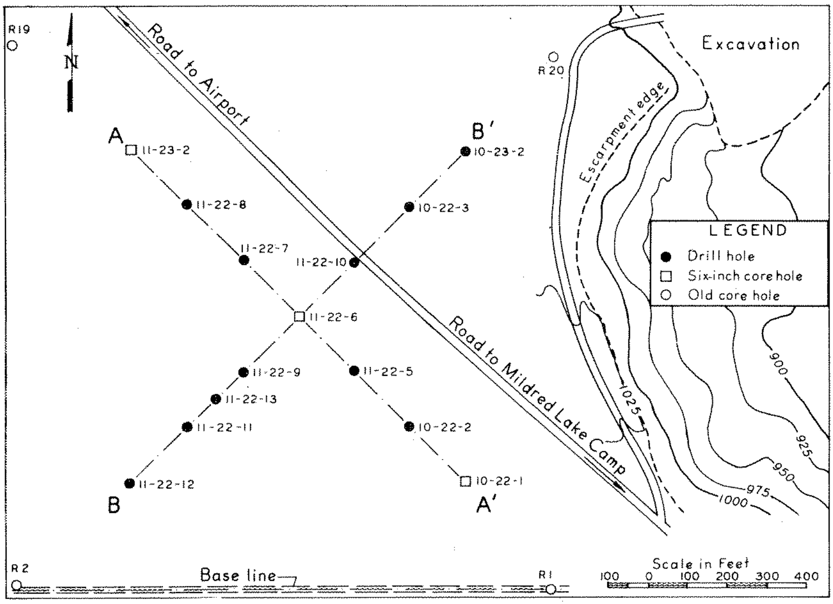

FIGURE 5. Location of drill and core holes of 1963 test drilling program.

It was found that, while processing the bituminous sand through the pilot plant at Mildred Lake, significant differences in processibility could be found on bituminous sands from different locations along the escarpment, although these different locations were all within a one-mile length of the outcrop. On-site digging with a mining wheel, which was elaborately instrumented for research purposes, indicated that oil sand exhibits quite variable digging characteristics.

DRILL-HOLE DATA

More than 300 holes have been drilled on Lease 17 to evaluate the bitumen reserves. Many of these holes have been drilled and cored with large-diameter equipment to provide feedstock to the pilot plant so that the processibility of the material remote from the escarpment locations could be tested. In summary, the data from these holes concur with the conclusions of Clark and Blair (1927) in that there is a succession of rich and lean sand beds in the McMurray Formation with interbedded barren clay and silt. These partings are more pronounced at the top and a more massive section richer in bitumen is present at the bottom of the formation.

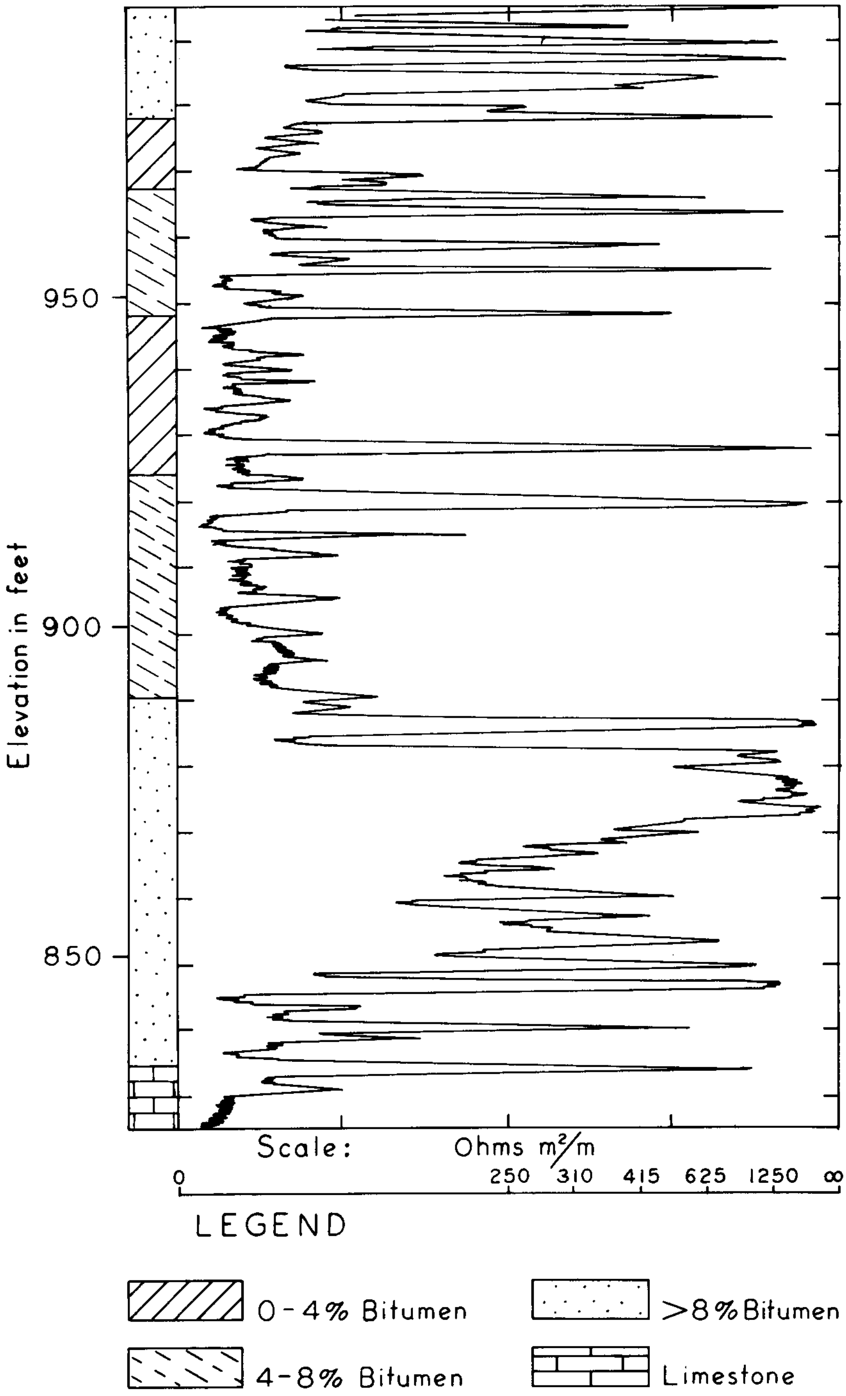

FIGURE 6. Graphic log and laterolog of hole 11-22-10 showing effect of bitumen saturation on resistivity.

An examination of the data taken from the holes indicated that substantial thicknesses of the McMurray Formation contained less than 6 per cent bitumen by weight. Studies to date have indicated that this is the economical lower limit of bitumen saturation of mined material which should be fed to the extraction plant (Ansley, 1963). If the material falls below this limit, it is presently considered advantageous to discard it with the overburden and sand tailings without charging it to the extraction unit. If this material were at the *top* of the McMurray section, it could be stripped with the overburden, and if at the *bottom* of the section, it could be left in place; but if such material were encountered *within* the McMurray Formation with feed material above and below it, the material would have to be either fed to the commercial plant or equipment provided to discard it. An examination of the holes which had been drilled on a pattern of quarter-mile spacing indicated that some thick lean bands could be correlated reasonably well between the holes but that others could not be traced from well to well. It was, therefore, decided to drill holes in the pattern of a cross on 200-foot centres (Fig. 5) to determine the continuity of the lenticular beds of the McMurray Formation. A location was chosen so that the centre of the cross would be 1,000 feet west of the outcrop, as the incidence of interbedding is considerably higher in this area than on the remainder of the proposed mining area of Lease 17. Three of the holes, 11-23-2, 11-22-6 and 10-22-1, were cored and the remainder were drilled. Electrical logs were run in all holes.

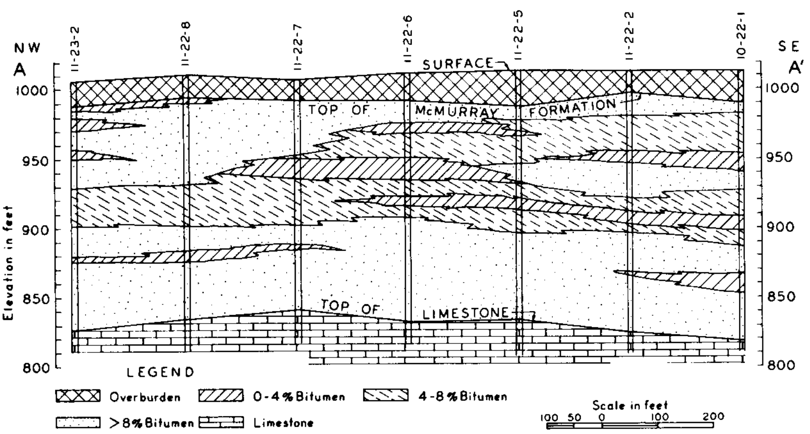

FIGURE 7. Northwest to southeast section of the McMurray Formation showing variations in bitumen content between holes 11-23-2 and 10-22-1.

A plot of one of the logs taken on the holes can be seen on figure 6. This plot is a trace of the laterolog with resistivity increasing to the right from zero on the left-hand side. As can be seen in this plot, the lithological breaks are well- defined by the laterolog. In general, the resistivity gives a fairly good idea of the bitumen saturation, with high resistivities indicating high bitumen saturations. The laterolog together with other logs has been used to develop a correlation with samples analysed from cored holes. The massive rich section at the bottom, as readily seen on this log, extends from elevation 835 feet to elevation 890 feet. The cross-hatching shown on the left-hand side of figure 6 classifies the oil sand by average bitumen concentration, as explained on figures 7 and 8.

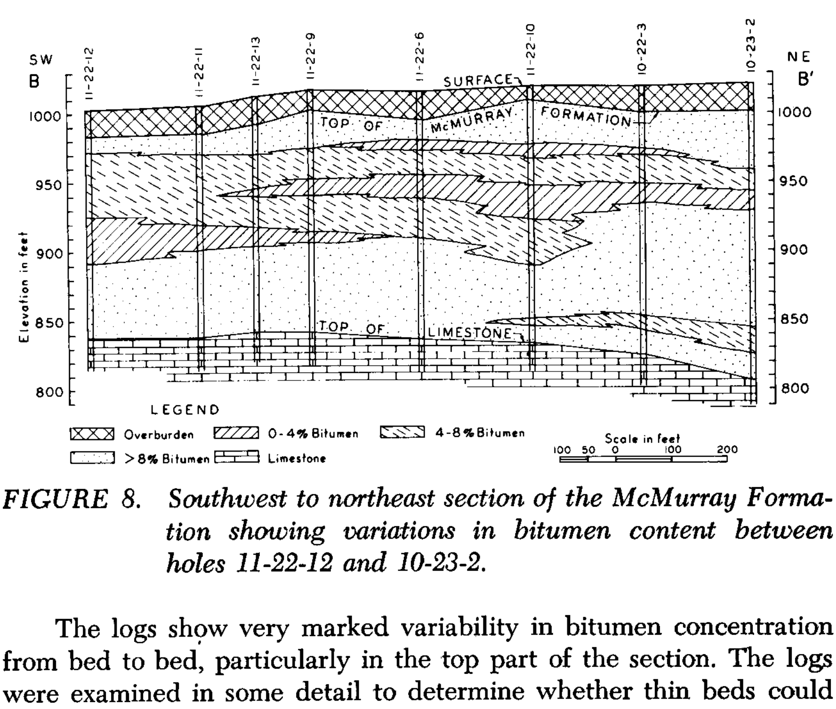

FIGURE 8. Southwest to northeast section of the McMurray Formation showing variations in bitumen content between holes 11-22-12 and 10-23-2.

The logs show very marked variability in bitumen concentration from bed to bed, particularly in the top part of the section. The logs were examined in some detail to determine whether thin beds could be traced from hole to hole. Even on 200-foot centres, most individual beds could not be traced. However, the McMurray Formation has been classified by bitumen saturation into three categories: 0 to 4 per cent bitumen, 4 to 8 per cent bitumen, and greater than 8 per cent bitumen. Figures 7 and 8 show two sections, each drawn through one arm of the cross. With these classes, it is possible to trace some bands for more than 500 feet.

SUMMARY

In general, the McMurray Formation in the area investigated with closely spaced drill-holes can be divided into three parts. The top portion consists of thinly bedded clays, silts, and sands, averaging fair to good feed for a commercial plant; the centre portion consists of interbedded clays, silts, and sands, and could be considered undesirable or marginal feed; the basal portion consists of massive sands with few silt and clay beds and is good-to-rich feed.

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COMPOSITION AND MOVEMENT OF FORMATION FLUIDS IN STRATA ABOVE AND BELOW THE PRE-CRETACEOUS UNCONFORMITY IN RELATION TO THE ATHABASCA OIL SANDS*

BRIAN HITCHON**

ABSTRACT

A high degree of correlation exists between the composition of formation fluids from strata above and below the pre-Cretaceous unconformity in Alberta. Potentiometric-surface maps indicate that there is free fluid connection across the unconformity and that formation-fluid movement in proximity to the unconformity is in a northeasterly direction and in whichever set of strata offers the least resistance to movement, i.e., those having the highest permeability. Study of the relationship of the environmental factor in the control of crude oil composition indicates that the position of the lower Cretaceous oil-sand deposits on the broad continental shelf which occupied practically all of Alberta in early Cretaceous time is reflected in the asphaltic nature of the crude oils. All of these factors are consistent with a major Cretaceous source for the oil in the Athabasca oil sands with a possible additional source in the pre-Cretaceous rocks.

INTRODUCTION

The Athabasca oil sands consist of the heavy-oil impregnated sands and silts of early Cretaceous age which unconformably overlie Paleozoic strata in northeastern Alberta. Hypotheses postulating an external source for the heavy oil fall into two categories: firstly there are those that favour a source in the Mesozoic strata above the pre-Cretaceous unconformity (McLearn, 1918; Hume, 1951) and secondly those that favour a source in the Paleozoic strata below the unconformity (Sproule, 1938, 1951; Williams, 1949; Link, 1951; Gussow, 1956). No information has been published comparing the composition of formation fluids in strata above and below the unconformity. If the compositions of formation fluids in strata above and below the unconformity are dissimilar, then the unconformity probably acted as a barrier to fluid flow. This implies a source for the heavy oil in the Athabasca oil sands from the Mesozoic strata. If, however, the compositions are similar, there are several possible sources. With good fluid connection across the unconformity there has been one source in either Cretaceous or Paleozoic rocks or several sources with mixing of the fluids in proximity to the unconformity. With poor fluid connection across the unconformity the similarity in composition is difficult to account for except by maturation. It seemed reasonable therefore to study the data available on the composition and flow of fluids in the strata above and below the unconformity in an attempt to determine the most likely source(s) of the heavy oil in the Athabasca oil sands.

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*Contribution No. 224

**Research Council of Alberta, Edmonton

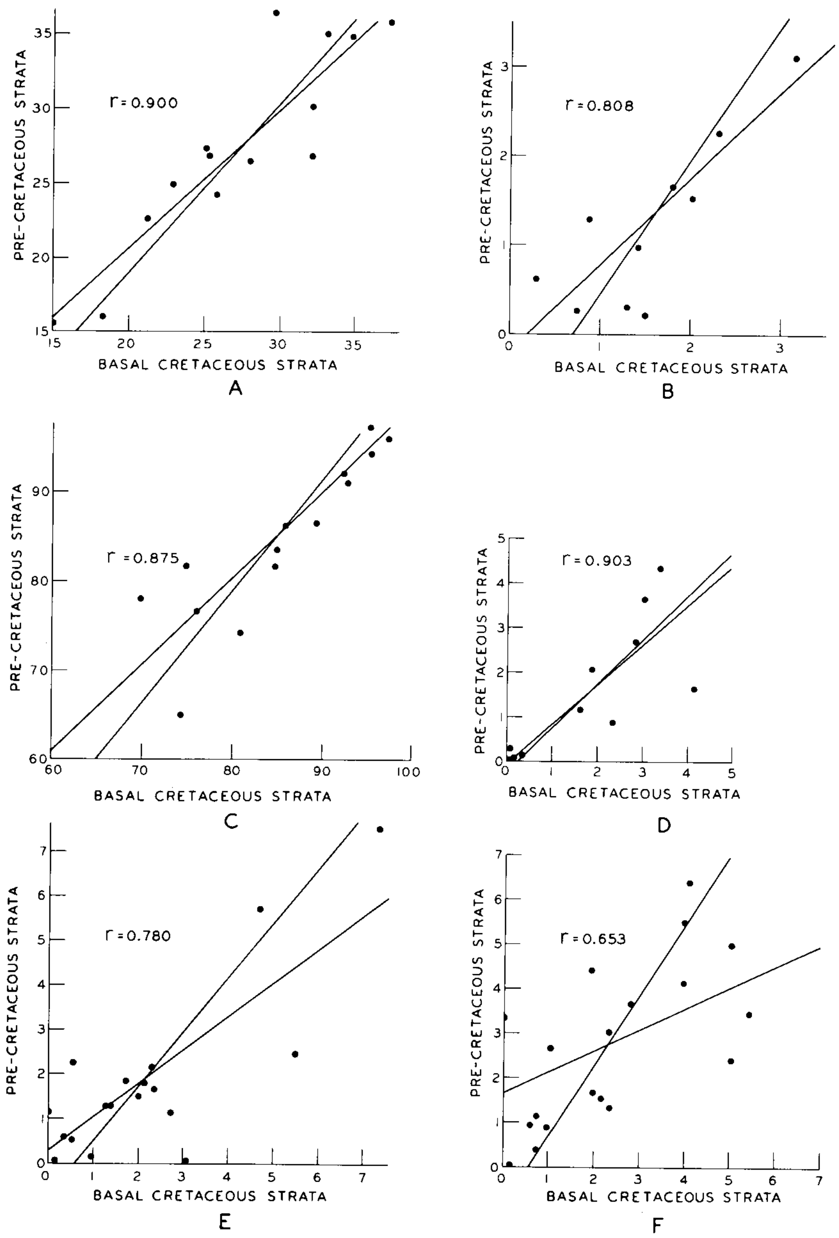

FIGURE 1. Scatter diagrams illustrating the relationship of pairs of selected attributes of crude oils and natural gases from strata in proximity to the pre-Cretaceous unconformity. A. °A.P.I. gravity of crude oils; B. Weight per cent sulphur in crude oils; C. Volume per cent methane in natural gases; D. Volume per cent butanes-plus in natural gases; E. Volume per cent nitrogen in natural gases; F. Volume per cent carbon dioxide in natural gases.

COMPOSITION OF FORMATION FLUIDS

Relationships between the composition of formation fluids from strata above and below the pre-Cretaceous unconformity may be illustrated conveniently by scatter diagrams and by maps of regional variations of their attributes. A paucity of suitable data for crude oil and natural gas precludes the illustration of most relationships by means of maps. Only a few of the analyses of crude oils and natural gases above and below the pre-Cretaceous unconformity are from the same well. Thus, analytical data from localities as far apart as two townships were combined in the scatter diagrams. Data were limited to samples taken within 250 feet above or below the unconformity, and most were from strata less than 100 feet from the unconformity.

The scatter diagrams in figure 1 illustrate the relationship between attributes from fluids above and below the pre-Cretaceous unconformity. The facts that the correlation coefficients (r) for the crude oil attributes, °A.P.I. gravity and weight per cent sulphur, are greater than 0.8 for pre-Cretaceous and basal Cretaceous reservoirs and that the ratios of the mean values are close to unity (0.99 and 0.83 respectively) are indicative of similar compositions of crude oils from strata above and below the pre-Cretaceous unconformity. Maps of regional variations of A.P.I. gravity for crude oils from these stratigraphic units are very similar.

For the various components of natural gases, correlation coefficients range from 0.65, for carbon dioxide, to more than 0.90, for butanes-plus, and the ratios of the mean values from 0.83, for butanes-plus, through 0.99 for methane, to 1.17 for carbon dioxide, indicating that at least the contents of the major components (hydrocarbons) are similar. Hydrogen sulphide was absent from most of the gases examined.

The chloride contents of formation waters in strata above and below the pre-Cretaceous unconformity are shown in figures 2 and 3. The intervals are multiples of the chloride content of present-day sea water (18,980 mg./1). There are many similarities between these two maps, although in detail they exhibit some differences. In general the chloride content of the formation water is slightly greater in the strata below the unconformity, than in the basal Cretaceous rocks.

It is thus apparent that there is a high degree of correlation in the composition of the formation fluids, at least to within an area of two townships, even though the range in values over the whole area is great.

FLOW OF THE FLUIDS

In order to determine the direction of flow of the formation fluids, potentiometric-surface maps were constructed for all pertinent stratigraphic units. These maps indicated the elevation, relative to sea level, of the surface to which fresh water would rise in wells drilled to any particular aquifer. The direction of fluid flow is from high potentiometric surfaces to low potentiometric surfaces and may be indicated conveniently by arrows drawn normal to the potentiometric-surface contours in the direction of the lowest elevation. The slope

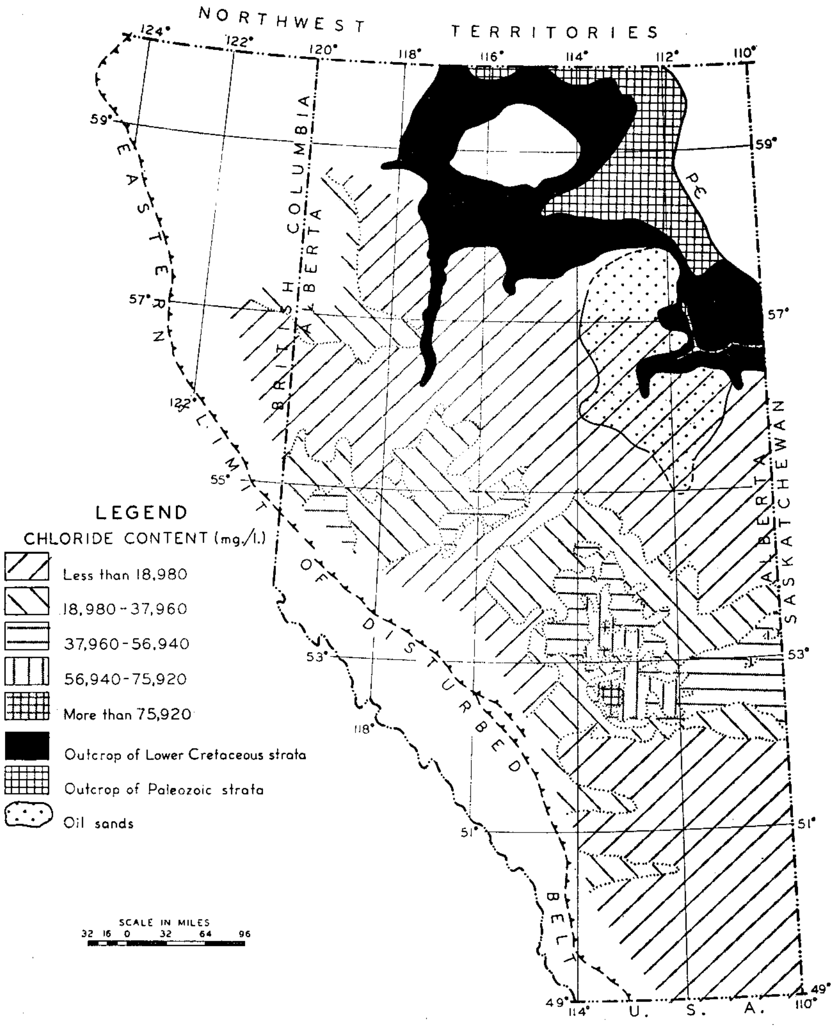

FIGURE 2. *Regional variations in the chloride content of formation waters from basal Cretaceous strata. Outline of the area underlain by the Athabasca oil sands is from Carrigy and Zamora (1960).*

of the potentiometric surface is related to the permeability. The flow pattern in the basal Cretaceous strata is shown in figure 4. Figure 5 which illustrates the flow in strata beneath the pre-Cretaceous unconformity combines the contours in the subcrop regions beneath the Cretaceous on the potentiometric-surface map of each of the pre-Cretaceous stratigraphic units shows the entire flow pattern.

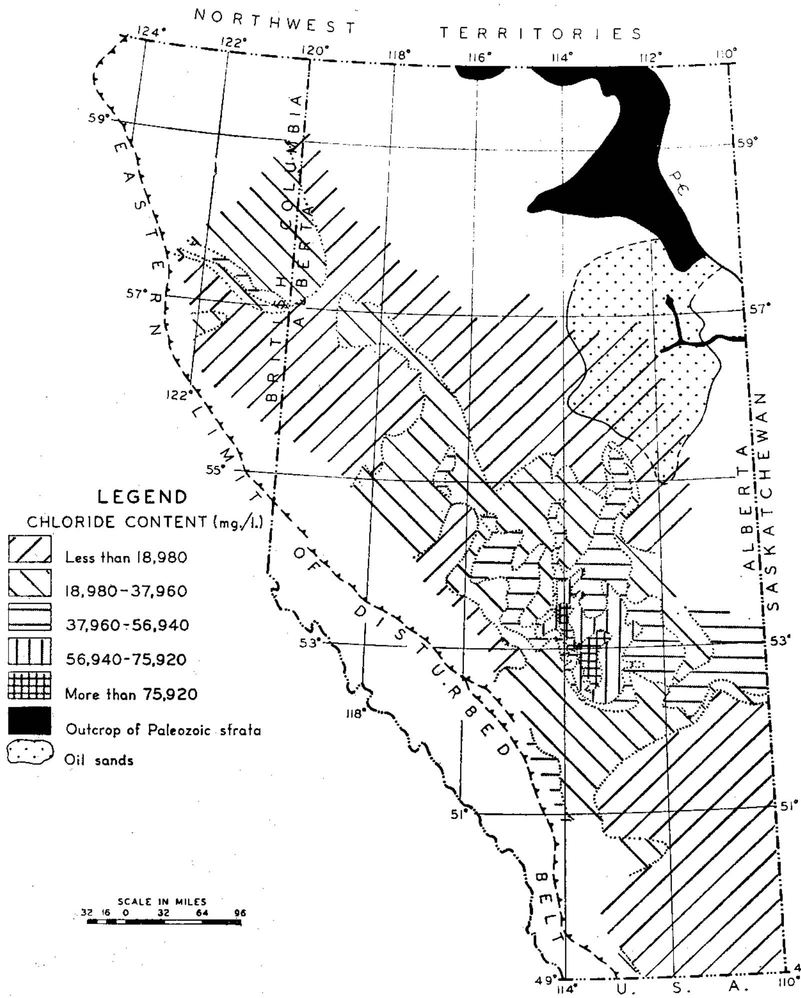

FIGURE 3. Regional variations in the chloride content of formation waters from strata immediately beneath the pre-Cretaceous unconformity.

For each individual pre-Cretaceous stratigraphic unit, the fluid flow is generally updip except for minor modifications in the subcrop region. Comparison of the potentiometric-surface maps in figures 4 and 5 reveals that the general flow direction is also updip for fluids flowing in proximity to the pre-Cretaceous unconformity, and that although there are many similarities between these two maps, they exhibit some important differences in detail. The many similarities imply essentially complete fluid connection across the pre-Cretaceous unconformity. Some of the changes in the direction of flow are

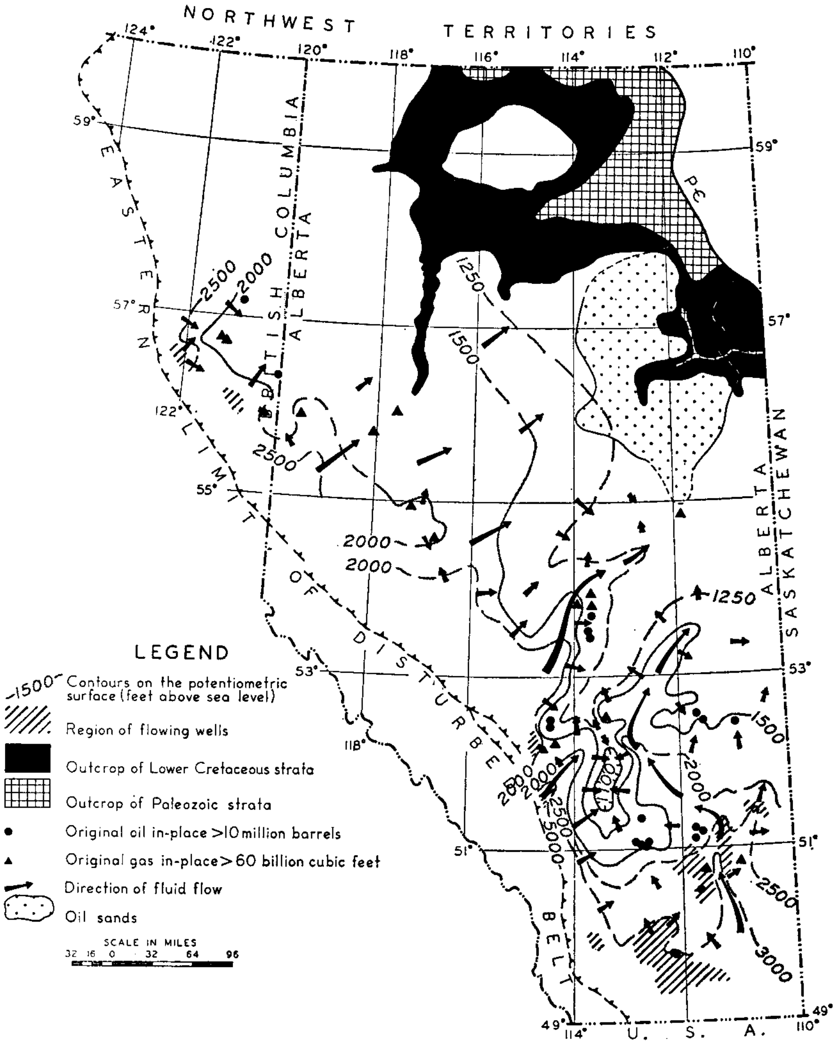

FIGURE 4. Contours on the potentiometric surface of formation waters from basal Cretaceous strata.

apparently related partly to lithological changes in the basal Cretaceous rocks near areas of non-deposition of the Ellerslie Formation (de Mille, 1960) and partly to the lithological variations and therefore permeability between strata above and below the unconformity.

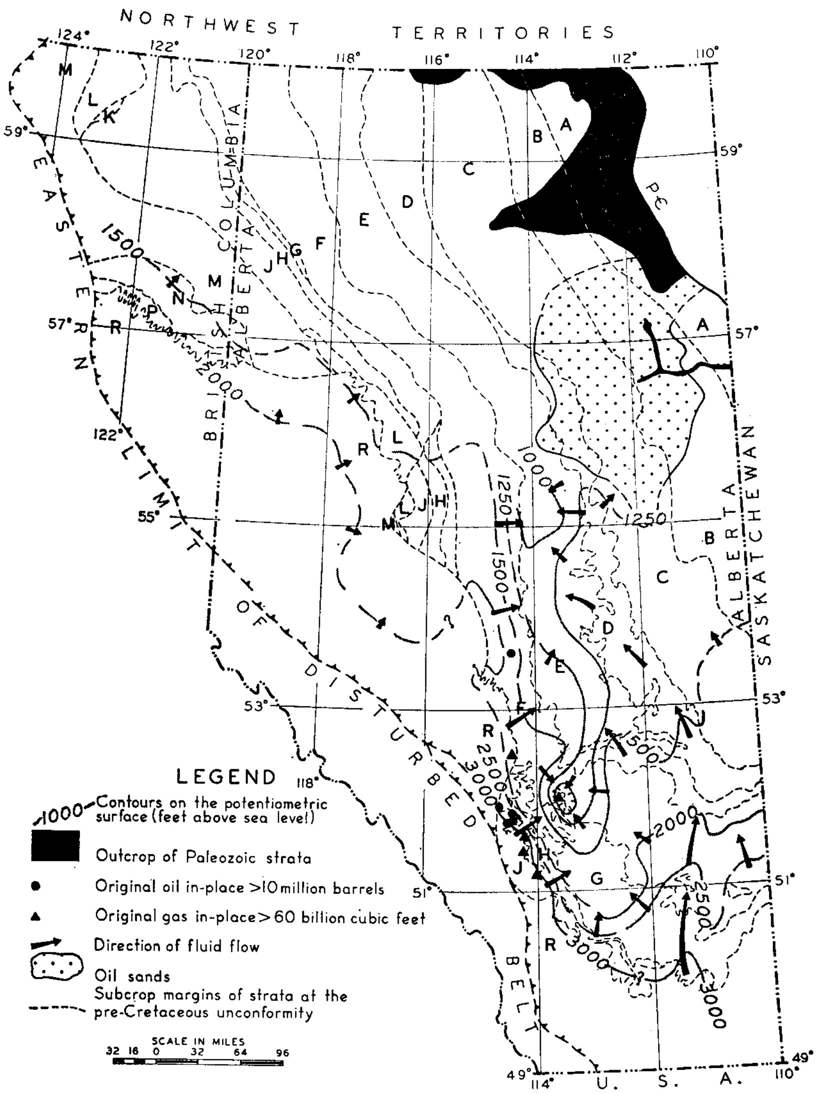

FIGURE 5. Contours on the potentiometric surface of formation waters from strata immediately beneath the pre-Cretaceous unconformity. The subcrop regions are indicated as follows:

A. Middle Devonian—Elk Point Group; B. Upper Devonian—Beaverhill Lake Formation; C. Upper Devonian—Woodbend Group; D. Upper Devonian—Winterburn Group; E. Upper Devonian—Wabamun Group; F. Carboniferous—Banff Formation; G. Carboniferous—Pekisko Formation; H. Carboniferous—Shunda Formation; J. Carboniferous—Turner Valley Formation (and lower Debolt Formation); K. Carboniferous—upper Debolt Formation; L. Permian; M. Triassic—Daiber Group and Halfway Formation; N. Triassic—Charlie Lake Formation; P. Triassic—Baldonnel Formation; R. Jurassic.

One of the most obvious examples of the differences in detail is the alteration to downdip flow in Paleozoic strata in the region immediately southwest of the Athabasca oil sands. The widely spaced 1250-foot and 1500-foot contours in figure 4 indicate good permeability in the basal Cretaceous rocks with flow towards the Athabasca oil sands. In the Paleozoic strata beneath, the closely-spaced contours indicate a rapid change in the potentiometric surface, a change probably associated with lower permeability accompanying a change in lithology across the boundaries between the Banff Formation and the Wabamun Group, and between the Wabamun Group and the Winterburn Group. The change to downdip flow represents an attempt to equalize the flow pressures beneath the pre-Cretaceous unconformity. Despite this attempt, the dominant flow is still in the basal Cretaceous strata as evidenced by comparison of the contour intervals in the two maps, and the fact that the flow regime has not been significantly altered in the highly permeable basal Cretaceous strata.

Similar analyses may be applied throughout Alberta: obviously the flow system across the pre-Cretaceous unconformity and within the strata above and below the unconformity is a complex movement of fluids in a general northeasterly direction towards the Athabasca oil sands. The dominant flow is in basal Cretaceous strata, and the pre-Cretaceous unconformity does not seem to be much of a barrier to fluid flow. It is clear that fluid movement in proximity to the unconformity is in whichever set of strata offers the least resistance to movement, i.e., those having the highest permeability.

SOURCE OF THE OIL

It has been demonstrated that the composition of formation fluids is similar in strata above and below the pre-Cretaceous unconformity, and that apparently fluids move freely across the unconformity. From the reasoning in the introduction, the crude oil has one source in either Cretaceous or Paleozoic rocks, or alternatively several sources in both Cretaceous and Paleozoic rocks with mixing of the fluids in the proximity of the unconformity.

Tar mats or *tar belts* characterize many shelf regions. Examples include the Athabasca oil sands, the Oficina Formation in eastern Venezuela, the foreland *tar belt* of the Mesopotamian-Persian Gulf basin (Weeks, 1958, 1961), and the Jurassic sands on the northwestern flank of the Williston Basin. These *tar belts* have several common features. All are vast accumulations of heavy crude oils in arenaceous sediments deposited in shallow waters. Characteristically, the sandstones subdivide and interfinger with more argillaceous strata as the region of deeper-water deposition is approached, and they act as highly permeable channels. Many of the *tar belts* are well exposed and the large quantities of *tar* are in contrast to the relatively small quantities of lighter crude oils found downdip in contemporaneous and penecontemporaneous strata.

As outlined by Hitchon (1960, 1963) the basin-shelf concept of the environmental factor in the control of compositions of crude oils and natural gases may be applied to the problem of determining the source of the vast amounts of heavy oil in the Athabasca oil sands and other *tar belts*. Briefly, the concept attempts to explain the association of vast quantities of heavy crude oils with natural gases rich in methane and nitrogen, and the association of small quantities of paraffinic crude oils with natural gases rich in hydrogen sulphide and carbon dioxide, on shelves and in basins respectively. Study of the precursors of crude oil and natural gas as found in recent sediments, and the work of Bitterli (1963) on western European primary bituminous rock sequences seems to support this concept.

In Western Canada, up to Cretaceous time, many and varied types of basins lay in the northwestern or western part of Alberta approximately coincident with the deeper parts of the present structural basin and the Rocky Mountains. Within these basins it is difficult to separate effects on formation fluids due to the environment of deposition from effects due to increasing maturation* accompanying increased depth of burial. The maturation effect may be distinguished from the environmental effect in regions where older strata were deposited in a shelf environment and the younger overlying strata in a deeper-water environment. In this situation if the maturation effect has been less active than the environmental effect, then the more deeply buried crude oils will be heavy and asphaltic, compared to the more paraffinic younger crude oils. Such a situation was described in Wyoming by Hunt (1953).

In early Cretaceous time the major areas of marine deposition lay to the north and south of Alberta with practically all of Alberta being a continental shelf at some time during deposition of the lower Cretaceous sediments. Such an environment is conducive to the development of heavy asphaltic crude oils and explains the presence of heavy oil in lower Cretaceous reservoirs at many places in Alberta.

In central and northern Alberta, the regional variations of A.P.I. gravity of crude oils in basal Cretaceous rocks (Hitchon *et al.*, 1961) indicate possible control of crude oil composition by the environment of deposition in that the A.P.I. gravities increase from topographic highs towards depositional lows. For the pre-Cretaceous stratigraphic units the general approximate coincidence of depositional and subsequent structural basins in the same region of crustal weakness has

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*Post-depositional effects may occur through many agents, and these may be termed maturation for ease of description. It must be recognized that temperature and pressure may not be the most important agents of maturation.

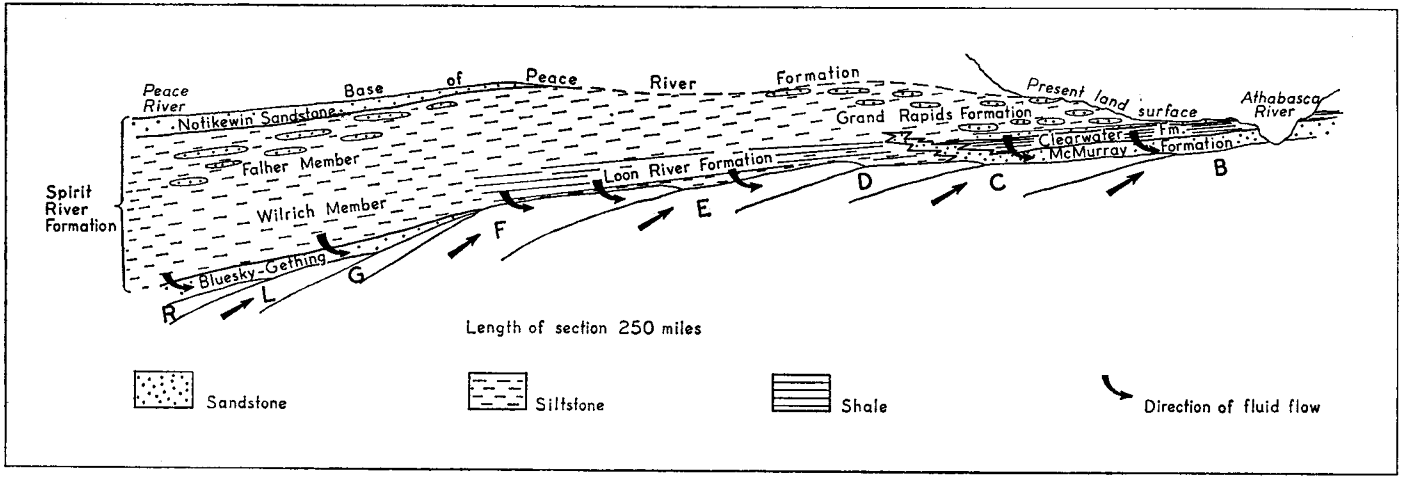

FIGURE 6. Diagrammatic lithological section from the Peace River (Tp. 84) to the Athabasca River (Tp. 91) showing the migration path of the heavy oil in the Athabasca oil sands.

made separation of environmental and maturation effects difficult. In addition, the heavy crude oils are all found in the subcrop regions, adjacent to the basal Cretaceous rocks in stratigraphic positions where contamination by basal Cretaceous crude oils or an ultimate origin from basal Cretaceous rocks cannot be ruled out.

The geological cross section (Fig. 6) shows the gradual westward increase in thickness of the Lower Cretaceous strata from the Athabasca oil sands towards the Rocky Mountains in British Columbia. In early Cretaceous times the relatively shallow depositional basin was probably asymmetrical, with its western margin within the present Rocky Mountains. Various Lower Cretaceous oil-sand deposits lie on the eastern side of the present structural basin, with the Athabasca oil sands at the eastern extreme.

Figure 6 shows lithofacies variations in the region between the Peace River and the Athabasca River. The basal Cretaceous rocks are predominantly arenaceous and comprise the reservoir rocks for the various oil-sand deposits. In the east they are overlain by marine shallow-water shales of the Clearwater Formation, which is followed by the arenaceous-argillaceous Grand Rapids Formation, which passes laterally into shale to the west. Marine shales predominate in most of the central region, but westward the upper part of the section becomes silty and sandy, with the Notikewin Formation, a dominantly arenaceous unit, appearing at the top of the succession. During Cretaceous time a depositional basin occupied a shallow depression on an unstable platform region extending from the Arctic to the Gulf Coast.

These argillaceous marine strata of Cretaceous age which overlie the basal arenaceous unit are believed to be the major source rocks for the heavy oils in the lower Cretaceous oil-sand deposits. This proposed source is supported by the evidence of the asphaltic nature of the crude oils. Also, the dominance of the fluid flow in the basal Cretaceous strata over that in the strata beneath the unconformity appears to mitigate against the pre-Cretaceous rocks as a major source of the oil. However, the oil is reported to be variable in nature (Ward and Clark, 1950), and this fact could be interpreted as implying a multiple source, although such an interpretation still permits a major Cretaceous source for the oil, with a minor additional source in the pre-Cretaceous rocks.

ACKNOWLEDGMENTS

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THE ORIGIN OF PETROLEUM PORPHYRINS: THE POSITION OF THE ATHABASCA OIL SANDS*

G. W. HODGSON**, ERIC PEAKE** AND B. L. BAKER**

ABSTRACT

The Athabasca oil sands contain a crude oil which is similar in metal-porphyrin content to other high-sulphur oils. It has a predominance of vanadyl porphyrin pigments over nickel pigments and while the bulk of the porphyrin material is decarboxylated, a small quantity exists as free acids and some as organic esters. These pigments were examined in the present study with reference to those present in conventional crude oils, other oil sands and related source environments. Conventional crude oil shows a trend toward dominance of the nickel porphyrins with increasing API gravity and decreasing sulphur. Low-sulphur low-gravity oils, as in the Utah and Madagascar oil sands, show a marked deficiency in vanadyl pigments. Examinations of the pigments in ancient sediments show the presence of porphyrin pigments, and recent sediments show the presence of chlorin pigments in addition to porphyrins. The younger the sediments, the more dominant are the chlorin pigments. Nickel complexes of porphyrins were found in open-sea marine sediments, but were overshadowed by vanadyl porphyrins in sediments from a restricted marine bay. Metal chlorins are common in recent sediments, in ground waters and in plants. Coal, peat and oxidized marine shales showed extensive degradation of pigment. Precambrian rocks are evidently devoid of porphyrin pigments.

The Athabasca oil is apparently typical of a source region of restricted circulation marked by high rates of deposition and strong reducing potentials.

INTRODUCTION

The origin of petroleum porphyrins relates directly to the origin of crude oil. Compared with the multitude of complex compounds comprising crude oil, the porphyrins are distinctive pigment compounds which can be studied in some detail. Porphyrins evidently arise from the same environments as the hydrocarbons which ultimately become crude oil, and therefore offer a means for defining more clearly the role of the environment in the origin of petroleum. The Athabasca oil sands are a major accumulation of crude oil, a study of which would be expected to yield information of considerable importance in the general understanding of the origin and accumulation of crude oil.

While the study of the origin and accumulation of petroleum in recent years has been pursued with some vigour, there have been few attempts to relate all of the factors which are involved in the overall process. Attention has been focussed upon the presence of hydrocarbons in recent sediments with particular emphasis upon the origin and distribution of the various compounds. Little is known of the mobilization and migration of the hydrocarbons from the source

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*Contribution No. 222

**Research Council of Alberta, Edmonton

sediments. The understanding of the migration processes as they relate to the removal of the hydrocarbons from the source to the site of accumulation is meagre, and the mechanisms governing the collection of crude oil at the ultimate reservoir are obscure.

To approach the problem of oil genesis on a broad front with appropriate attention directed to each of the factors outlined would require an effort of considerable magnitude, well beyond the scope of the present study. The present study was limited therefore to an examination of the position of the pigments in the general picture of oil genesis, with particular reference to the Athabasca oil sands as an important example of an oil accumulation. An examination was first made of the occurrence of pigments in conventional crude oils, augmenting published data with new data. Next, the oil sands were put into perspective with the conventional oils on the basis of their pigment contents. Thirdly, the Athabasca oil sands were compared with other oil-sand accumulations containing both high-sulphur and low-sulphur oils. Ancient shales are commonly regarded as being related to the genesis of petroleum, and attention was then directed to the pigments existing in some Lower Cretaceous and Devonian shales in Alberta. This was followed by measurements of pigments in unconsolidated sediments, with attention directed to deltaic, freshwater, and marine sediments. Special attention was given to samples taken from the water-sediment interface and particulate matter existing in lake waters and in springs. The study of the pigments in these environments led to an examination of the pigments found in plants, and consideration was given to the possibility that at least some of the petroleum pigments came from plant pigments other than the chlorophylls. Thus, the information accumulated in the present study, while not comprehensive in depth, has defined more clearly the distribution of petroleum porphyrins and how petroleum geochemistry can add to our understanding of the position of the Athabasca oil in comparison to other crude oils.

PETROLEUM PORPHYRINS

The term porphyrin is generally taken to mean a tetra pyrrole pigment of the structure shown in figure 1. Obviously there is scope for a large number of porphyrin compounds, by substitution of various organic groups on the several peripheral and linking positions occupied by hydrogen atoms. This, and several other basic features of petroleum porphyrins, are described in some detail in another communication (Hodgson *et al.*, 1962). Perhaps one of the more unexpected features of porphyrins is the rather few compounds which evidently exist in petroleum. The major porphyrin pigment is reported to be deoxophylloerythroetioporphyrin, which exists in crude oil as vanadyl and nickel complexes (Treibs, 1935a). The structures of the metal complexes of this compound are shown in figure 2. It is convenient in porphyrin geochemistry to refer to these compounds as H_2P_p, VOP_p, and NiP_p where P refers to pigment and $_p$ to porphyrin. The concentration of such compounds in crude oil generally ranges from 1 to 500 p.p.m.

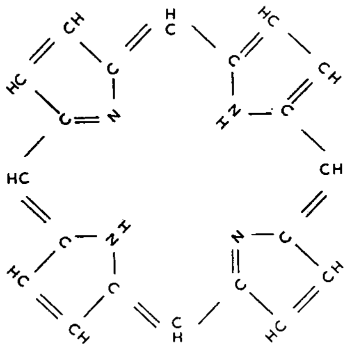

FIGURE 1. Porphyrin structure.

The concentration of petroleum porphyrins in crude oil is usually determined by a modification of the Treibs technique (Groennings, 1953). This depends upon a digestion of the oil with a solution of hydrobromic acid in glacial acetic acid to remove the complexed metals from the porphyrin compounds. The pigments are thereby rendered acid-soluble and are readily separated from the bulk of the crude oil. Table 1 shows the pigment content of selected crude oils from various parts of the world. Included in the table are published data from Alberta, Saskatchewan, Venezuela, Oklahoma, Texas, Kansas, and California. It indicates that the trace-metal content of an oil, especially the vanadium and nickel content, is related to the porphyrin content. However, on a molar basis the amount of porphyrin present is always inadequate to account for the total vanadium and nickel.

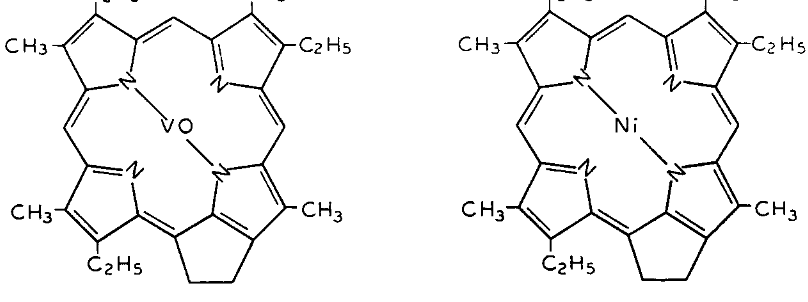

FIGURE 2. Structure of vanadyl and nickel porphyrins found in petroleum.

TABLE 1. CONTENT OF PORPHYRINS AND METALS IN CRUDE OILS FROM CANADA, UNITED STATES AND VENEZUELA

Oil	Age	Ni (p.p.m.)	V (p.p.m.)	Porphyrins (p.p.m.)	Carboxylated Porphyrins (per cent, of total)
Malmo, Alberta	Devonian	0.7	0.6	3	—
Pembina, Alberta	Cretaceous	1.3	0.6	4	—
Malmo, Alberta	Cretaceous	1.2	1	5	—
Roselea, Manitoba	Mississippian	3	4	11	—
West Texas*	**	5	23	12	—
Bawlf, Alberta	Devonian	5	2	13	—
Malmo, Alberta	Devonian	6	2	38	—
Redwater, Alberta	Devonian	10	4	40	—
Smiley, Saskatchewan	Cretaceous	2	1	49	—
Tia Juana, Venezuela	**	24	187	60	—
Campbell, Alberta	Cretaceous	5	10	65	—
Coleville, Saskatchewan	Mississippian	32	94	110	—
Bantry, Alberta	Cretaceous	18	57	117	—
Conrad, Alberta	Jurassic	24	73	135	—
Rhodes, Kansas*	Mississippian	33	133	135	0.0
Vermilion, Alberta	Cretaceous	35	90	162	—
Tatums, Oklahoma*	Pennsylvanian	71	148	165	0.0
Lagunillas, Venezuela*	Tertiary-Cretaceous	41	317	170	0.1
Wilmington, California*	**	60	46	200	—
Santa Maria, California*	Tertiary	130	280	300	1.8
Backaquero, Venezuela*	Cretaceous	53	430	380	0.2
North Belridge, California*	Pleistocene	83	23	390	5.4
Boscan, Venezuela*	**	66	900	1680	—

*Data from Dunning *et al.* (1960)

**No age given

The following procedure was developed in the present investigation to permit the direct determination of VOP_p and NiP_p in crude oil:

> The oil was extracted by methanol, so that a concentrate of porphyrin material could be obtained. The spectrum of the resulting methanol solution in general showed the absorption features characteristic of the porphyrin material. In practice, the oil (about 2 grams) was dissolved in benzene (about 10 ml.) and to this was added about 100 ml. of methanol. The asphaltic material was thus thrown out of solution; precipitation of the asphaltic material was aided by the addition of about 5 grams of cellulose powder. The mixture was allowed to settle and the supernatant liquid containing porphyrin compounds, decanted through a filter paper. A second extraction was carried out in a similar manner with the addition of further benzene (about 20 ml.) followed by methanol (about 100 ml.). Subsequent extractions were done until the yield of further porphyrin material became negligible. The extracts were then combined and an estimate of the quantity of VOP_p and NiP_p made from the spectrum. For more precise work, the foregoing solution was reduced to incipient dryness, and the pigments taken up in *n*-hexane. An aliquot of the *n*-hexane solution was taken for chromatography on silica gel. The NiP_p fraction was eluted by 50-50 v.v. hexane-benzene. Elution of the pigment mixture from silica gel with 50-50 v.v. chloroform-benzene removed the VOP_p fraction. Concentration of the elutes followed by spectral examination of the 550 mμ and 570 mμ peaks of NiP_p and VOP_p respectively gave the contents of these pigments in the crude oils using an extinction coefficient of 2.1×10^4 l./mole/cm.

This procedure was used in the present investigation and in a related one for selected crude oils from Japan. The data are given in table 2, along with a summary of the corresponding data for oil from Japan (Hodgson *et al.*, 1963). Included as well are data for the total vanadium, nickel, sulphur and A.P.I. gravity.

The contents of the two pigment classes in the oils generally fall in the ranges of 1 to 300 and 1 to 100 p.p.m. for VOP_p and NiP_p respectively. For heavy oils, or more particularly for oils with high sulphur content, the VOP_p content exceeds that of NiP_p. This is consistent with the observations of Radchenko and Sheshina (1955) who attributed a major part of the VOP_p in petroleum to secondary biological action. The molar concentrations are such that only about 20 per cent of the metal content can be accommodated in porphyrin pigments.

Crude oils also contain other porphyrin pigments, principally the corresponding pigments with added carboxylic acid groups. These are evidently complexes of deoxophylloerythrin, which might be written as VOP_pCOOH and NiP_pCOOH. While it might be expected that these could be recovered by a simple alkali wash of the oil, it was found that recourse to a chromatographic separation avoided the difficulties arising from extensive emulsion formation. Further, Blumer and Omenn (1961) found that acid extraction when applied to extracts from bituminous materials, i.e. solvent partitioning, often fails to achieve the expected separation. This is caused by the presence

TABLE 2. CONTENT OF METALS, SULPHUR AND METAL PORPHYRINS IN CRUDE OILS FROM CANADA, JAPAN AND TRINIDAD

Oil	Age	Formation	Gravity (A.P.I.)	Ni (p.p.m.)	V (p.p.m.)	S (per cent)	VOP_p (p.p.m.)	NiP_p (p.p.m.)
Smiley	Cretaceous	Viking	35.2	2.8	1.1	0.1	Trace	Trace
Pembina	Cretaceous	Cardium	34.3	0.8	0.4	0.2	Trace	Trace
Roselea	Mississippian	Mission Canyon	34.0	2.9	4.3	0.2	7	1
Daly	Mississippian	Lodgepole	33.1	5.3	7.0	0.2	14	1
Morinville	Devonian	Wabamun	35.4	2.8	2.2	0.3	4	4
Springburn	Devonian	Beaverhill Lake	37.1	6.2	1.2	0.4	2	5
Trinidad, AT 3	Tertiary	Cipero	—	17.0	14.0	0.4	8	8
Redwater	Devonian	Leduc	33.9	8.8	4.7	0.5	7	15
Trinidad, Seep No. 1	Tertiary	Cipero	—	1.8	1.6	0.6	11	9
Duhamel, D 2	Devonian	Nisku	35.4	3.9	2.9	1.0	10	8
Stettler, D 2	Devonian	Nisku	29.6	15.2	11.4	1.4	90	Trace
Stettler, D 3	Devonian	Leduc	28.2	13.8	16.2	1.9	55	30
Wapella	Jurassic	Shaunavon	23.6	17.0	29.8	2.3	110	30
Lac Ste. Anne	Devonian	Wabamun	22.9	27.0	84.0	2.7	175	15
Cantuar	Jurassic	Vanguard	14.9	52.3	135.0	3.1	165	40
Eastend	Jurassic	Lower Shaunavon	21.6	33.0	83.5	3.4	290	65
Coleville	Mississippian	Banff	13.4	5.0	13.3	3.6	210	25
Lloydminster	Cretaceous	Wainwright	15.0	48.0	105.0	3.8	220	20
Yabase, Sotoasahikawa, R-27	Tertiary	Onnagawa	35.6	5.0	1.0	0.17	23.7	64.3
Amarume (Light)	Tertiary	Funakawa	38.8	1.0	1.2	0.18	0.7	2.4
Yabase, Koya R-30	Tertiary	Tentokuji	28.2	3.0	0.5	0.20	2.2	9.1
Yabase, Koya R-63	Tertiary	Onnagawa	35.1	8.0	1.5	0.21	5.3	17.5
Yabase, Koya-C-11	Tertiary	Tentokuji	24.8	5.0	1.5	0.27	2.0	13.7
Amarume (Medium)	Tertiary	Funakawa	31.9	1.0	0.5	0.28	1.0	3.6
Yabase, Sotoasahikawa R-114	Tertiary	Onnagawa	30.2	30.0	7.5	0.28	19.7	95.9
Yabase, Koya R-93	Tertiary	Onnagawa	30.4	50.0	6.5	0.41	8.1	25.3
Ishinazaka R-28	Tertiary	Shibikawa	18.7	15.0	11.0	1.04	54.0	54.0
Chokaizan R-16	Tertiary	Funakawa	8.2	50.0	170.0	3.57	16.5	47.8

of certain ether-soluble materials which alter the partition coefficients in favour of the organic phase.

VOP$_p$COOH and NiP$_p$COOH were determined as follows:

An aliquot of the total extract in hexane was put on cellulose powder in a column. Elution with diethyl ether and 50-50 benzene-propanol removed the carboxylated pigments. These were alkali-extracted from the organic solvents. Successive extractions were made with one per cent sodium hydroxide followed by ether backwashes and filtration of the aqueous solution to remove co-extracted VOP$_p$ and NiP$_p$ and suspended matter. The aqueous solution was acidified with hydrochloric acid and the carboxylated pigments were extracted with ether. This gave a solution of the NiP$_p$COOH and VOP$_p$COOH pigments, the concentrations of which were readily determined in the presence of each other through the respective absorption maxima at about 550 and 570 mμ, using an extinction coefficient of 2.1 x 10^4 1./mole/cm.

In all oils, the concentration level of the carboxylated pigments was much lower than that of the corresponding decarboxylated pigments, as found by Dunning *et al.* (1960). Table 3 gives the quantities of such pigments in the oils from Japan, as reported earlier. The content of the carboxylated pigments was less than one p.p.m. in all oils. In most oils the vanadyl and nickel pigments were mutually exclusive, with NiP$_p$COOH being predominant in the low-sulphur oils and VOP$_p$COOH in the high-sulphur oils.

TABLE 3. CARBOXYLATED PORPHYRIN PIGMENTS IN TERTIARY CRUDE OILS FROM JAPAN*

Oil Field	Formation	Gravity (°A.P.I.)	Porphyrin Content (p.p.m. of oil)	
			VOP$_p$COOH	NiP$_p$COOH
Yabase, Sotoasahikawa R-27	Onnagawa	35.6	**	$\leqslant$0.03
Amarume (Light)	Funakawa	38.8	**	**
Yabase, Koya R-30	Tentokuji	28.2	**	0.2
Yabase, Koya R-63	Onnagawa	35.1	$\leqslant$0.1	0.1
Amarume (Medium)	Funakawa	31.9	**	$\leqslant$0.05
Yabase, Koya C-11	Tentokuji	24.8	**	0.4
Yabase, Sotoasahikawa R-114	Onnagawa	30.2	0.03	**
Yabase, Koya R-93	Onnagawa	30.4	$\leqslant$0.02	**
Ishinizaka R-29	Shibikawa	18.7	$\leqslant$0.1	$\leqslant$0.1
Chokaizan R-16	Funakawa	8.2	0.7	**

*After Hodgson *et al.* (1963)
**Below detection limits

There have been some reports of the presence of other porphyrin and porphyrin-like pigments in crude oils. Notably, there has been the report of chlorin pigments by Howe (1961). Chlorins are dihydroporphyrins which might, therefore, be regarded as hydrogenation or reduction products of porphyrins. Chlorophyll *a* for example, is a well-known chlorin, and might be compared with hemin, a well-known naturally occurring porphyrin. Generally, chlorins are less stable than porphyrins. The spectra of chlorins have principal absorption features in the 600-700 mμ range, while porphyrin features are in the 500-600 mμ range; briefly, chlorins are green and porphyrins are red. While chlorin-like material from crude oil has been reported from time to time, possibly the pigments are either partly oxidized porphyrins such as MP_pOH (Fisher and Dunning, 1961) or secondary chlorins formed through some reduction of porphyrin pigments (Blumer and Omenn, 1961).

The porphyrins of petroleum are commonly regarded as having arisen from pigments of living matter. They appear to be more closely related to the chlorophyll compounds than to the hemin compounds. This is indicated by the spectra of the pigments which show a substitution on one of the bridging carbon atoms. This substitution corresponds to the fifth ring which is present in chlorophylls but absent in hemin compounds. The major petroleum porphyrin pigments are of the chlorophyll type, although small quantities of hemin-like porphyrins have been reported (Fisher and Dunning, 1961). While it would be reasonable to attribute the origin of the major petroleum porphyrins to chlorophylls of plants, with the hemin-like compounds arising from animals, the presence of hemin-type pigments in plants has also been recognized (Fisher and Dunning, 1961).

ATHABASCA OIL

The oil of the Athabasca oil sands has been examined from time to time for its porphyrin content. Hodgson and Baker (1957) measured the thermal stability of the porphyrin pigments in the oil and related this to the trace-metal content. Champlin and Dunning (1958) reported 30 p.p.m. of a nickel porphyrin complex in the oil. This compound appeared spectrally identical to the nickel complexes in conventional crude oils. Vanadyl complexes totalled about 222 p.p.m. of the oil. These calculations were based upon extinction coefficients of 3.4×10^4 l./mole/cm. The corresponding nickel and vanadium contents were 80 and 180 p.p.m. Thus, as pointed out by Champlin and Dunning (1958), only about one-tenth enough porphyrin is present to complex all of the vanadium and nickel present. Comparison with several other heavy oils showed that the Athabasca oil was not unique in this respect. Further, with its high sulphur content the Athabasca oil shows the same predominance of vanadyl complexes over nickel complexes as do the conventional high-sulphur oils.

The porphyrin contents of three samples of Athabasca oil were measured in the present study by the method outlined above for crude oils. The results of a related investigation concerned with establishing the molar extinction coefficients for a series of porphyrins and their metal complexes indicated a value of 2.1×10^4 for petroleum porphyrins, rather than the 3.4 × 10^4 used by Champlin and Dunning. This results in a higher apparent porphyrin concentration for a given spectral analysis. The results of these analyses are given in table 4. Included are values for vanadium, nickel, and sulphur. A rather wide spread in values is apparent, but the existence of a certain uniformity is also evident.

TABLE 4. CONTENT OF METALS AND PORPHYRIN COMPLEXES IN THREE SAMPLES OF ATHABASCA OIL

	Sample No.		
	1	2	3
		Source	
	Bitumount	G.C.O.S. lease 4 (typical)*	G.C.O.S. lease 4 (atypical)*
Sulphur %	5.0	4.8	3.7
Nickel, p.p.m.	77	100	31
Vanadium, p.p.m.	220	130	75
NiP_p, p.p.m.	45	22	21
VOP_p, p.p.m.	500	370	338

*Great Canadian Oil Sands Limited lease 4 samples were provided by Dr. K. A. Clark as typical and atypical of the Athabasca oil sands in general.

A search was made for carboxylated porphyrin pigments in two samples of Athabasca oil. About 2 p.p.m. of VOP_pCOOH was found in each. The analytical methods involved in the foregoing general analyses were not specific for differentiating between decarboxylated metal complexes of porphyrins (MP_p) and esters of carboxylated metal porphyrins (MP_pCOOR). Pigments separated from these two samples were subjected to saponifying conditions (10 per cent aqueous KOH, refluxed for 30 minutes) to test for the existence of such esters. A small but clearly defined portion of the alkali-insoluble material appeared to be saponified, at concentrations of 11 and 14 p.p.m. of the oil, respectively. The esters, with absorption maxima at 568 and 410 mμ, thus appeared to be VOP_pCOOR. NiP_pCOOR was not detected.

Champlin and Dunning (1958) reported the presence of several metal complexes of porphyrins in the Athabasca oil. These pigments may represent the VOP_p, VOP_pCOOH and VOP_pCOOR classes of compounds reported in the present study, or they may of course relate to a number of VOP_p compounds.

Another pigment which appeared to be present with the major pigments was marked by an absorption feature at 588 mμ. Attention has recently been drawn to this peak by Costantinides and Arich (1963), but no identification of the peak was attempted.

MELVILLE ISLAND OIL SAND

In considering the pigment content of the Athabasca oil it is of interest to relate it to the recently reported Arctic oil sand of Melville Island. This oil sand has ben described by Sproule (1962) and Hunt (1963) and is more fully reported by Sproule and Lloyd elsewhere in the present volume (p. 27). It is sufficient to note in the present discussion that the oil sand is exposed on the northern tip of Melville Island in the Canadian Arctic and is believed to be of Triassic age. The sample used in the current study was provided by Dr. Sproule. It appeared to be weathered, as would be expected of surface outcrops, but the likelihood of weathering was reported to be very small, since it was obtained from a permafrost environment (Sproule, 1963). The sample was extracted exhaustively with benzene, and the benzene solution was filtered to remove as much fine suspended material as possible. The benzene was removed by distillation and evaporation, leaving a hard, shiny pitch-like material, very much more viscous than the Athabasca oil. The sulphur content was found to be somewhat lower than expected for such a viscous oil—about 2.3 per cent. The vanadium content was low, 15 p.p.m.; the nickel content was 81 p.p.m. Since the analytical approaches used in the foregoing experiments failed to show any porphyrin pigments in the Melville Island oil, a portion of the raw sand was directly extracted with methanol. This yielded an extract containing a vanadyl porphyrin in the amount of about 0.8 p.p.m. of the contained oil. Nickel pigments were below detection limits, i.e. less than 0.2 p.p.m. for oils. The Treibs-Groenning HBr-HOAc digestion method showed no further pigments in the extraction residue. Evidently the failure to detect pigments in the extracted oil was related to the fact that a significant amount of suspended mineral matter was filtered off and discarded during the course of benzene extraction of the oil from the sand. This mineral matter may have carried the pigments as there is evidence, as will be noted later, that porphyrin pigments tend to be associated with clay minerals. An oil having the sulphur content and viscosity of the Melville Island oil would be expected to contain more than 100 p.p.m. of VOP_p. The question then arises as to whether the pigment content for the Melville Island oil sand is a true reflection of the character of the oil, or whether it was caused by the weathering which the oil sand may have experienced at some time. Porphyrin pigments, particularly metal complexes, are usually regarded as very resistant to degradation, but reports have been made which indicate their susceptibility to oxidative degradation (Costantinides and Batti, 1957). However, limited study in the current investigation failed to show any susceptibility to oxidation when raw Athabasca oil sand was suspended in hot water through which air was bubbled for several days, even though the oil was thereby dislodged from the sand. Later iron and vanadium salts were added to promote oxidation, but no evidence of destruction of the pigments was obtained.

These few data indicate that Melville Island oil sand is different from the Athabasca oil sand.

CALIFORNIA OIL SANDS

The Athabasca oil sands are similar in a number of ways to oil sands in California. Two samples from each of two areas in California were examined. One area was near Carpinteria in Santa Barbara County. One sample was of Carpinteria oil-sand outcrops of Pleistocene age; the other was from a well producing from fractures in the underlying shale. The other two samples were from oil-saturated sands at Edna, San Luis Obispo County. Both were from the Pismo Formation which ranges from Pliocene to Miocene in age. One was a heavy black oil produced from a well adjacent to the outcrop and the other an oil extracted from oil-sand outcrop material. The pertinent data for these four samples are in table 5. In all samples, the vanadyl porphyrin content exceeds that of the nickel porphyrin. The range in concentration is very broad: 75 to 1130 p.p.m. for the vanadyl porphyrins, and 5 to 390 p.p.m. for the nickel porphyrins. These sulphur-rich oil sands and oils are marked by pigment distributions in which the vanadyl pigments are predominant, in keeping with the patterns noted for conventional oils and the Athabasca oil.

TABLE 5. CONTENT OF METALS AND PORPHYRINS IN CALIFORNIA OIL SANDS AND OILS

Sample	V (p.p.m.)	Ni (p.p.m.)	S (% of oil)	VOP_p (p.p.m. of oil)	NiP_p (p.p.m. of oil)
Carpinteria oil-sand oil	255	115	3.7	1060	110
Carpinteria oil	210	110	2.5	1130	390
Edna oil-sand oil	200	120	4.2	75	5
Edna oil	13	65	2.1	390	120

It is perhaps significant to note that while there is some similarity between the produced oil and oil-sand oil in the Carpinteria location, the Pismo Formation samples show a marked divergence.

LOW-SULPHUR OIL SANDS

In the oil sands of Utah and Madagascar the sulphur content of the contained heavy oils is very low, being no more than 0.6 per cent. Samples of these oil sands were extracted and examined in the usual manner. The data are given in table 6, and the significant feature is that the content of vanadyl porphyrins is very low, being

TABLE 6. CONTENTS OF METALS AND PORPHYRIN COMPLEXES IN LOW-SULPHUR OILS FROM OIL SANDS

Sample	V (p.p.m.)	Ni (p.p.m.)	S (%)	VOP_p (p.p.m.)	NiP_p (p.p.m.)
Utah	10	88	0.4	*	220
Madagascar	16	170	0.6	*	75

below detection limits, while the nickel porphyrins are present in some abundance.

It is pertinent to observe that gilsonite examined by Sugihara and McGee (1957) with a sulphur content of only 0.3 per cent, also showed the nickel porphyrin content to be predominant.

It is now of some interest to turn to the occurrence of porphyrins in a wide variety of other natural environments.

SHALES ASSOCIATED WITH THE ATHABASCA OIL SANDS

The Athabasca oil sands are associated with shales. Some are believed to be of fresh-water origin; others of brackish-water and marine origin. Five shale samples were extracted with mixed benzene-methanol (90-10 v.v.) and the extracts examined for pigment content. The results are shown in table 7. The nickel porphyrin pigments fall in the range of <0.001 to 0.2 p.p.m.; the vanadyl, from 0.002 to 0.3 p.p.m. In all cases, except one marine shale sample, the vanadyl pigment was more abundant than the nickel porphyrin. This marine shale sample was examined further, and no indication was found of the corresponding carboxylated pigments, either free acid or esterified. Related work (Hodgson *et al.*, 1963) had shown the presence of NiP_pCOOR, but not VOP_pCOOR, in a Miocene shale in Japan. Demetallation of the Athabasca marine VOP_p and NiP_p pigments showed the usual petroleum free phyllo-type porphyrin pigments.

TABLE 7. CONTENT OF PORPHYRIN PIGMENTS IN SHALES ASSOCIATED WITH ATHABASCA OIL SAND

Sample No.	Environment of deposition	NiP_p (p.p.m.)	VOP_p (p.p.m.)
1	Fresh-water	0.03	0.3
2	Fresh-water	*	0.002
3	Brackish	0.05	0.3
4	Marine	0.2	0.08
5	Marine	0.02	0.08

*Below detection limits, i.e. <0.001 p.p.m.

The presence of pigments in the shales closely associated with the oil sands drew attention to the pigments in the finely divided minerals in the sands themselves. In the normal hot-water separation processes for the production of oil from the oil sands as developed by Dr. K. A. Clark over the years (e.g. Clark, 1950), a suspension of clay minerals (kaolinite and illite) and finely divided quartz is obtained. A few samples of the mineral suspension were examined in the present study for porphyrin and oil content. The pigment content, which was principally a vanadyl porphyrin, was about 22 p.p.m. of the mineral matter, more than ten-fold greater than in the shales described above. Referring the pigment content to the oil accompanying the suspended mineral matter, the pigment content was about 380 p.p.m. of the oil, roughly the same as in the oil of the oil sands.

A further example of the association of suspended solids with pigments was afforded by an experiment in which water was forced to "seep" through a column of oil sand in the laboratory. A cloudy effluent was obtained which carried about one microgram of vanadyl porphyrin per litre of water. Related to the solid material carried by the seeping water, the pigment was about 3000 p.p.m. of the solids, very much higher than in the associated shales.

PRECAMBRIAN ROCKS

It is generally assumed that petroleum pigments arise from biogenic material, and it was of interest therefore to consider their presence in rocks of Precambrian age, in which little biogenic material would be expected. Five samples of pegmatite containing carbon in the form of graphite, and a slate sample were examined. All six samples showed the presence of chlorin-like material, but no porphyrins. All of the pegmatite samples showed evidence of lichen growth on the surface of the samples and this could not be completely removed. Chlorophylls from the lichens undoubtedly gave rise to the chlorins found in the extracts. Limits of detection were about 0.0005 p.p.m. for porphyrin material.

Pegmatitic graphite probably bears little relation to ordinary commercial graphite; however, a sample of the latter was examined and found to be devoid of porphyrin pigments for similar limits of detection.

Treatment of the above extraction residues with HBr-HOAc to remove any strongly adsorped pigments also failed to show the presence of any porphyrin pigments.

DEVONIAN SHALES

Ireton shale samples (RG 15/59, cored 734-744′) were examined for porphyrin pigments, and both nickel and vanadyl pigments were found. The nickel pigment was present to the extent of 0.8 p.p.m. of the shale or 1700 p.p.m. of the extractible organic matter in the shale; the vanadyl pigment, about 0.08 and 170 p.p.m. respectively. The nickel pigment was further studied, but no indication was obtained of any NiP_pCOOH nor NiP_pCOOR. Evidently the pigment was NiP_p, indistinguishable from the usual petroleum nickel pigment. There was no evidence to suggest that the vanadyl pigment was different from the VOP_p of crude oil.

The pigment content of the extractible material of the shales is very much greater than that of the associated Devonian crude oils. Perhaps this has some significance in connection with the high pigment content of the suspended mineral matter in the Athabasca-oil-sand-separation process.

Another shale of Devonian age was examined for pigments. This was a badly weathered Perdrix shale, and the pigments, if present, were below detection limits, i.e. less than 0.0005 p.p.m. of the shale.

COAL

Before passing on to more recent sediments, consideration might be given to the pigments which occur in sediments which have had a somewhat different geochemical history. Perhaps one of the most interesting environments is that of coal formation.

Coal has long been known to contain porphyrin pigments (Treibs, 1935a, 1936). However, the coal pigments are believed to be made up of the "etio" class of porphyrins, i.e. those of the hemin series, without the fifth ring which is common to the chlorophyll series.

In the present study two coals were examined: a subbituminous coal and a lignite. They were subjected to the usual extractive procedure using benzene and methanol so that an indication might be obtained as to whether the pigments were complexed with metals. Earlier work by Treibs (1935b) had used a digestion approach which obscured the nature of the pigments in the coal. But the extractive approach in the present study failed to remove any pigments, probably because of the strong absorptive capacity of the coals, and HBr-HOAc digestion was required. The subbituminous coal from Lethbridge, Alberta (Monarch Coal Company) yielded 0.16 p.p.m. pigments (dried weight). There was no indication of esters of the pigments, but roughly 10 per cent of the pigment aggregate or 0.015 p.p.m. of the coal was present as free carboxylic acid pigments following the digestive procedure. The pigment aggregate showed the "etio" type of spectrum, a spectrum distinct from the usual petroleum porphyrin spectrum.

Lignite coal from Roche Perce, Saskatchewan, also required the digestion method; a pigment aggregate of 0.05 p.p.m. was obtained. All but about one per cent of the pigment in the total extract was soluble in dilute alkali thereby indicating the presence of free carboxyl groups. The spectra were of the "etio" type.

Two surprising features thus are apparent from the porphyrins occurring in coal: one, that the concentration level is very low, and two, that the pigment is not of the chlorophyll type, a pigment class which might have been expected if coal were believed to arise from plant material. Both observations are probably satisfactorily explained by attributing them to oxidation effects; in the one case causing major destruction of the total pigment, and in the other causing partial destruction of the original chlorophyll-type molecule in which the fifth ring was broken giving rise to a hemin-type pigment.

It is pertinent now to report on the pigments found in modern recent sediments since such sediments are commonly regarded as the initial site of petroleum hydrocarbons. The information summarized in the following sections will be reported more fully in subsequent communications.

MACKENZIE DELTA SEDIMENTS

The sediments of the delta of the Mackenzie River in Arctic Canada are unique in the present study in that they occur in a permafrost condition. Samples were obtained from a core hole drilled below a delta lake near Inuvik, and sediments from 30 feet below the water-sediment contact were examined for porphyrin pigment. Vanadyl and nickel complexes of porphyrins were present in roughly equal portions of about 0.01 p.p.m. of the dried sediments. Both pigments were evidently decarboxylated, since neither showed alkali solubility either before or after saponification attempts. In addition, the deltaic sediments showed the presence of metal complexes of chlorins, with absorption maxima at 650 and 620 mμ. The bulk of this chlorin material was in an esterified state in the sediments, and was more abundant than the porphyrin material, its concentration being about 0.1 p.p.m. There were, also, free pigments, i.e. not metal-complexed, in greater abundance. These were of the pheophytin type, i.e. chlorophylls with the magnesium replaced by two hydrogens. Samples from lower levels in the hole showed similar pigments with some indication of a conversion of esters to free acids.

PEAT

A sample of peat from the Edmonton area showed large amounts of chlorin pigments (750 p.p.m. of the dried weight). These pigments were mainly carboxylated free chlorins, H_2P_cCOOH—530 p.p.m., and their esters, H_2P_cCOOR—210 p.p.m., where P_c is "pigment, chlorin." Carboxylated metal chlorin complexes of the type MP_cCOOH accounted for only 10 p.p.m. The majority of the chlorins appeared to be pheophytin and pheophorbide types. However, a pigment with an adsorption maximum at 720 mμ was also present in appreciable quantities. Metal porphyrin complexes of the type MP_pCOOH were present to the extent of about 0.5 per cent of the total pigment or 3 p.p.m. of the dried weight of the sample studied. Such pigments are well known in marine and fresh-water sediments.

FRESH-WATER SEDIMENTS FROM BIG LAKE, ALBERTA

Fresh-water sediments have been recently examined for pigments (Hodgson *et al.*, 1960; Hodgson and Peake, 1961). The most significant results of this work were the finding of metal complexes of both porphyrins and chlorins in recent sediments. The present study provided additional data, in particular with respect to the porphyrin pigments in the sediments of Big Lake, a few miles northwest of Edmonton, Alberta. Sediments from only 2 feet below the surface of a mud-flat showed metal porphyrins to the extent of about one per cent of the total contained pigments. The porphyrin pigments appeared to be indistinguishable from the vanadyl pigments of petroleum (mainly VOP_p with a trace of VOP_pCOOH). In addition, metal chlorins (principally MP_cCOOH) were present to the extent of about 5 per cent of the total pigment aggregate. The most abundant pigments were free chlorins, H_2P_cCOOH and H_2P_cCOOR.

Five feet below the surface the vanadyl porphyrin pigment class represented about 18 per cent of the total pigment aggregate. There the total pigment was about 0.5 p.p.m. (dried weight), compared with about 0.8 p.p.m. for the sample from the two-foot level.

SEDIMENTS FROM LAKE WABAMUN, ALBERTA

Sediments from immediately below the water-sediment interface in Lake Wabamun, a typical shallow Alberta lake with rapid deposition, showed a metal porphyrin complex of a type not found in the deeper and older sediments of Big Lake. The Lake Wabamun porphyrin possessed a free carboxyl group and appeared to be a precursor of a petroleum-type porphyrin. The spectrum showed absorption maxima at 575 and 400-405 mμ, similar to those of the vanadyl complex of pheoporphyrin. It represented one per cent of the total pigment or 1.4 p.p.m. of the dried weight of sample. Removal of the metal from the complex by hydrobromic and acetic acid digestion revealed a porphyrin with a rhodo-type spectrum similar to that of pheoporphyrin, a compound believed to be intermediate between chlorophyll and deoxophylloerythroetioporphyrin (Hodgson and Peake, 1961). The pheoporphyrin-like material was the only porphyrin to be detected at the sediment-water interface; however, larger quantities of other pigments were present.

The total pigment content of the Lake Wabamun sediment was 145 p.p.m. with approximately 135 p.p.m. being free chlorins and

8.1 p.p.m. metal chlorin complex. The majority of the free chlorin was esterified with only 14 per cent or 20.6 p.p.m. containing a free carboxyl group. These pigments are typical of sediments and have been described previously (Hodgson *et al.*, 1960; Hodgson and Peake, 1961).

MARINE SEDIMENTS, MATSUSHIMA BAY, JAPAN

Marine sediments have been examined on many occasions for pigments related to chlorophylls. For example, Krejci-Graf (1936) reported "chlorophyll" contents of Black Sea sediments per 100 gm. of mud as follows: in gyttja and gyttja-clays, 0.02 to 3.31 mg.; calcareous sapropel (deep-sea), 11.91 mg.; black clayey sapropel (deep-sea), 74 to 99 mg. Orr, Emery and Grady (1958) reported similar pigment contents in marine muds off the coast of California. Recently the pigments in marine muds from Matsushima Bay on the east coast of Japan were examined more fully. Matsushima Bay is a shallow restricted marine bay near Sendai, Japan. Detailed information on its physical environment and the pigment content of its sediments has been reported in another publication (Hodgson *et al.*, 1963). The same variety of chlorin pigments was observed for marine sediments as for the fresh-water sediments. In a survey of sediments from various locations in the bay, the total pigment content varied from 1.6 to 75 p.p.m. of the sediments of which 75 to 95 per cent was H_2P_cCOOR. Spectral characteristics tended to support identification of the pigment as pheophytin *a*. Five to fifteen per cent of the total pigments were of the class H_2P_cCOOH—probably pheophorbide *a*. Metal chlorin complexes were observed in quantities about 3.1 per cent of the total pigments or about 0.5 p.p.m. of the sediments. There was also indication of metal porphyrins in the sediments consisting of vanadyl and nickel complexes with the former more predominant. The total content of these metal porphyrins amounted to about 0.005 p.p.m. of the sediments, perhaps less than 10 per cent of the general class MPCOOH. In similar samples from a nearby unrestricted bay—Ishinomaki Bay—the nickel porphyrin complexes appeared to be more abundant than vanadyl complexes. An attempt was made to find two other classes of pigments—esters of carboxylated pigments and decarboxylated pigments. Findings indicated the presence of a chlorin metal complex of the MP_c type and traces of chlorin MP_cCOOR (other than chlorophyll). Further investigations of these sediments during the present study confirmed the above findings.

"VERY RECENT" SEDIMENTS

The term Recent sediments is usually taken to mean sediments that are as old as a few thousand years. Chemically and biologically this term therefore includes a very wide range of conditions. The results in the two foregoing sections applied to both buried and interface sediments. It is pertinent to consider now what might be termed as "very recent" sediments. Two samples were examined: one, an organic fertilizer prepared from treated sewage; the other, material filtered from the North Saskatchewan River by the City of Edmonton water-supply filtration plant.

The treated-sewage fertilizer is distributed by Sturgeon Processors Ltd., Edmonton, Alberta, under the trade name "Organite." It showed the presence of free chlorins and metal complexes of chlorins and porphyrins. Pigments similar to those in recent sediments were present at concentrations up to 5 p.p.m. of the dried sample weight. The two general classes of pigments consisted of H_2P_cCOOH and H_2P_cCOOR constituting the free chlorins, and MP_cCOOH and MP_cCOOR or MP_c the metal complexes. The predominant pigment was H_2P_cCOOR at a concentration level about 5 p.p.m. followed by H_2P_cCOOH at just below 1 p.p.m., MP_cCOOR, 1 p.p.m. and MP_cCOOH, 0.3 p.p.m. There was an indication of a metal porphyrin complex with a spectral absorption maximum at 570 mμ, which was due to either MP_p or MP_pCOOR. It was present in the treated sewage in quantities about one tenth that of the metal chlorin complexes.

The sediment from the filtration plant was made up of very finely divided clay minerals which had been suspended in the river water. Acetone extractions of this sediment showed the presence of the usual suite of recent sediment pigments. Free chlorin pigments were observed at a concentration level between 0.1 and 1.0 p.p.m. and metal complexes of chlorins were present at approximately the same level. The evidence for metal porphrin complexes was not conclusive, but there was some indication that this material was present.

LAKE WATER

After considering ancient sediments, recent sediments and "very recent" sediments, it is now pertinent to discuss the pigments in the waters associated with the sediments.

Prophyrin and chlorin pigments are not very soluble in water, even when they have free carboxyl groups. Water taken from Big Lake showed metal chlorin complexes of 0.002 p.p.m. There was an unconfirmed indication of metal porphyrins as well, at a very much lower concentration. A more extensive examination was made of water from Lake Wabamun, a larger lake in the Edmonton area. The following compound classes, in addition to chlorophylls, were observed.

H_2P_cCOOR	0.000,04 p.p.m.
MP_cCOOR	0.000,08 p.p.m.
$H_2P_cCOOH + MP_cCOOH$	0.000,02 p.p.m.

Although the Lake Wabamun water was obtained from below a thick ice cover in late winter, there was still evidence of suspended mineral matter. The question arises as to whether the pigments were in true solution or were merely associated with the suspended matter.

SPRING WATER

It was of interest to consider whether pigments are carried in migrating ground waters. An examination was made of the water from a spring from a shallow groundwater horizon in an escarpment of the North Saskatchewan River near Edmonton. A content of chlorin pigments of about 0.0001 p.p.m. was observed, but again, whether these pigments were in true solution or were carried on particulate matter was not clear. There was some indication that the pigments were carried in association with iron salts which were being deposited at the site of the spring.

PLANTS

In the present study, two kinds of plants—a red marine alga and spinach—were examined for pigment content. Data from a previous study of a seaweed, *Chaetomorpha crassa* (Ag.) kg., were also available (Hodgson *et al.*, 1963). All of these plants contained large quantities of chlorins and some metal chlorins of the same types as found in recent sediments — H_2P_cCOOH, H_2P_cCOOR, MP_cCOOH, and MP_cCOOR. The metal chlorins were evidently the same as those reported earlier for fresh-water sediments by Hodgson and Peake (1961). The free chlorins were present in the seaweed to the extent of 1500 p.p.m. whereas metal chlorins represented less than 2 p.p.m. Reports of metal porphyrins and a decarboxylated metal chlorin, MP_c (Hodgson *et al.*, 1963), were not confirmed.

DISCUSSION

Some of the foregoing sections have dealt with petroleum pigments in crude oils, and have drawn attention to the position of the oil of the Athabasca oil sands in the general spectrum of crude oils. It is clearly near one end of the spectrum. Further, the Athabasca oil has been compared with oil in the other oil sands, and a direct relationship between the content of vanadyl pigments and sulphur in these oils has been noted.

The presence of porphyrin and chlorin pigments in environments closely related to the formation of crude oil has been examined as summarized in table 8. No porphyrin pigments were found in Precambrian rocks. Most other samples showed the presence of porphyrin pigments. The content of the pigments in some environments was extremely low—about 0.001 p.p.m. for chlorins in natural waters—and in others very high—about 1500 p.p.m. in seaweed.

TABLE 8. SUMMARY OF CLASSES OF PIGMENTS FOUND

	Metal Complexes			Free pigments
	Vanadyl	Nickel	Metal uncertain	
Petroleum	VOP_p, VOP_pCOOH	NiP_p, NiP_pCOOH		
Athabasca oil-sand oil	VOP_p, VOP_pCOOH, VOP_pCOOR	NiP_p, NiP_pCOOH		
Melville Island oil-sand oil	VOP_p			
California oil-sands oils	VOP_p	NiP_p		
Low sulphur oil-sands oils	VOP_p	NiP_p		
Lower Cretaceous shales	VOP_p	NiP_p		
Ireton shale	VOP_p	NiP_p		
Coal	*	*		
Mackenzie-delta sediments	VOP_p, VOP_cCOOR	NiP_p, NiP_cCOOR		H_2P_cCOOR
Peat			MP_pCOOH, MP_cCOOH†	H_2P_cCOOH, H_2P_cCOOR
Big Lake sediments	VOP_p, VOP_pCOOH		MP_cCOOH††	H_2P_cCOOH, H_2P_cCOOR
Lake Wabamun sediments			MP_pCOOH, MP_cCOOH	H_2P_cCOOH, H_2P_cCOOR
Matsushima Bay sediments	VOP_pCOOH	NiP_pCOOH	MP_cCOOH, MP_c	H_2P_cCOOH, H_2P_cCOOR
"Very Recent" sediments			MP_pCOOR or MP_p, MP_cCOOR or MP_c, MP_cCOOH	H_2P_cCOOH, H_2P_cCOOR
Lake water			MP_cCOOH, MP_cCOOR	H_2P_cCOOH, H_2P_cCOOR
Spring water	**	**	**	**
Plants			MP_cCOOH, MP_cCOOR	H_2P_cCOOH, H_2P_cCOOR

*Probably both VOP_p and NiP_p classes of pigments present; H_2P_p found, after HBr-HOAc digestion.

**Chlorin pigments were present, but class identity not clear.

†MP_p, metal pigment porphyrin.

††MP_c, metal pigment chlorin.

It is important now to try to establish a perspective for the position of the pigments in the origin of crude oil. While it has been customary to attribute the porphyrin pigments of crude oil to the natural pigments of living organisms, care must be exercised in choosing the specific starting pigments. The presence of the chlorophyll-type structure in the petroleum pigments suggests that a chlorophyll, e.g. chlorophyll *a*, might be the initial pigment, and the abundance of this compound in nature strengthens the suggestion. If chlorophyll *a* were assumed to be the initial pigment, a sequence of simple chemical reactions such as the following would account for the development of petroleum pigments and would therefore define to some considerable degree the chemical environment of the genesis of petroleum:

demetallation — removal of magnesium from chlorophyll *a*
saponification — breakdown of esters
hydrogen transfer — vinyl saturation and aromatization
reduction — oxygen removal
decarboxylation — removal of -COO- groups
metal complexing — addition of vanadyl or nickel.

The compounds found in the various environments pertinent to the genesis of petroleum direct attention to one outstanding feature. The various environments would be expected to contain intermediate pigment compounds related to the foregoing sequence, and while the metal complex of pheoporphyrin was indicated, the most striking feature was that metal complexes of pigments indistinguishable from vanadyl chlorins occur in all of the early environments, most significantly in the structure of living plants. This focusses attention on the possibility of vanadyl porphyrins of petroleum arising from compounds in plants other than chlorophylls. While the role of such pigments in plants is obscure at this time, it is possible that vanadyl pigments are components of enzyme systems. The sequence of environmental reactions for vanadyl compounds thus is simplified to:

hydrogen transfer — vinyl saturation and aromatization
reduction — oxygen removal
decarboxylation — removal of -COO- groups

There is unconfirmed evidence for the presence of vanadyl porphyrins in addition to vanadyl chlorins in living matter contributing to the source of crude oil, and if the presence of these is established, the sequence of reactions for the evolution of petroleum vanadyl pigments is further simplified to two processes—reduction and decarboxylation. There is evidence to attribute the decarboxylation step to the reservoir environment (Hodgson *et al.*, 1962), leaving only the reduction step for the recent sediment environment.

In any case, the most difficult hurdle in accounting for the presence of vanadyl pigments in petroleum is evidently overcome, by noting that the complexing of the vanadyl ion with the pigment is not necessarily a reaction of the genesis environments, but rather a prior reaction in the plants. This has been an interesting parallel with the fact that significant quantities of hydrocarbon material are available in plant organisms before reaction in the genesis environments takes place, supporting the current theories of Baker (1962) and Meinschein (1962) in which emphasis is placed upon the simple preservation and collection of hydrocarbons existing in the plants prior to deposition.

On the other hand, there is no evidence for the presence of nickel complexes in either the very early genesis environments (water and very recent sediments) or in plants. The entire sequence of reactions listed initially appears to be required to explain the presence of nickel porphyrins in crude oil. However, the complexing of nickel with pigments is accomplished much more readily than the corresponding vanadyl reaction and presents no particular obstacle in the sequence (Baker and Hodgson, 1961). The later environments of genesis for marine sediments apparently show the presence of nickel porphyrins, indicating that demetallation, hydrogen transfer, reduction and metal complexing have taken place. This is apparent, however, only for the off-shore sediments, and not for the restricted-bay sediments.

Thus, the development of vanadyl and nickel pigments of petroleum appear to start from different points. Evidently the vanadyl pigments are derived from vanadyl pigments of living organisms, while the nickel pigments come from chlorophyll *a* or some closely related pigment. The relative abundances of vanadyl and nickel pigments in crude oil might therefore be expected to reflect the nature of the environments of genesis of the oils. Perhaps the conditions affecting the development of nickel pigments might be the most subject to chemical control from the environment because of the longer sequence of reactions involved for the nickel than for the vanadyl system. Specifically, attention might be directed to the complexing reaction and its sensitivity to pH, since saponification and demetallation of chlorophyll *a* present no serious obstacles (Hodgson and Hitchon, 1959). Metal complexing reactions for the pigment compounds are always favoured by low pH conditions; conversely, they are suppressed by high pH environments. Restricted-bay sediments are marked by pH values generally higher than those in the corresponding open-sea conditions, and there is evidence that nickel porphyrin pigments tend to dominate in the latter, in keeping with the suggested controlling factor of pH (Hodgson *et al.*, 1963).

Further, a restricted-bay environment (Matsushima Bay, Japan) exhibited a more severe reducing condition, and it might be expected that such an environment may therefore not only suppress the formation of nickel complexes of porphyrins leading to a dominance of vanadyl pigments, but also favour the introduction of sulphur into the developing crude oil through the reduction of sulphates by sulphate-reducing bacteria to hydrogen sulphide which reacts readily with organic debris. The suggested reaction paths thus appear consistent with the facts for living organisms and sedimentary environments. For example, the Athabasca oil is one in which the vanadyl pigment is predominant accompanied by a high sulphur content which probably arose from a highly reducing environment of deposition. The low-sulphur oils, as would be expected, are marked by a dominance of nickel porphyrin.

It is important to touch upon the migration of hydrocarbons and associated pigments. Undoubtedly, such migration does occur and probably in a migrating-water system. Although the data at hand are little more than exploratory in nature, one might expect the composition of a migrating mixture of hydrocarbons to be affected by the nature of the starting material, the components adsorbed by mineral surfaces during migration of the oil-water system, and the components accumulated by the migrating system. There are indications that the nature of the starting material in the sediments may be very dominant. This matter has been discussed by Hitchon in a companion paper in the present volume (p. 71). Hitchon's expression of basin-shelf control on the nature of the surviving organic matter is consistent with the evidence of oceanographic studies (Emery, 1963) and draws attention to the extensive destruction of organic material being deposited well offshore, while the near-shore material in protected areas is well preserved in the water column and subsequent sediments.

The cases for loss of pigments to the sediments and for the accumulation from the sediments during migration of the oil-water system are not well defined. The former, usually referred to as natural chromatography, has been advanced by Bonham, (1956), Hodgson *et al.* (1959), and Nagy *et al.* (1961), but no unequivocal examples have been brought forward. The effect of pigment accumulation during the water-hydrocarbon migration has been studied even less. While it has been established that the generation of pigments during migration is unlikely (Park and Dunning, 1961), little or no consideration has been given to the possibility of accumulation of pigments from the sediments during migration. However, in the present study evidence has evolved to show that suspended solids in natural waters carry significant quantities of pigments, and finely divided solids in oil-sand accumulations have remarkably high pigment contents. All of this is in keeping with the findings of Witherspoon and Nagashima (1957) who found that the metal and pigment-containing components of crude oil were closely associated with material in the oil recovered by ultracentrifuging. On the other hand, the proportion of the total pigment of the Athabasca oil accounted for in this manner appears to be quite small. Probably, the mode of entry of pigments into the developing crude oil is through continued intimate association of the pigments and organic matter from the original point of deposition through to the final point of accumulation, with the pigment and hydrocarbon components being subjected to essentially the same adsorption and accumulation factors. However, if there were a difference in the behaviour of the vanadyl and nickel pigments, the vanadyl pigments would tend to be lost more readily from a migrating system by adsorption processes, as exemplified by commonly used laboratory chromatographic separations, leading to a dominance of the nickel pigments. For accumulation processes, migration through a sediment rich in vanadyl pigments (characteristic of a near-shore, highly reducing sediment) would favour a dominance of vanadyl pigments over nickel pigments in the final crude oil.

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COMPOSITION OF ATHABASCA BITUMEN FRACTIONS AS DETERMINED BY STRUCTURAL-GROUP ANALYSIS METHODS

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ABSTRACT

The bitumen contained in an 11-kilogram sample of Abasand bituminous sand was separated into a series of fractions by solvent extraction, and subsequent elution chromatography on fuller's earth and silica gel. The chemical structure of twelve of these fractions has been studied. A quantitative estimate of the number of methyl and methylene groups has been made from the infrared spectrum. The aromaticity has been determined by van Krevelen's graphical densimetric method for the asphaltene and resin components and by the n-d-M method for the oil components. A method of structural-group analysis developed by the authors has also been applied. In this case, the composition has been expressed in terms of five hydrocarbon groups. This method consisted of a correlation of the following properties: the elementary composition, molecular weight, density (20°C), refractive index (n_D^{20}), and the aromaticity. It has been shown that the composition of these fractions varies from a colourless, saturated oil of molecular weight 360 containing three rings per average molecule, to a black-brown solid of molecular weight 2500, in which half of the carbon atoms were aromatic and whose average molecule contained 23.6 rings.

INTRODUCTION

This paper summarizes the results of the determination of the hydrocarbon skeletal structure of Athabasca bitumen fractions largely by means of structural-group analysis methods.

The bituminous sand used in this investigation was an 11-kilogram sample taken from a 150-ton shipment from Abasand Oils Limited, Fort McMurray, Alberta, which contained 15.8 per cent bitumen by weight.

PREPARATION AND PROPERTIES OF FRACTIONS

The bitumen was fractionated in the following manner. An 11-kilogram sample of bituminous sand was first extracted with *n*-pentane, followed by an extraction with carbon tetrachloride. The latter extract was defined as the asphaltenes. The pentane extract was then separated into oil and resin components by chromatography on Florex. The resin component was separated into six fractions by elution from the Florex with a series of increasingly polar solvents. The oil component was further separated into nine fractions by elution chromatography on silica gel by using methylcyclohexane and toluene as eluants. A quantitative summary of the preparation of these fractions is shown in figure 1, the quantities being expressed as percentages of the bitumen.

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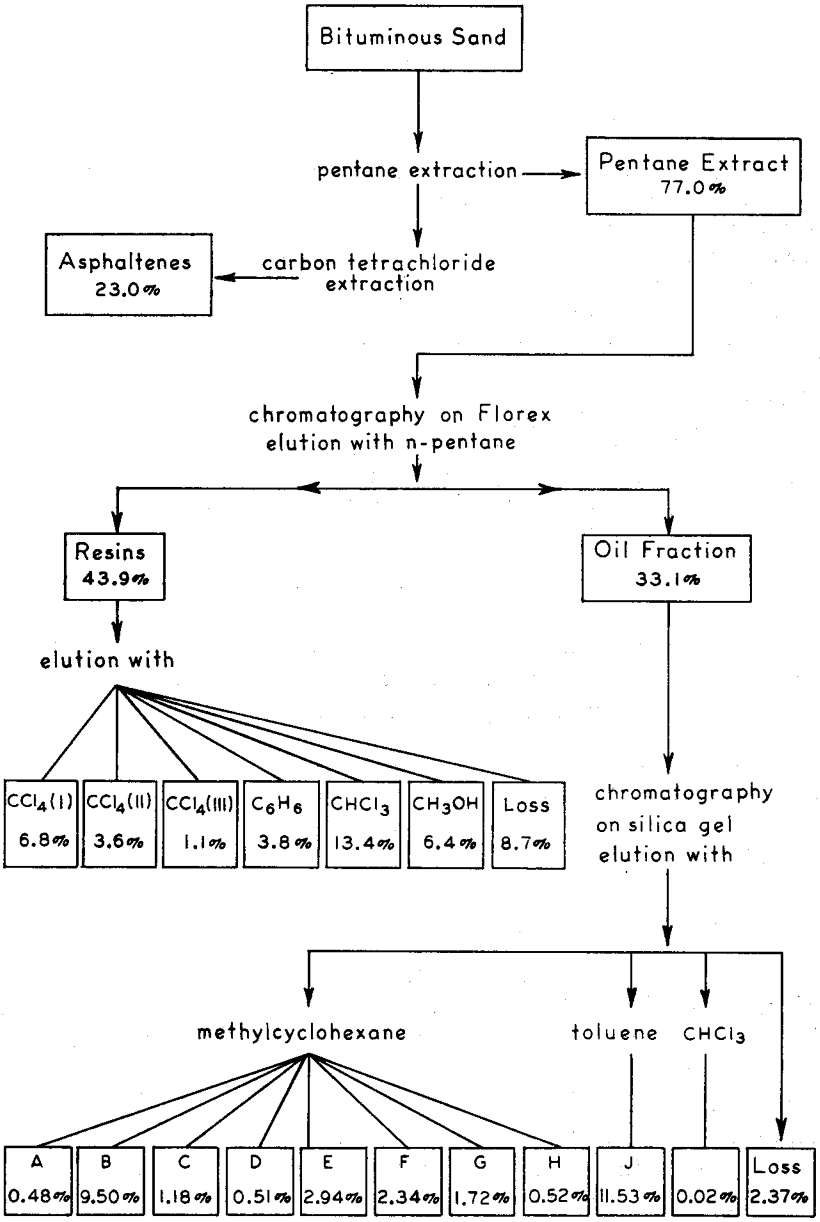

FIGURE 1. *Summary of steps in the preparation of bitumen fractions (expressed as percentages by weight of the bitumen content).*

TABLE 1. PROPERTIES OF BITUMEN FRACTIONS

Fraction	Ash (%)	C* (%)	H (%)	S (%)	N (%)	O (diff.) (%)	Molecular Weight	d^{20}	n_D^{20}
A	—	—	—	—	—	—	363	0.8747	1.4777
B	—	86.46	13.35	0.07	—	0.12	360	0.8860	1.4830
C	—	86.19	13.24	0.14	—	0.43	317	0.9009	1.4877
D	—	84.43	12.09	1.09	—	2.39	643	0.9263	1.5044
E	—	85.78	12.12	1.66	—	0.44	582	0.9247	1.5139
F	—	84.74	11.59	2.07	—	1.60	431	0.9420	1.5236
G	—	85.50	11.21	2.32	—	0.97	369	0.9575	1.5319
H	—	—	—	—	—	—	341	0.9677	1.5387
J	—	86.09	10.39	4.86	—	0.0	340	0.9905	1.5512
CCl_4(I)	0.03	83.68	9.44	6.23	—	—	519	1.058	—
CCl_4(II)	0.30	81.60	9.17	6.90	0.72	1.61	592	1.060	1.594
CCl_4(III)	0.06	—	—	6.06	—	—	577	1.052	—
C_6H_6	0.62	80.18	8.42	6.87	0.66	3.87	821	1.116	1.631
$CHCl_3$	1.19	79.17	9.31	6.73	0.95	3.84	976**	1.085	1.612
CH_3OH	2.46	77.42	9.19	6.96	1.02	5.41	834**	1.085	1.609
Asphaltenes	2.45	79.01 6.6	7.98	8.07	1.01	3.93	2492**	1.158	1.665

*The elementary analyses refer to the "ash-free" basis.

**These molecular weights were determined cryoscopically in phenanthrene, the remainder cryoscopically in benzene

For convenience, the fractions eluted from the Florex are referred to by the name of the solvent with which they were eluted and the fractions eluted from the silica gel have been designated alphabetically.

The properties of these fractions are tabulated in table 1.

CHARACTERIZATION OF THE BITUMEN FRACTIONS

Some quantitative information about the saturated hydrocarbon groups has been obtained by Goodspeed and Montgomery (1962) from the infrared spectra of these fractions. They were able to determine quantitatively the number of methyl and methylene groups from the absorption bands at 7.28 and 3.42 microns. These results are tabulated in table 2 since they have been used in conjunction with the Montgomery-Boyd structural analysis method in establishing the structure.

TABLE 2. DETERMINATION OF METHYL AND METHYLENE GROUPS FROM INFRARED DATA

Fraction	No. of Carbon Atoms per Average Molecule	
	$CH_3 (7.28\mu)$	$CH_2 (3.42\mu)$
B	5.7	15.3
C	4.6	12.3
D	9.0	25.1
E	7.6	21.3
F	5.5	13.5
G	4.6	10.8
J	4.1	8.3
CCl_4(II)	7.0	13.3
C_6H_6	10.0	16.5
$CHCl_3$	12.2	24.2
CH_3OH	10.8	17.3
Asphaltenes	24.6	—

Since the aromaticity was a required property to apply the structural-group analysis method developed by the authors (Montgomery and Boyd, 1959), this property was determined by the van Krevelen graphical densimetric method (van Krevelen and Schuyer, 1957) for the asphaltenes and resins and by the n-d-M method (van Nes and van Westen, 1951) or its modifications for the oil fractions. These values are given in table 3.

The principal means by which the hydrocarbon skeletal structure was determined was the application of the structural-group analysis method. By this method, it was possible to determine the number per average molecule of each of the following five hydrocarbon groups: paraffinic carbon atoms (C_1); non-junction saturated ring

TABLE 3. DETERMINATION OF AROMATICITY

Fraction	C_a (%)	Method
B	0.0	n-d-M^1
C	0.0	n-d-M^1
D	5.3	n-d-M-Karr^2
E	17.2	n-d-M-Karr^2
F	22.6	n-d-M-Karr^2
G	26.4	n-d-M-Karr^2
J	33.4	Hazelwood - Karr^{2,3}
CCl_4(II)	47.0	Graphical densimetric method^4
C_6H_6	51.0	Graphical densimetric method^4
$CHCl_3$	39.0	Graphical densimetric method^4
CH_3OH	40.0	Graphical densimetric method^4
Asphaltenes	52.0	Graphical densimetric method^4

[1] van Nes and van Westen (1951)
[2] Karr, Wendland and Hanson (1962)
[3] Hazelwood (1954)
[4] Krevelen and Schuyer (1957)

carbon atoms (C_2); saturated ring carbon atoms in ring junction positions (C_3); non-junction aromatic ring carbon atoms (C_4); and aromatic ring carbon atoms in ring junction positions (C_5). The manner in which this method is applied to this type of material has already been reported (Boyd and Montgomery, 1962a). The determination of these groups is made by the simultaneous solution of five equations; representing three chemical and two physical properties, namely, the total number of carbon atoms per molecule, the total number of

TABLE 4. RESULTS OF THE MONTGOMERY-BOYD STRUCTURAL ANALYSIS
(expressed as number of carbon atoms of a given type per average molecule)

Fraction	C_1	C_2	C_3	C_4	C_5	R*
B	14.5	7.5	3.9	— 0.2	0.2	3.0
C	10.2	8.8	3.9	— 0.1	0.1	3.0
D	29.2	4.8	1.9	6.9	2.3	3.1
E	26.1	5.7	1.4	5.4	3.3	3.3
F	17.5	5.2	0.7	3.8	3.4	3.0
G	12.3	6.2	0.8	3.5	3.6	3.2
J	7.7	7.2	1.1	4.0	4.4	3.8
CCl_4(II)	15.2	5.4	2.0	10.8	6.9	5.6
C_6H_6	18.2	5.8	3.7	16.2	10.5	8.0
$CHCl_3$	29.7	7.1	3.1	12.8	11.3	8.2
CH_3OH	27.2	3.7	1.4	12.8	8.6	6.0
Asphaltenes	51.0	11.4	16.8	55.4	27.6	23.6

*Number of rings per average molecule, calculated from $C_3+C_5=2R-2$

hydrogen atoms per molecule, the total number of aromatic carbon atoms per molecule, the molar volume and the molar refraction. The calculations were made on an IBM 1620 Data Processing Machine. The results of this analysis are shown in table 4.

Before considering these results, it seems desirable to consider the assumptions and approximations which were made in obtaining them. Since this method is only applicable to a hydrocarbon, it was necessary to correct the molar volume and the molar refraction to a "foreign-atom-free" basis. The method (Boyd and Montgomery, 1961) consisted essentially of "removing" the foreign atoms without affecting the structural contributions to the molar volume and molar refraction and without altering the number of carbon and hydrogen atoms. This method was considered adequate on the basis of the small number of foreign atoms involved (atomic per cent < 4.0) and the accuracy of the structural analysis method itself.

The number of aromatic carbon atoms per molecule is not readily determined experimentally with sufficient accuracy for bituminous materials. By using the following technique, it was possible to define the structure within certain limits. Values of C_1 to C_5 were determined for various arbitrarily chosen values of the aromaticity considered likely on the basis of the calculated values in table 3. These various analyses were assessed, and structural limits were set for each fraction on the basis of the following assumptions: (1) all physically impossible analyses were disregarded (i.e. all negative results); (2) the number of cyclic carbon atoms per ring was 4.0; and (3) the sums of CH_3 and CH_2 groups as determined from the infrared spectra (Table 2) were equal to C_1+C_2 or $C_1+C_2+C_3$. The limits assigned to each fraction expressed in terms of the aromaticity varied from a maximum of 10 per cent for the asphaltenes, 6.5 per cent for the resin fractions and 5 per cent for the oil fractions. Fractions A, B, and C were assumed to contain no aromatic carbon on the basis of their infrared spectra (Boyd and Montgomery, 1962b). The results given in table 4 are the arithmetic means of the two limiting compositions for each fraction.

For the asphaltene and resin fractions, a modification of this method (Boyd and Montgomery, 1962a) in which C_2 and C_3 were assumed present in negligible quantities permitted a determination of the number of aromatic fused ring (C_5) and aromatic non-fused ring (C_7) junction carbon atoms. The results of this second analysis are given in table 5. The ratio C_5 to C_7 may be taken as an indication of the relative proportion of fused aromatic rings to non-fused aromatic rings.

TABLE 5. DETERMINATION OF THE DEGREE OF CONDENSATION OF THE AROMATIC RINGS IN THE RESIN AND ASPHALTENE FRACTIONS

Fraction	C_5	C_7	C_5/C_7
CCl_4(II)	3.0	4.4	0.68
C_6H_6	6.1	5.9	1.03
$CHCl_3$	7.4	4.7	1.57
CH_3OH	6.5	2.5	2.60
Asphaltenes	20.9	16.7	1.25

DISCUSSION

The fractions into which the bitumen has been separated range from a totally saturated colourless oil of molecular weight 360, which comprised 10 per cent of the bitumen, to the brown-black solid asphaltene fraction with a molecular weight of 2500 where approximately 50 per cent of the carbon atoms were aromatic and which constituted 23 per cent of the bitumen.

The oil fractions (one third of the bitumen) range from totally saturated fractions through a series containing gradually increasing proportions of aromatic carbon to fraction J which was 34 per cent aromatic. All of the oil fractions appeared to contain an average of three rings per molecule.

The resin fractions (44 per cent of the bitumen), with molecular weights in the range 592 to 976, contained 37 to 49 per cent aromatic carbon atoms and 6 to 8 rings per molecule. The most convenient basis upon which to compare these fractions is the ratio C_5 to C_7 (Table 5). This ratio increased in the order in which the fractions were eluted from the Florex, from a value of 0.68 for the CCl_4(II) fraction to 2.60 for the methanol fraction. This would suggest that the proportion of fused aromatic rings was increasing compared with the number of non-fused aromatic rings in the direction CCl_4(II) to methanol.

The most striking fact about the analysis of all fractions was the high proportion of saturated carbon groups present in all fractions, and in particular the large portion of the saturated carbon atoms present in paraffinic form and the comparatively small amount of carbon present in the form of cyclic saturated groups. To illustrate this feature, the percentages of paraffinic carbon atoms (C_1) in the saturated portion of the molecule for all fractions has been tabulated in table 6. These figures would seem to indicate that oil fractions B, C and J are the only ones containing a significant number of saturated rings.

A second fact concerning the saturated part of the molecule was obtained by assuming that the number of methyl groups (Table 2) represents the maximum number of linear side chains possible. The average chain-length was calculated as C_1/CH_3. These values are included in table 6. There is very little variation in these figures, the average value for all fractions being 2.5.

TABLE 6. CHARACTERISTICS OF SATURATED PORTION OF THE MOLECULE

Fraction	$C_1/(C_1+C_2+C_3)\times100$	C_1/CH_3
B	56.0	2.5
C	44.7	2.2
D	81.3	3.2
E	78.6	3.4
F	74.8	3.2
G	63.7	2.7
J	48.1	1.9
CCl_4(II)	66.5	2.2
C_6H_6	65.0	1.8
$CHCl_3$	74.4	2.4
CH_3OH	84.2	2.5
Asphaltenes	64.4	2.1

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SHEARING STRENGTH CHARACTERISTICS OF ATHABASCA OIL SANDS

R. M. HARDY* AND R. A. HEMSTOCK**

ABSTRACT

Knowledge of the *in-situ* shearing strength of oil sand is essential in mining Athabasca oil by open-pit mining methods.

The physical properties of the oil sand are examined firstly in terms of source, deposition, composition and oil impregnation and secondly in terms of the components mineral matter, oil, water, and gas.

Measurement of the shearing strength of cores showed that on release of stress gas comes out of solution in the oil phase. Gas pressure in the pores increases with increasing temperature of the oil sand, and the oil phase, due to its high viscosity, has the effect of delaying the dissipation of the gas pressure. Reduction in strength due to gas pressure is largely recovered if the gas pressure is dissipated.

Under *in-situ* conditions the oil sands tested had strength deformation characteristics consistent with those of a dense sandstone.

The conventional concepts of effective stress as used in soil mechanics appear to be completely applicable to the determination of the strength of oil sands. These concepts require that the pore pressures of all fluids in the soil be numerically evaluated, but as yet quantitative relationships have not been evaluated between magnitudes of pore pressure and stress release nor for the rate of gas pressure dissipation.

INTRODUCTION

IMPORTANCE OF SHEARING STRENGTH

This paper deals with the studies completed to date to establish the shearing strength characteristics of Athabasca oil sands to the extent that these are related to the economic development of Lease 17 at Mildred Lake by Cities Service Athabasca, Inc. Knowledge of the *in-situ* shearing strength of oil sand is essential in winning Athabasca oil by open-pit mining methods. Mining methods will in part be dictated by the strength of the formation. If benches are used in an open pit, they must be capable of supporting mining equipment without danger of either local or deep-seated shearing failures. The shear strength of the material will also affect the power required for the actual digging of the oil sands, or alternatively the effectiveness of a given weight of explosive if blasting techniques are used.

Pit walls in certain locations will be over 200 feet high. There must be no danger of sliding failures along the active mining face because of hazards to personnel and equipment. The long-term side

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slopes must also be stable, at least until the pit is backfilled with tailings. The safe slopes to which faces of such heights can be cut, both for short- and long-term periods, are dependent on the shear strength characteristics of the oil sand, and these slopes, of course, are of considerable economic importance in the mining operations.

The safe slope of either the mining face or the pit sidewalls has a direct effect on the design of the mining equipment and hence on the economics of the operation. If flat slopes are necessary, more of the mineable material will have to be left in place and much greater "reach" will be necessary in the mining machine for a given height of embankment.

In the mining operation there will be occasions when equipment will travel over, or perhaps remain on, *in-situ* oil sand. Either the strength of the material must be sufficient to support the equipment directly or roads or fills must be constructed on the oil sand in such a way that the safety and reliability of the operation is assured. In either case a knowledge of the shear strength of the oil sand under conditions of surface exposure as well as at depth is essential.

Finally the extraction and refining equipment and ancillary facilities such as power plants will require heavy and stable foundations. Since the whole area is underlain by the McMurray Formation, the strength of this material will have to be known to design these foundations properly.

PHYSICAL PROPERTIES OF OIL SAND

Unless otherwise noted, the work reported here is based on samples taken from the McMurray Formation adjacent to the Athabasca River on Lease 17, about 20 miles downstream from the town of Fort McMurray. In this area, overburden thickness varies from 0 to 100 feet and the oil sands range from 35 to 200 feet in thickness.

Physical properties of the oil sand are most easily understood if they are examined first from the standpoint of geological history in terms of source, deposition, composition and oil impregnation, and secondly in terms of the components which presently are parts of the oil sand which include minerals, oil, water and gas, and with particular reference to the inter-relation of these *in-situ*. The geological history has been described in detail by various authors (McLearn, 1917; Ells, 1932; Sproule, 1938, 1951; Williams, 1949; Falconer, 1951; Hume, 1951; Kidd, 1951; Link, 1951; Montgomery, 1951; Corbett, 1955; Mellon and Wall, 1956; Carrigy, 1959).

From the standpoint of the physical properties the most important feature is that sometime between the deposition of the McMurray (Cretaceous) and Pleistocene deposits, oil migrated into the water-wet McMurray Formation. During the oil sand's geological history there may have been a load of up to 4,000 feet of sediments which covered the McMurray Formation in this area prior to middle and later Tertiary erosion (Corbett, 1955). There was also some unknown ice load during glacial periods.

It is logical then to expect that the McMurray sands will exhibit physical properties up to failure similar to other sandstones with the same grain-size distribution and in a like state of consolidation. The sand grains are in grain to grain contact as in normal sandstone. Each sand particle is surrounded by a water film and the remaining void space is filled with water, oil and a gas phase in varying proportions.

The mineral matter in the McMurray sands is 95 to 98 per cent uniform white quartz sand. The remainder is essentially clay with minute quantities of associated minerals. Typical grain-size analyses are shown in figure 1, together with a curve of dune sand from Imperial Valley in California to illustrate the similarity of grain size to present-day dune sands. These sands which contain most of the oil are interlayered with fine silt or clay lenses, and there are occasional lenses of hard dense siltstone which contain little or no oil. These hard layers contribute little to the overall stability of the McMurray strata, but their existence must be considered in mining and stripping.

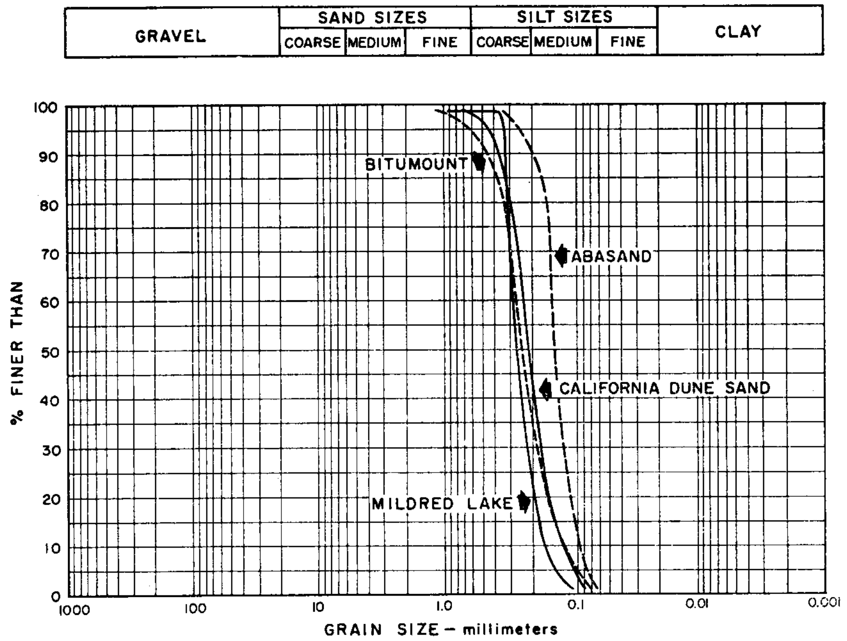

FIGURE 1. Grain-size curves of three typical samples of the Athabasca oil sands and a sample of dune sand from California.

To study the oil-sand skeleton, a small piece of fresh oil sand was frozen, and the oil extracted by means of solvents while the sample was still frozen. The removal of the oil left a very friable white sandstone. Similar clean sands are visible as outcrops at several locations in the McMurray area and in one case (Carrigy, 1959, p. 60) clean white sands may be seen standing at a steep angle and overlain by oil-impregnated sands.

Void ratios measured on undisturbed sand vary from 0.55 to 0.8. The voids are filled with water, oil and gas. Water is nearly fresh in the upper layers and becomes more saline at depth. Oil at Mildred Lake is about 8° A.P.I. and at the formation temperature of 41°F (5°C) it is a very viscous material (about 13.0×10^6cp). The oil content varies from zero to a maximum of about 20 per cent based on the total weight of sample. Sand with less than 6 per cent oil has been arbitrarily defined as "lean" oil sand. Rich oil sand averages about 12 per cent oil. Laboratory work has confirmed that gas is present in much of the oil-impregnated McMurray sands. It fills the unsaturated void space and may also be dissolved in the oil. Analyses show that the gas contains nitrogen, carbon dioxide, methane and traces of other light hydrocarbons.

Permeabilities of undisturbed oil sand have been measured by Dr. K. A. Clark (1959) and he has shown that the permeability of massive good grade oil sands generally averages about 50 millidarcys. These results have been confirmed by other laboratory work. However, there is still some concern that the permeabilities measured may have reflected changes in the samples caused by release from reservoir environment. It is certain, however, that there is significant overall permeability in the oil sands because water flows into core holes in the McMurray Formation. Much of this seepage occurs in zones of low oil saturation, but it also probably occurs through joints or fractures in the formation generally.

EXPERIMENTAL DATA

SHEARING STRENGTH TESTS

The shearing strength characteristics of the oil sand have been studied using the techniques and nomenclature of the field of soil mechanics. It is beyond the scope of this paper to describe in detail the test procedures and equipment used and to discuss the theory on which these are based. However, these are given in detail by Bishop and Henkel (1962).

Series A Tests

The first attempts to establish the strength characteristics of oil sands were made in 1947-48 in the Civil Engineering Department of the University of Alberta as an M.Sc. project undertaken by T. G.

Tustin (1949). This work was done with the co-operation of Dr. K. A. Clark and involved a series of laboratory strength and consolidation tests on "undisturbed" and remoulded samples.

The samples came from the Bitumount pilot plant area. They were taken from shallow depths in oil-sand deposits that had been previously stripped. Some were taken as block samples and some in shelby tubes. No efforts were made in shipping and handling the samples to maintain the original stress and temperature conditions on the samples. In the interval between sampling and testing, therefore, the samples were subjected to stress release and temperature increases to at least 75°F (24°C).

In this testing program standard laboratory consolidation (Tschebotarioff, 1951) and triaxial compression tests were run on three sets of samples. For the first set the samples were cut from "undisturbed" samples of oil sand with the samples oriented at right angles to the bedding planes. For the second set the samples were oriented parallel to the bedding planes, and the third set was prepared from remoulded oil sand. On samples from each of these three sets, a series triaxial tests was run under "consolidated undrained" conditions. Further, the complete sequence of consolidation and triaxial compression tests were duplicated with one sequence being run with the samples at 73°F (23°C) and the second at 14°F (—10°C). The actual oil and water contents of the samples are not accurately known but the samples were from "rich" oil sand.

The original interpretations of the test results in many respects were erroneous because several pertinent factors which have since come to light were not recognized. However, the following conclusions from these tests appear to be valid:

(a) The consolidation test results showed the material to behave as a dense sand irrespective of whether the material was at 14°F or at 73°F.

(b) In the triaxial compression tests the material behaved essentially as if it were a saturated remoulded cohesive soil with very low shearing strength. In 47 separate tests the shearing strength ranged from 0.08 to 0.50 tons per square foot, with an overall average of only 0.19 tons per square foot. Only a slight difference was recorded between the strengths of the "undisturbed" and remoulded samples.

All samples, irrespective of test temperature, behaved essentially as disturbed material. The similarity of the results on frozen and unfrozen samples suggests that gas or oil in the voids governed the strength and deformation characteristics of the samples. These factors were not appreciated at the time the tests were run.

The low strength values indicated by these tests are realistic to the extent that they are consistent with the performance of oil sand after it has been disturbed. However, they are completely unrealistic for undisturbed oil sand. Exploratory mining operations, for example, indicate *in-situ* shearing strengths greatly in excess of these test values. In addition, exposures of the oil-sand deposits along the valley of the Athabasca River are standing on steep stable slopes for heights exceeding 100 feet. Stable slopes of these dimensions require shearing strengths much greater than those indicated in these tests.

These tests, therefore, gave no indication of the *in-situ* strength and failed to indicate the factors which govern the shearing strength of the oil sand. They did, however, show the lower limit to which the shearing strength can deteriorate, and for all practical purposes it may become zero for "rich" oil sand. In contrast the indications were that the oil sand has *in-situ* strengths comparable to that for soft sedimentary rocks.

Series B and C Tests

The question of the *in-situ* shearing strength of the oil sand was given attention early in the research program in connection with the development of Lease 17 at Mildred Lake. Preliminary tests on core samples using conventional methods of sampling and laboratory testing confirmed that unrealistically low shearing strengths were indicated. A series of tests (Series B) was therefore designed in which attempts were made, using freezing techniques, to maintain the *in-situ* strength conditions until the samples were set up ready for test. Undrained triaxial compression tests were run with the confining pressures and temperatures at time of testing being the test variables.

These tests showed the material to behave essentially as a normal dense sandstone. Within the accuracy of the tests no loss in strength due to increasing temperature occurred. This result at first appears to be contradictory to the results of the Series A tests. However, the temperature of the samples was allowed to increase at atmospheric confining pressure in the Series A tests, while in the Series B tests increase above the *in-situ* temperature was permitted only with the sample being under a confining pressure.

The strength characteristics from the Series B tests appeared to be reasonably realistic in terms of the known *in-situ* performance of the soil. There were, however, indications that at least some of the *in-situ* oil sand has strength values considerably exceeding those indicated by the test results.

In the Series C tests a similar set of triaxial compression tests was run on a group of core samples of "rich" oil sand from a shallow depth. In this series the temperature of the samples was not permitted to exceed the *in-situ* temperature of the oil sand of 41°F (5°C) from the time the cores were extracted from the bore holes until the tests were completed. Freezing techniques were also used for the samples in this series. These tests also showed the material to behave essentially as a dense sandstone.

Series D Tests

At this stage of the studies it was appreciated that the testing done to determine the shear strengh characteristics of the oil sand had all been on specially selected samples of "rich" oil sand. In the meantime, the field research program directed at mining problems had indicated a wide variation in *in-situ* shearing strengths. It was therefore decided to set up a strength testing program that would give results on samples representative of all grades of materials encountered in the McMurray Formation profile.

Consideration was given to the use of various conventional methods available for measuring *in-situ* shearing strengths such as the shear vane and cone penetrometer tests. However, for various reasons these methods were discarded, and it was finally decided to use unconfined compressive strength tests run in a field laboratory using portable testing equipment. This decision was influenced by the economic desirability of being able to combine the testing program with a drilling and coring program primarily designed for geological purposes. In embarking on this field testing program it was appreciated that the precision of carefully run laboratory triaxial compression tests would be sacrificed, but it was anticipated that this would be at least partially compensated for by the much greater number of samples that it would be possible to test in the field program.

This field testing program was put into operation during the winter of 1961-62 and continued until July, 1962. It involved tests on selected 2-inch cores taken incidental to the geological drilling and sampling program. A few tests (about ten) were run on 6-inch cores. A total of about 220 cores were tested from depths below the surface ranging from 13 to 234 feet.

The drilling and sampling was done with truck-mounted Failing 1500 rotary drill rigs. The cores were cut using a "fish tail" bit, with the cores being retained in the stationary inner tube of a double tube core barrel. Bentonite drilling mud was used.

The cores were logged by geologists. The selection of samples for strength tests proved more difficult than was anticipated. Much of the "lean" oil sand was so friable that intact samples could not be secured. On the other hand some samples that contained clay or shale inclusions were so hard that they could be dropped on the floor without fracturing. Generally the samples secured for the strength tests were from comparatively "rich" oil sand.

For each of the samples tested for strength, unit weights, percentages of solids, oil and water, and percentages of material passing the 100-, 200-, and 325-mesh sieves were determined. For the strength tests the unconfined compressive strength, per cent strain at failure and temperature of sample were recorded. The program was planned to run the strength tests within the temperature range 36° to 40°F (2° to 4.5°C). However, the core temperatures were influenced by the drilling mud temperatures and experience showed that an air temperature of —20°F (—29°C) or less was necessary to keep the drilling mud temperature below 40°F. Generally the temperatures of the recovered cores were within 4°F of the temperature of the drilling mud. Early in the testing program it was noticed that there appeared to be a reduction in strength with increasing time from the recovery of the core until the sample was tested. Following this, "aging" time was recorded for each sample as being the time from recovery of the core to the time the sample was tested.

For the samples tested the oil contents ranged from 0.43 to 20.7 per cent by weight. However, the oil content for 89 per cent of the samples exceeded 10 per cent, for 23 per cent of the samples it was within the range of 12 to 15 per cent, and it exceeded 15 per cent for 55 per cent of the samples. The water contents ranged from a trace to 12.6 per cent by weight, but for 73 per cent of the samples it was less than 2 per cent. The temperatures at which the samples were tested ranged from 33°F to 52°F (0.5°C to 11°C) except for one group of 35 tests run at the end of the program when the air temperatures, and therefore the test temperatures, were higher. However, in the main program 54 per cent of the samples were run at a temperature of 40°F (4.5°C) or less and for 80 per cent of the samples the temperature was less than 45°F (7°C). The per cent strain at failure ranged from 0.06 to 8.9, but for 67 per cent of the tests it was less than 2.0.

The unconfined compressive strength test results are plotted on a log scale against depth of sample on figure 2, the unconfined compressive strength being double the shearing strength (Bishop and Henkel, 1962). There is a considerable spread in the test results, but the best fit line (Line A) is reasonably well defined considering the fact that all samples tested have been included in this plot, and it therefore reflects the effects of variations in oil content, test temperature, aging time, overall density and so forth. Despite these variations the results definitely show that a marked reduction in strength occurs with depth of sample, with the strengths measured by unconfined compressive strength tests. This trend is particularly marked if the best fit Line A (Fig. 2) is compared with Line B (Fig. 2) which is the strength line for the *in-situ* oil sand if it is assumed that it has an effective angle of internal friction comparable to the slopes of outcroppings of the oil sand in the area, and with zero pore pressure acting. The trend is even more definitely indicated in figure 3 in which the strength values from the tests completed during winter and early spring air temperatures have been averaged over 20- and 40-foot bands from depths of 60 to 235 feet. These average values are plotted against depth to the mid-point of the band.

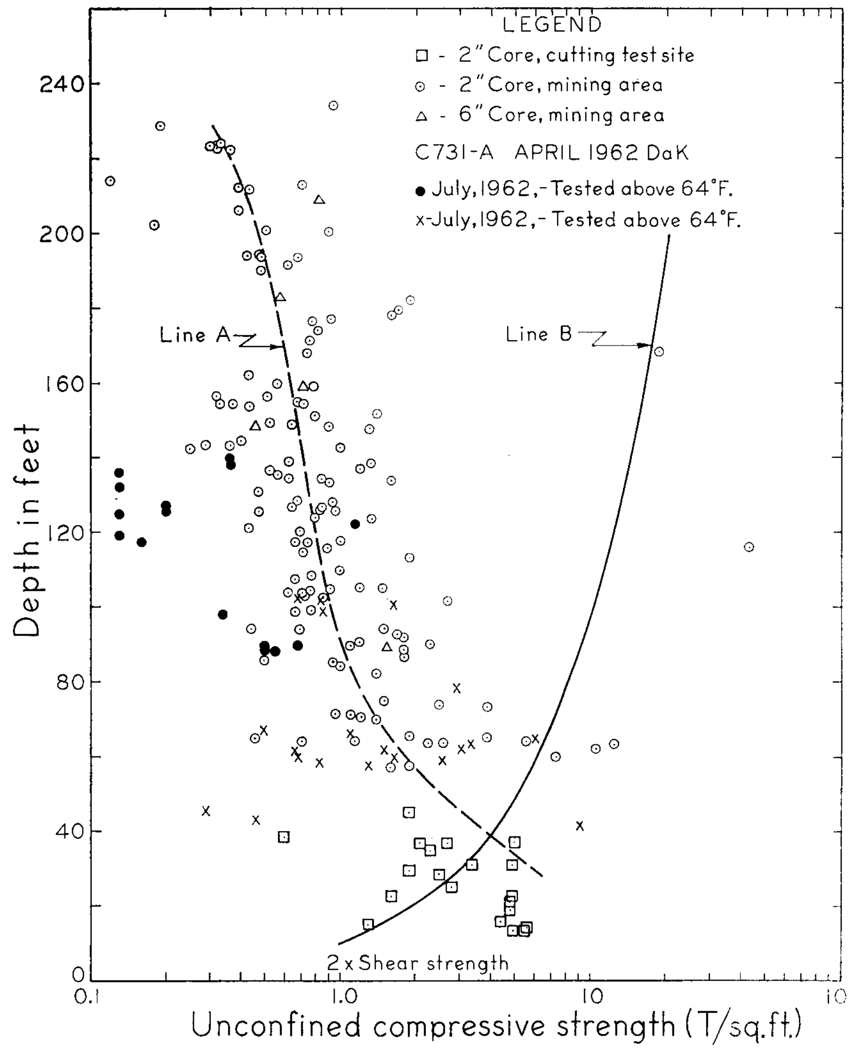

FIGURE 2. Log unconfined compressive strength of Athabasca oil sand samples versus depth below surface.
A. best fit line
B. strength of in-situ *oil sand derived from natural slopes.*

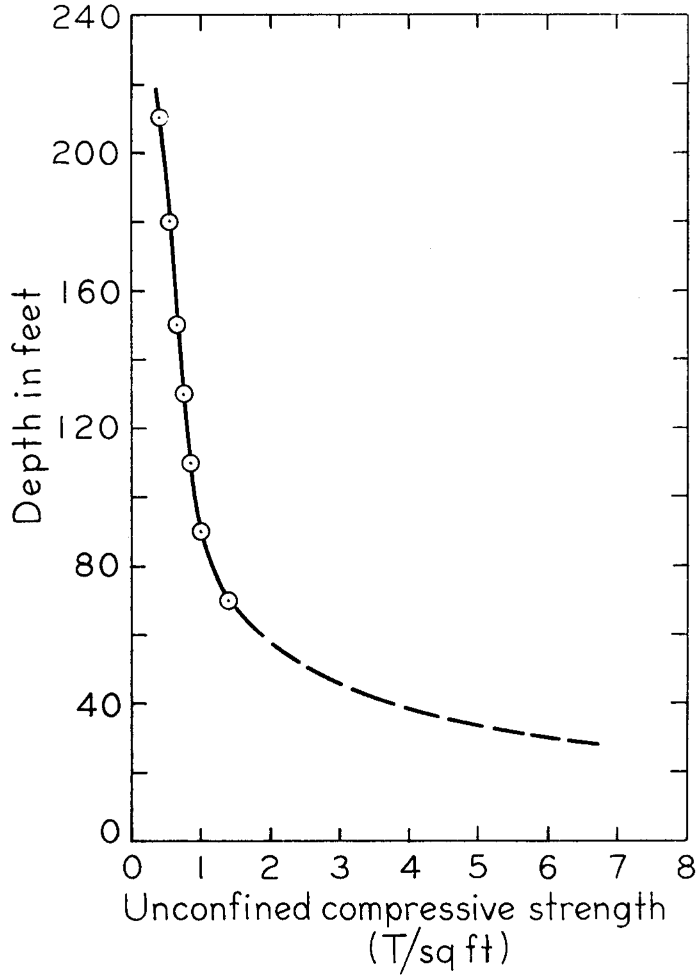

FIGURE 3. *Unconfined compressive strength of Athabasca oil sand samples versus depth, at early spring air temperatures. Plotted points are average of tests within bands of 20 and 40 feet in thickness.*

Detailed analyses of the test data showed several other general trends in sample behaviour. These included an increase in per cent strain at failure with increasing depth of sample, a decrease in strength with increased aging time and also with increased temperature at time of testing, and a decrease in strength with increasing oil content. However, the data were not sufficient to permit numerical values to be assigned to these variations.

EVIDENCE OF ESCAPE OF GAS

A considerable amount of qualitative evidence has accumulated of the escape of gas from oil sand under certain circumstances. Ells

(1926) appears to have been the first to record the phenomenon. Evidence of it was noticed in some samples in the Series B tests.

During the course of the Series D tests several incidents occurred which indicated substantial escape of gas from some oil-sand samples. In one case a 6-inch core 6 to 8 feet long of "rich" oil sand was observed to be splitting spontaneously along a longitudinal axis. This occurrence must have been the result of internal gas pressure in the sample. Similar splitting on longitudinal planes also occurred during the testing of some of the oil-rich samples. In addition, an effervescent effect was noticed on several freshly recovered cores. This appeared to be due to gas escaping from the sample and causing a bubbling in the drilling mud coating the sample. A further striking example of gas escape occurred in a sample of thinly layered shale and oil sand. Within 15 minutes after recovery of the core it was noticed that the surface of the sample had assumed a corrugated shape due to expansion of the oil-sand layers.

Series E Tests

In view of the evidence of gas release from the oil sand, and the fact that it appeared to be associated with both temperature and stress release, it was decided to run a series of tests in which the stress release would be more accurately controlled than was possible using previous techniques. A pressure core barrel was available in which samples from depth in conventional bore holes using drilling mud could be obtained without release of *in-situ* confining pressure. This device was built several years ago by Jersey Production Research (formerly Carter Production Research). The barrel is equipped with a conventional bit to cut 2⅜-inch diameter cores up to 6 feet in length. A linkage retracts the core retainer and closes a valve to seal the cores and drilling fluid at the bottom-hole pressure. Retention of pressure prevents alteration of the core due to stress release while the core barrel is being brought to the surface.

This pressure core barrel was used at the Mildred Lake project in July, 1962. Sixteen core samples were taken at depths varying from 26 to 150 feet. Twelve of these were retained at Mildred Lake and used for unconfined compressive strength tests to secure a higher range of test temperatures than were available from the Series D tests. The results of these tests are shown on figure 2 along with the Series D test results.

Four of the cores were frozen in the core barrel using dry ice immediately after the barrel was taken from the bore hole. Freezing techniques were used in the handling of these cores during shipping to and storing at the laboratory. The tests on these samples were designed to determine the effect of stress release on the strength characteristics with the effect of temperature increase above the *in-situ* temperature being eliminated. In addition, the effect of reapplication of confining pressure following at least partial dissipation of gas pressure in the sample was given attention.

The test procedures required for this series of tests were far from being routine in the field of soil testing for engineering purposes. As a result a number of technical difficulties developed in the testing program. In addition, the behaviour of some of the samples was somewhat unexpected, and therefore the performance of some of the initially tested samples was interpreted as being the result of test defects. There is also some evidence in the test results that the *in-situ* stress conditions were not in fact completely maintained in the sampling, handling and test set-up procedures. In some respects this series of tests should be regarded only as a pilot set of tests. However, at least qualitatively, they did indicate several important strength characteristics of the oil sand, of which the most significant was that the material appears to conform to effective stress concepts for the shearing strength of soils (Bishop and Henkel, 1962).

The use of the concepts of effective stress for engineering design purposes requires that the magnitudes of the pore pressures be estimated. The Series E test results did not provide quantitative data that permit the relationship between the magnitudes of pore pressure and stress release to be developed. In addition, while the results indicated a substantial recovery of strength on release of the gas pressure in the samples, they do not permit the magnitude nor rate of gas pressure dissipation to be quantitatively evaluated.

CONCLUSIONS

(1) McMurray oil sand is a four-phase system within the concepts of soil mechanics. The four phases are: solids, liquid water, gas, and highly viscous bituminous oil. This is in contrast to the usual concepts of soil mechanics in which soil masses are considered to be two-phase systems, solids and liquid water, or three-phase systems, solids, liquid water and air. The oil and gas phases of the material have a marked effect on its strength and deformation characteristics because, first, on release of stress gas comes out of solution in the oil phase, and second, the gas pressure in the pores increases with increasing temperature of the oil sand. The oil phase due to its high viscosity has the effect of delaying and to an extent preventing the dissipation of the gas pressure such as would normally occur in a partially saturated soil. The effect of the gas pressure is to decrease the effective stress in the oil sand mass and thereby decrease the shearing strength (Bishop and Henkel, 1962). Field experience and the laboratory tests show that the final effect may be to reduce the shearing strength from that of a soft rock to that of a viscous mass of sand and oil with the strength being dependent merely on the viscosity of the oil.

(2) Reduction in strength due to gas pressure in the oil sand is largely recovered if the gas pressure is dissipated. Under *in-situ* conditions it may be completely recovered in time.

(3) The conventional concepts of effective stress as used in soil mechanics appear to be completely applicable to the determination of the strength of oil sands. However, these concepts require that the pore pressures in the soil be numerically evaluated, irrespective of whether they are due to gas pressure or, as in more conventional practice, due to seepage water. The studies have shown that the gas pressures within the samples increase in general with increasing magnitude of stress release as well as with increasing temperatures above the *in-situ* temperature, but the test results do not provide quantitative data that will permit the relationship beween magnitudes of pore pressure and stress release to be developed. Neither do they permit the rate of gas pressure dissipation to be evaluated.

(4) Under *in-situ* conditions the oil sand at Lease 17 has strength and deformation characteristics consistent with those of a dense sandstone.

ACKNOWLEDGMENTS

This paper is presented with the approval of the management of Cities Service Athabasca, Inc., under whose auspices the studies were made. The Series B tests were run by Mr. E. W. Brooker and the Series C tests by Dr. S. Thomson, both of the Civil Engineering Department of the University of Alberta. The Series D and E tests were run by the staff of R. M. Hardy and Associates Ltd. The test facilities of the Department of Civil Engineering of the University of Alberta were used for the Series A, B, C and E tests.

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STEAM-DRIVE — A PROCESS FOR IN-SITU RECOVERY OF OIL FROM THE ATHABASCA OIL SANDS

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R. W. Zwicky[*]

ABSTRACT

This *in-situ* method of recovery of oil from the Athabasca oil sands was developed by Shell Canada Limited. The method involves the injection of steam and alkaline aqueous solutions into the oil sands. The recovery scheme was developed as a result of ten years of geological reconnaissance and interpretation, laboratory research, and a series of pilot experiments in the field. The results of these experiments are discussed. From these experiments and subsequent studies, the parameters influencing technological and economic feasibility were developed and are herein described.

INTRODUCTION

It has long been accepted that the low-cost strip mining of the Athabasca oil sands is restricted to areas of low overburden and that by far the greater quantity of oil can not be economically produced by this method. Because of this, Shell Canada Limited decided many years ago to concentrate its research on an *in-situ* recovery method which would be applicable to the greater portion of the deposit.

The Athabasca oil is so viscous that primary and secondary recovery methods common to the petroleum industry are not useful for producing the bitumen. In order to produce the oil without moving the sand it is necessary to make the oil flow.

Shell's steam and chemical drive method accomplishes this. The process requires that injection and production wells be drilled in regular patterns such as a five-spot pattern. The wells are opened to the oil-sand formation and a horizontal disc-like fracture propagated connecting sets of injectors to producers. Steam and aqueous alkaline solutions are then injected into the fracture. The oil adjacent to the fracture is heated, and is then emulsified and driven to the producer as an oil-in-water emulsion. Figure 1 is a schematic drawing of the field system while figure 2 shows the supporting plant required for a commercial-sized operation.

GEOLOGY

The Athabasca oil sands are found mainly in the Lower Cretaceous McMurray Formation. This formation overlies the eroded Devonian Waterways Formation with angular unconformity and is overlain by the Cretaceous Clearwater Formation.

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[*] Shell Canada Limited

Relief on the Devonian surface is very gentle. It is caused for the most part by erosion, but in some areas the topography of this erosional surface has been masked somewhat by post-McMurray folding. The configuration of the erosional surface is vague with some indication of northwest-trending, large, elongated domes and depressions. Relief on these features is in the order of 200 to 400 feet in 10 to 15 miles. The smaller structural features have no discernible orientation. These structures have a relief of about 100 feet in 1 mile. It has been postulated that the observed post-McMurray folding is the result of slumping of the beds into collapsed solution caves in the underlying Devonian Prairie Evaporite Formation.

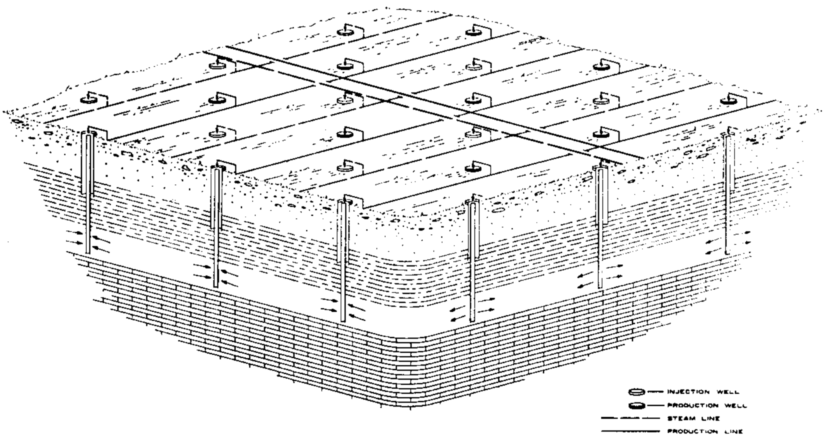

FIGURE 1. Layout of steam-drive flood pattern.

Overburden on the oil sands consists of the predominantly shaly Lower Cretaceous Clearwater and later formations and Pleistocene sediments. The overburden has been completely eroded in a narrow band along the Athabasca River but ranges in thickness up to about 1700 feet within the Government-designated bituminous-sands area. Overburden on the leases that Shell has explored west of the Athabasca River averages 1050 feet of which 700 to 1000 feet is shale with up to 350 feet of Pleistocene sands and gravels.

Regionally the McMurray Formation thins to 50 feet over the highs and thickens to 300 feet in the lows. The result is a general flattening at the top of the McMurray Formation. This reduction in relief is not complete because of the post-McMurray folding.

Regional correlation within the McMurray Formation is extremely difficult; however, on a local scale continuity is definite. Figure 3 (a cross section through a part of Shell's leases) shows the type of correlation possible over several miles, based on similarities of cores and characteristics of log curves.

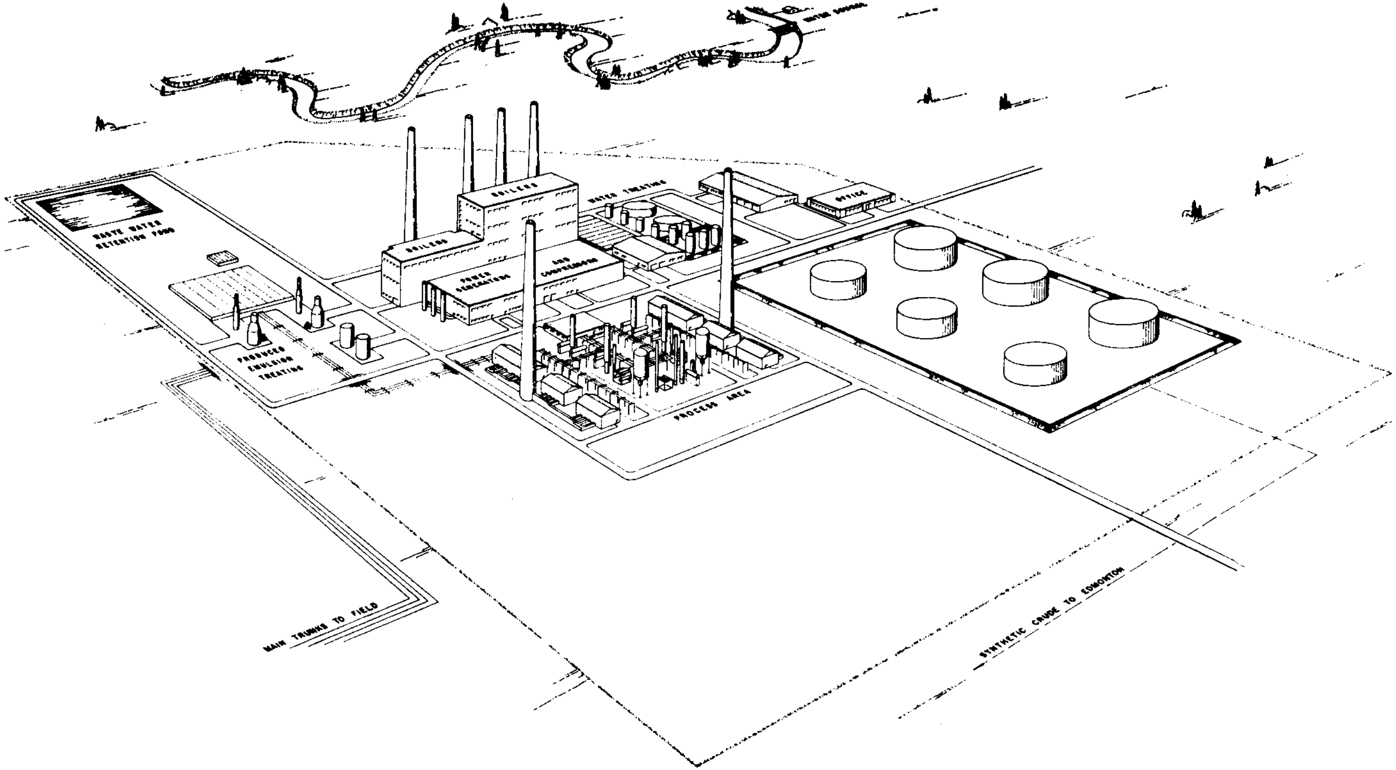

FIGURE 2. Layout of commercial-sized plant.

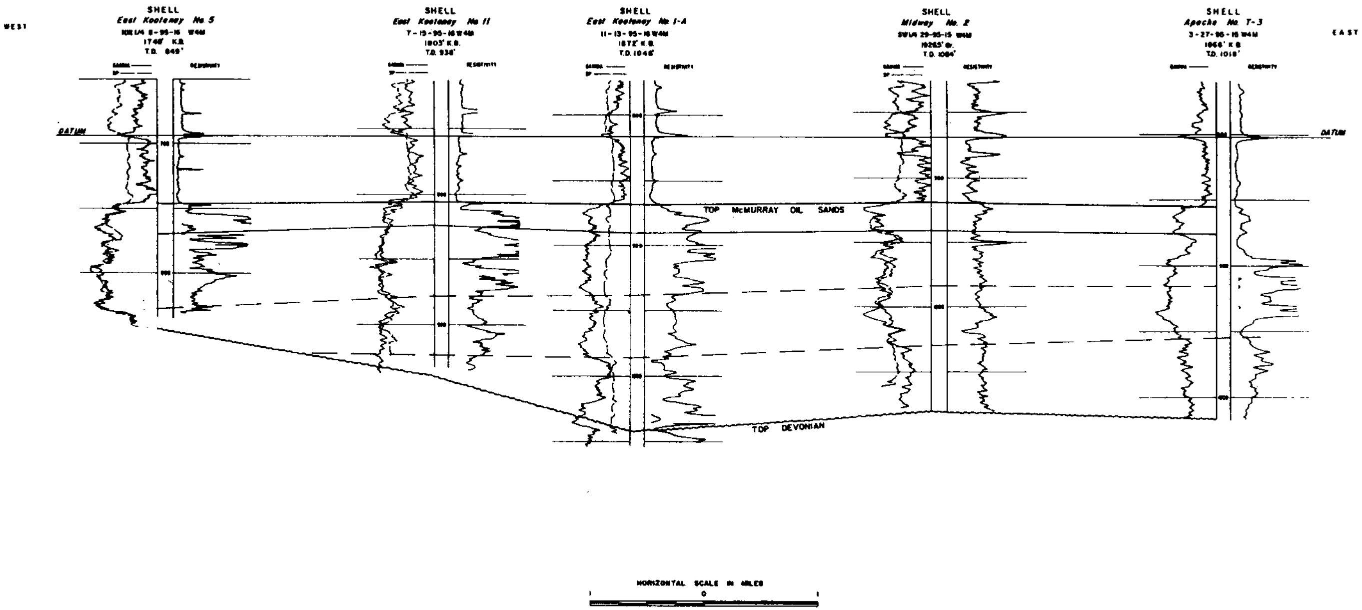

FIGURE 3. *Correlation within McMurray Formation on Shell's leases west of the Athabasca River.*

Mineralogically, the McMurray oil sands consist of subangular to rounded, well sorted, translucent quartz grains. Analyses have shown that minor constituents include feldspar, mica, and some heavy minerals. These sands are usually unconsolidated, very few being locally cemented by iron sulphides. Grain size within the McMurray Formation varies from coarse sands and pebbles to silts and clays.

The measured porosity of the oil-saturated sands varies from 38 to 42 per cent, while the oil saturation varies from 40 to 50 pore volume per cent. On a weight basis, the average oil saturation of the good sands is 10.4 per cent of the dried water-free sands.

The environmental criteria observed in core and outcrop and the regional geological framework established from cross sections and maps indicate a geological province in which the sands consist of three main types. The depositional pattern shows an alluvial environment to the east where a westward-flowing, meandering, sand-laden river deposited clean, point-bar sands on the low relief of the Devonian surface. Westward, in the vicinity of Shell's project leases (Fig. 4), the sands are those of a river system near its mouth with associated deltaic and marine sands where it emptied into the advancing sea. Sands in the area of the subject leases have been formed in the shallow submarine portion of this delta. Detailed log and lithological correlations in this area show a very definite lateral continuity of this deltaic sand. The massive deltaic sands and river point-bar deposits were transgressed by winnowed shoreline marine sands and eventually overlain by thick marine clays and shales of the Clearwater Formation.

DEVELOPMENT OF SHELL'S PROCESS

INITIAL EXPERIMENTS

In 1956, Shell's laboratory research program on methods of producing the petroleum reserves of the Athabasca bituminous sands led to the discovery that certain aqueous alkaline solutions of synthetic detergents were capable of wetting, emulsifying, and suspending the oil* contained in samples of the McMurray Formation.
Still further research revealed that heated, critically prepared solutions of sodium hydroxide were even more efficient in emulsifying the oil.

This laboratory research was verified in 1957 when, on what is now Shell's Bituminous Sand Lease No. 26 (Fig. 4), 800 barrels of an alkaline solution of a synthetic detergent were injected into Shell West Athabasca No. 9 well. Bitumen emulsions, containing 4 to 30

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*Oil and bitumen are used interchangeably to refer to the petroleum contained in the Athabasca sands.

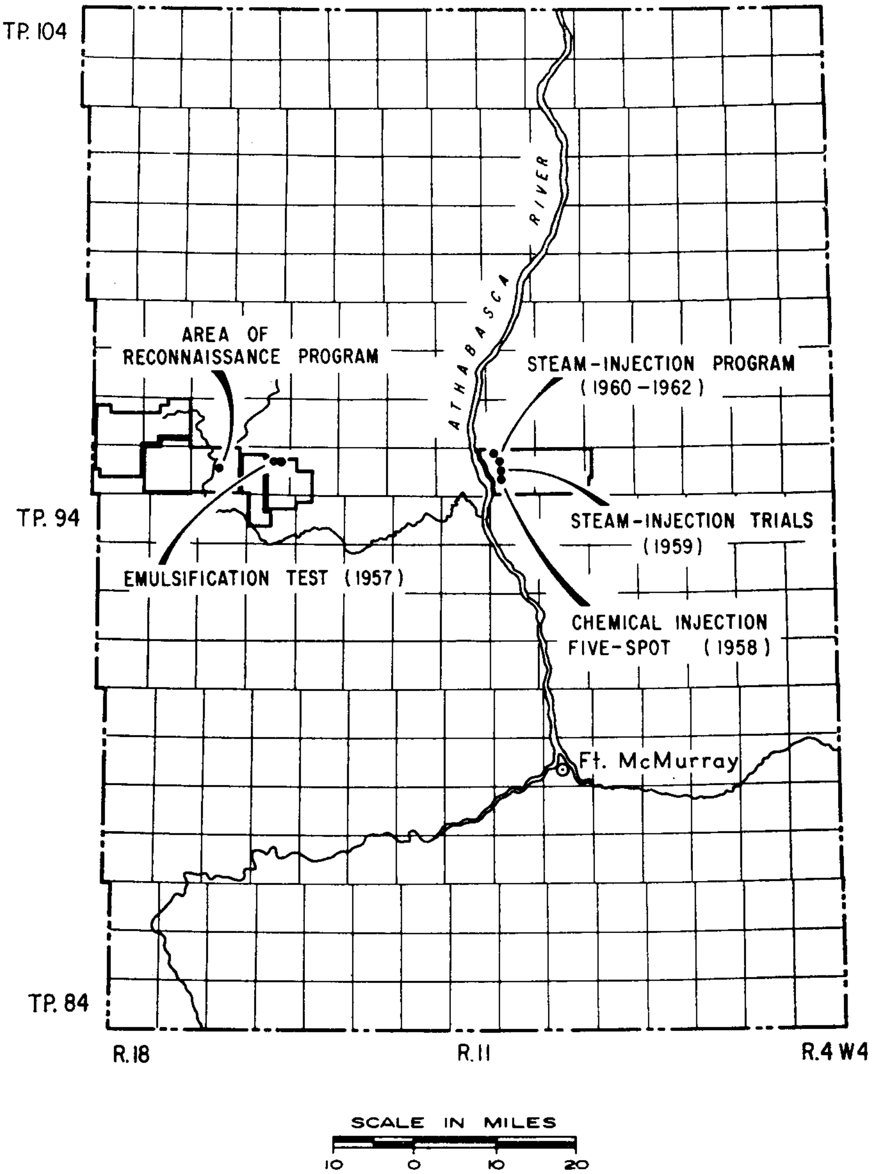

FIGURE 4. Locations of Shell's test program sites 1957-1962.

per cent oil, were produced from Shell Apache No. T-1 well, 26 feet away from the injector. This was the first attempt at *in-situ* recovery to give significant oil production from the Athabasca oil sands. This field test showed that the natural permeability in most of the oil-saturated intervals in the McMurray oil sands was too low to obtain the high fluid-injection and production rates required for commercial production.

Simultaneous study of the lithology of the McMurray Formation and measurements of injectivity-pressure relationships had led to the conclusion that horizontal fractures could be induced in the formation, and that such fractures might be used to supplement the low natural permeability of the formation. Accordingly, in 1958 a field experiment was initiated on Shell's Bituminous Sands Lease No. 13 to determine the applicability of these new findings. Five wells, in a five-spot pattern on 0.03 acre, were drilled to the base of the McMurray Formation at approximately 200 feet, and, as expected, it was possible to induce and propagate horizontal fractures between the wells. After creation of the fractures, dilute alkaline solutions of critical concentration were injected into the fractures and the bitumen in the adjacent McMurray sand was emulsified, and transported to and recovered from the central producing well. However, oil-production rates could not be maintained at satisfactory levels.

Concurrent with these field studies, continuing laboratory investigations revealed that steam was capable of enhancing the action of the critically prepared alkaline solutions. Such studies also suggested that the injection of steam would increase the sweep and displacement efficiencies of the dilute aqueous alkaline solutions, would give a high overall recovery, and that in time steam might be used as the principal injection fluid with minor but necessary intermittent use of the aqueous alkaline fluids.

In 1959, Shell conducted field work on methods of completing wells for the injection of steam and made trial runs at injecting steam into fractures in the McMurray sand.

1960-1962 EXPERIMENT

In 1960, a highly instrumented and extensive pilot program of the process, combining the use of steam and alkaline solutions was started on Lease No. 13 in an oil-saturated interval beginning at a depth of 150 feet and extending to a depth of 219 feet. The program was designed to provide basic information for the evaluation of the technical and economic feasibility of the recovery process. The layout of the test is shown on figure 5.

To set up the experiment, five operating wells for injection and production were drilled to form a single five-spot pattern. In addition six temperature-observation wells were drilled and completed within and without the five-spot to permit observation of the progress of the experiment. The formation within and around the five-spot was fractured by using dilute aqueous alkaline solutions. After the establishment of communication between the wells via the induced horizontal fractures, sand-exclusion liners were set in the operating wells.

To begin the experiment, heated aqueous alkaline solutions were injected through the central well and fluids were withdrawn from the corner wells and vice-versa, until a radial, horizontal flow-path was

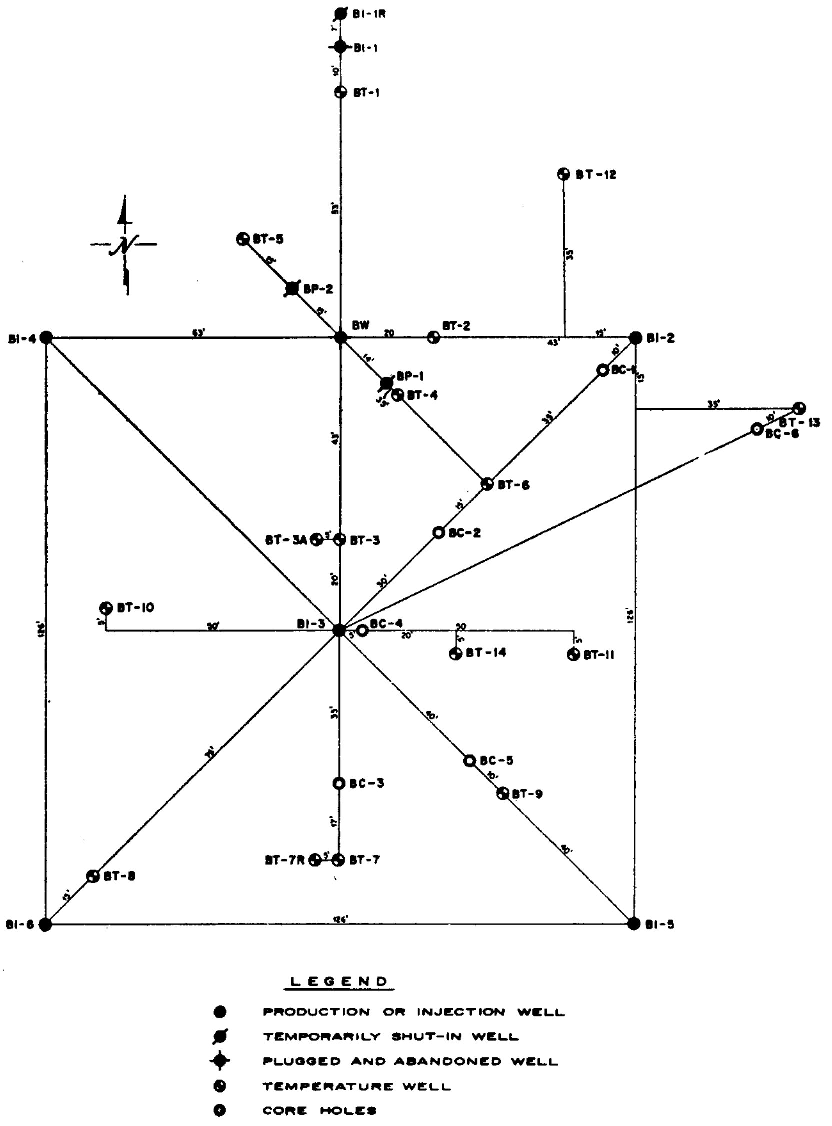

FIGURE 5. Plan of 1960-1962 steam-injection program.

established at the base of the oil sands. Fluid injection into the central well—and withdrawal from the corner wells—continued until the fluid conductivity of this path permitted steam injection at desired rates and pressures. Steam and aqueous alkaline fluids were injected into the central well for approximately nine months, during which the oil in aqueous emulsions was withdrawn for the corner wells only. Additional temperature-observation wells were drilled at this time.

This work was completed by August, 1961, and the technique for creating a highly permeable horizontal path of sufficient thickness at the base of the oil sands had been developed. Higher-capacity steam-generating equipment was installed in November, 1961.

During the experimental program to January, 1962, a significant portion of the fluids injected leaked away into the reservoir beyond the pattern area and was not recovered at the producing wells: the pattern was not confined. Thus, on January 1, 1962, of the 138,125 barrels of fluid injected, 52,604 barrels, or 38 per cent of the volume had been recovered. A volume equal to 62 per cent of the injected fluids was displaced into the fractures and permeability developed beyond the five-spot area. The quantity of bitumen displaced from the five-spot into the reservoir beyond the pattern area could not be determined directly but rather was estimated by assigning to the volume of fluid leaking off into the reservoir during a set time-period an oil content equal to that of the fluids withdrawn from the producing wells during the same time-period.

The 2,457 barrels of oil contained in the fluids withdrawn from the corner wells together with the calculated 7,623 barrels of oil displaced from the five-spot area into the reservoir beyond the pattern amounted to 10,080 barrels by January 1, 1962. Saturations measured in the cores cut at the start of the program in wells BI-4, BP-1, and BT-4 (Fig. 5) indicated that there were approximately 44,000 barrels of bitumen inside the pattern area in the McMurray sand between depths of 150 and 219 feet. The total of 10,080 barrels removed represents 22.9 per cent of the oil originally in place in the pattern.

To prepare for the final production test—injection of fluids into the corner wells and withdrawal of fluids from the central well of the five-spot pattern—five additional temperature-observation wells were drilled and completed between January and March, 1962. Steam, at rates severalfold higher than those used previously, was then injected into the corner wells. This reversal of the injection pattern was undertaken in order to confine the flow of fluids in the reservoir to the pattern area as much as possible. Slugs of alkaline solutions were injected into the producing well, when necessary, to maintain or restore productivity. In this way a volume equal to 78 per cent of

TABLE 1. INJECTION AND PRODUCTION SUMMARY, 1960-1962 PROGRAM

Period	Steam injected, in terms of steam condensate (Bbls)	Water and aqueous solns. (Bbls)	Total fluid injected (Bbls)	Produced fluids (Bbls)	Produced fluid / Injected fluid (%)	Produced oil (Bbls)	Produced oil / Produced fluids (av. cut) (%)	Oil displaced into surrounding reservoir (Bbls)	Total oil displaced from pattern (Bbls)	Per cent of oil in virgin reservoir displaced* (%)
Prior to ** April 1962	26,994	111,131	138,125	52,604	38.0	2,457	—	7,623	10,080	22.9
April	9,442	81	9,523	1,073	11.3	127	11.8	0	127	0.3
May	9,881	54	9,935	4,036	40.6	1,033	25.6	0	1,033	2.3
June	8,047	1,521	9,568	7,474	78.0	2,118	28.3	0	2,118	4.8
July	—	—	—	1,281	—	517	40.3	0	517	1.2
Total, April-July	27,370	1,656	29,026	13,864	47.8	3,795	27.4	0	3,795	8.6†
Grand Total	54,364	112,787	167,151	66,468	39.7	6,252	—	7,623	13,875	31.5

*Original oil in place is 44,000 Bbls using average saturations measured in cores BI-4, BT-4 and BP-1.

**While injecting into central well, subsequent to April 4—injection was in corner wells BI-2, BI-4, BI-5 and BI-6.

† or 11.2 per cent of oil remaining inside the five-spot area at the start of step 4.

the injected fluids was recovered during the latter stages of the experiment. The monthly and total fluid balances for the test are given in table 1.

In the four-month period of the final production test, 3,795 barrels or 8.6 per cent of the original oil in place were produced. This, together with the 22.9 per cent displaced in previous operations, gives a total displacement of 31.5 per cent of the oil originally inside the five-spot. The peak production rate of oil reached 90 barrels a day which was produced in 300 barrels of total fluid, an oil-water ratio of 0.43 to 1.

The production behavior observed in the period from April to July, 1962, and summarized on figure 6, represents the performance to be expected from a single producer in a steam-drive operation. The test also showed no significant decline in the oil to water ratios, even after more than 30 per cent of the original oil has been displaced from the pattern.

The final step of the program was to provide a better understanding of the manner in which bitumen was displaced and to verify the calculated values of the amount of oil displaced from the five-spot. Six core-holes were drilled and logged (BC-1 to BC-6, Fig. 5). Average values of the residual oil saturations in each of the post-injection core-holes are shown on figure 7 for successive 10-foot intervals as compared to the oil-saturations measured at the beginning of the program. The displaced oil is the difference between the original saturation and the residual saturation. Average residual saturation and average per cent displacement (displacement efficiency) are shown for each core hole on the figure. A study of these data indicates that at the time the experiment was stopped, a time chosen arbitrarily in order to obtain information on the dynamics of the process, oil residuals had been reduced in some intervals to as low as 7 per cent. This occurred close to an injection well where the displacement process would naturally be more advanced. The absence of any interval farther from the injection well showing the same high degree of depletion is evidence that channeling of injected fluids was not imminent at the time the experiment was ended. It is also apparent from figure 7 that a considerable vertical distance had been swept, even though steam was injected into only the bottom few feet of the formation. For example, in BC-1 the average depletion in a zone which covers the lower 49 feet of the McMurray Formation is 74 per cent of the original oil in place.

The average percentage of oil displaced from the interval from 150 to 219 feet, as measured in the five core-holes within the pattern, is 43 per cent as compared to the volumetrically calculated average of 31.5 per cent referred to earlier. The reason for this is that oil depletion in the southwest quadrant adjacent to BI-6 was significantly

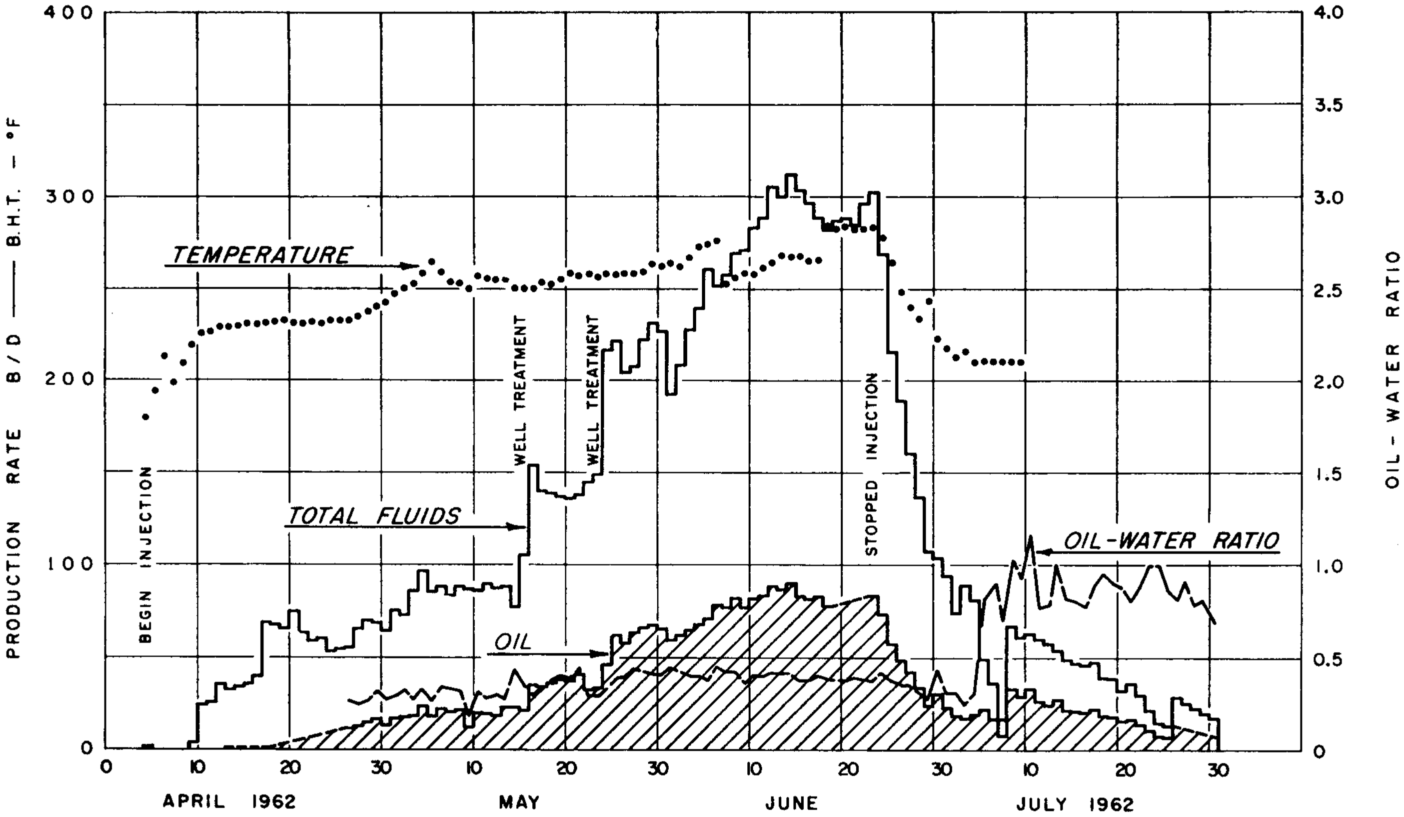

FIGURE 6. *Production test data April - July 1962, showing temperature, total fluids produced, oil produced and oil-water ratio.*

less than the depletion which occurred in the other three quadrants. Well BI-6, at the southwest corner of the pattern, was completed differently from the other operating wells, and in such a manner that formation sweep in its vicinity was severely restricted throughout the experiment. The lower temperatures observed in the southwest quadrant at the end of the operations are evidence of this poor sweep. Further substantiation of the poorer depletion which occurred in the southwest quadrant is obtained when the depletion measured in the core from well BC-3, the only well which gives some indication of the amount of depletion in the southwest quadrant, is compared to the depletion measured in the cores from wells BC-1, BC-2, BC-4, and BC-5. The average depletion in BC-3 is 20 per cent, while the average for the other four wells is 49 per cent.

The performance of the experimental operation between April and July, 1962 showed that oil production paralleled total fluid production. Total fluid production also closely followed total fluid injection.

APPLICATION OF PROCESS

RECOVERY, DISPLACEMENT, AND SWEEP EFFICIENCIES*

Based on Shell's laboratory research, theoretical work, and data established in the field experiments conducted between 1960 and 1962, a recovery efficiency of at least 50 per cent and possibly as high as 70 per cent of the oil in place in the clean McMurray sands is anticipated from this recovery process.

The 1960-62 experimental program was stopped while favourable production response was still being obtained in order to get cores from the reservoir at a time when direct evidence of the dynamics of the process would be revealed, and thereby confirm its mechanism as theoretically deduced from laboratory studies.

The oil-sand body inside the five-spot between 150 and 219 feet was stratified and had at least one thin but continuous shaly zone approximately 20 feet above the base of the McMurray sand. Good oil-displacement occurred as much as 50 feet above the base of the

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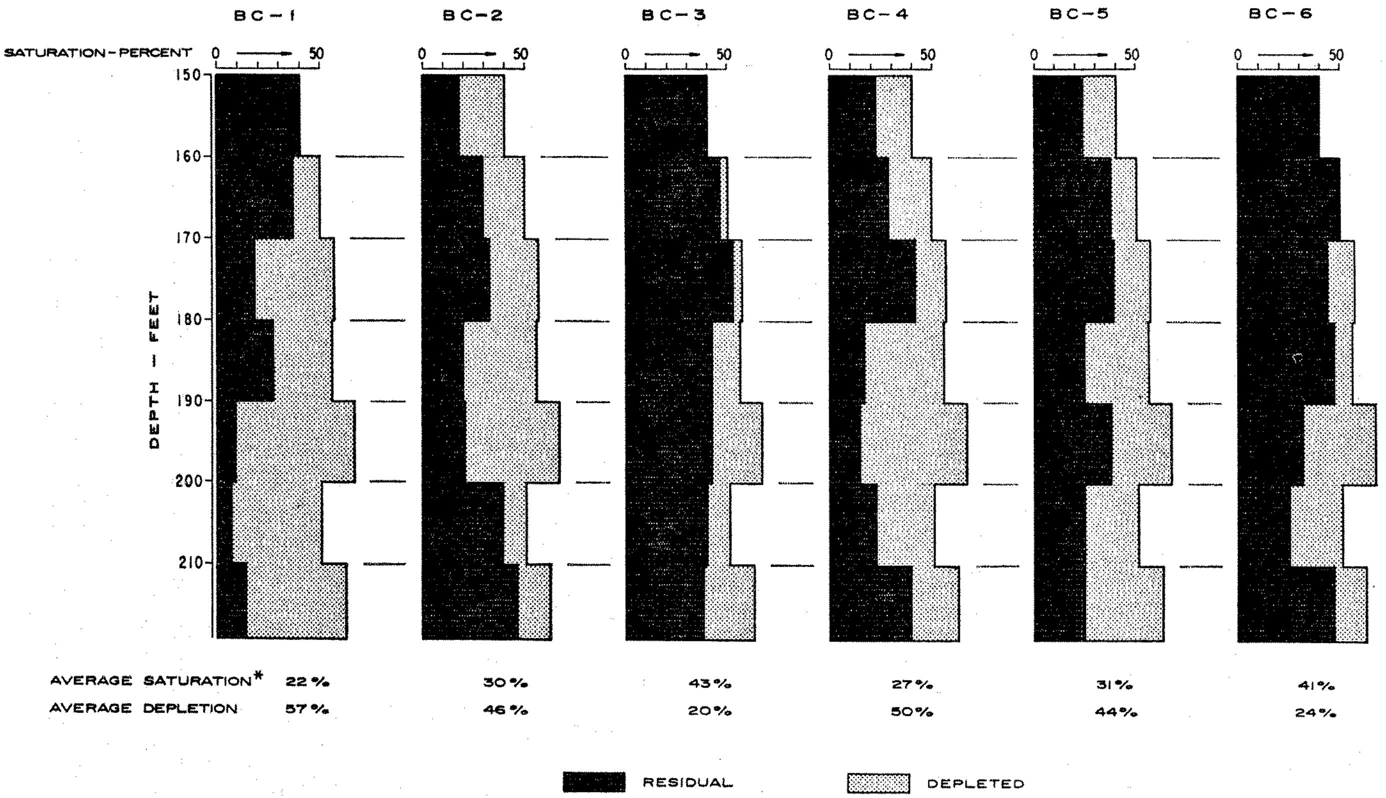
***Sweep efficiency** is defined as the percentage of the entire producible interval through which the displacing fluids pass.

Displacement efficiency is defined as the percentage of the original oil-saturation which has been removed at any point in this swept volume. The average displacement efficiency is the average of the displacement efficiencies observed at all points in the swept volume.

Recovery efficiency is defined as the percentage of the original oil in place in the entire producible interval which has been produced and is equal to the product of the sweep efficiency and the average displacement efficiency.



* ORIGINAL AVERAGE SATURATION 55 % OF PORE VOLUME MEASURED FROM CORES IN WELLS BI-4, BT-4 & BP-1

FIGURE 7. Oil depletion by steam-drive process during 1960-1962 program.

sand and in every well cored appreciable oil was displaced above this shale zone, observations indicating that thin impermeable zones will not interfere with the process.

The production data show that during the final period of the experiment, long after steam condensate appeared in the producing well, there was no significant decrease in either the rate of oil production or in the oil-to-water ratio. This leads to the conclusion that by continuing the experiment, greater depletion could have been achieved, and that recovery efficiency in the area swept would finally equal or better the greatest displacement efficiency measured. This was 74 per cent of the initial oil in place and was measured in the lower 49 feet of well BC-1.

As stated previously, 31.5 per cent of the oil originally in place was removed from the pattern area. At a displacement efficiency of 50 per cent, the average from core analyses in wells BC-1, BC-2, and BC-4, drilled between BI-2 and BI-3 (Fig. 5), the volume of the pattern swept would have to be at least 63 per cent of the total. The performance of the experiment showed that the southwestern portion of the test area was the least affected by the process. This is confirmed by core analysis, since the average displacement in the cores from BC-3 was 20 per cent as compared to the 50 per cent average in the core holes mentioned above. Thus, the displacement within the total pattern area must average less than 50 per cent. It follows that at the time that the experiment was terminated the volume of reservoir swept was greater than 63 per cent of the total. Model experiments with prototype fluids and bead packs in the laboratory together with the field result have led to the conclusion that a sweep efficiency of 70 per cent can be anticipated for a fully developed injection and production pattern.

Combining the displacement efficiency of at least 70 per cent with a sweep efficiency varying from 70 to 100 per cent yields, a recovery efficiency ranging from 50 to 70 per cent. Recovery efficiency of this magnitude is in general far greater than that obtainable in conventional oil-reservoirs.

INJECTION RATES AND PRESSURES

Injection rate and pattern spacing are the two principal variables in the steam-drive process that must be taken into account in order to combine optimum economic results and sound conservation practices. One of the most significant factors affecting the overall economic efficiency of this process is the quantity of heat lost to the underlying and overlying formations. As the heat loss increases, the greater is the fraction of the produced oil that must be used as fuel for heating the injected fluids. The heat loss can be reduced by heating the oil-bearing sand more rapidly, by injecting steam at higher rates and by decreasing the well spacing. However, as injection rate and well density are increased, capital costs for surface and subsurface facilities increase rapidly. Also, the injectivity of steam is physically limited by the permeability that can be developed in the oil-laden sand, by the maximum pressure that can be applied to the formation without causing rupture in a vertical plane, and finally by the distance between injection and production wells:—the pattern spacing.

PRODUCTION RATES

Because initially no significant volume of gas or other compressible fluid is present in the formation, the production rates should be approximately equal to the injection rates of steam, measured as condensate. The production volume would be greater than this if a vapour phase of uncondensed steam and hydrocarbon gases stripped out of the bitumen accumulated in the reservoir. This will occur as injection proceeds, and the vapour volume is expected to eventually occupy 25 per cent of the reservoir volume as shown by laboratory studies. Thus, for an injection rate of 100 tons of steam per well per day and a five-spot development pattern, a minimum fluid-production rate of 560 barrels per well per day should be obtained. This would increase with time to an overall average of 700 barrels per well per day. The field experiments indicated that oil cuts of 25 to 30 per cent can be sustained, and therefore peak bitumen-production rates between 175 and 210, or approximately 200, barrels per well per day may be expected for the above injection rate.

SPACING CONSIDERATION

It is possible to calculate the total amount of heat that must be injected to raise the temperature of the entire reservoir to a value which is adequate to complete the production process. If none of the heat injected into the oil-laden McMurray sand is lost to overlying and underlying formations, the heat required to raise the temperature could be calculated directly from a knowledge of the heat capacity of the formation and its volume. However, some of the heat injected into the reservoir will be lost by conduction to oil-free formations, and this loss of heat is a function of both the life of the project and the spacing. Methods for calculating these losses have been developed; hence the total heat required to raise the formation temperature to some required value may be calculated. From the total heat requirements, the heat liberated by a unit weight of steam, and a selected injection rate, the duration of the steam-injection period may be calculated.

Based on laboratory and field experiments, it has been concluded that the maximum temperature to which the formation will have to be raised is 350°F. For example, at the injection rate of 100 tons of steam per well per day and with 4-acre spacing of five-spot patterns, steam injection continued for an estimated 900 days would raise the temperature of the affected reservoir volume to 350°F.

In an economic-sized system comprised of a multitude of injectors and producers in a regular array, individual five-spots will be in operation for a period longer than that required for steam injection alone. Initially, a short period of time will be required to induce horizontal fractures hydraulically with aqueous solutions. No significant production will occur immediately from such fracturing. Bitumen production is to be expected as soon as the entire disc-shaped permeable path between wells has become heated. To facilitate initial heating, hot fluids and steam will be first injected into the production wells, and after a period of time, during which about one half of the path length between wells has become heated, injection will be switched to the injection wells. Knowledge of heat transmission in the reservoir permits prediction of the time required to extend the heated zone along the entire length of the path between wells and it is at this point that significant bitumen production occurs (Fig. 8). The production rate builds up rapidly to the peak rates described above, because all conditions will have been satisfied for vertical growth of the steam-heated zone and horizontal displacement of the hot aqueous emulsion of oil.

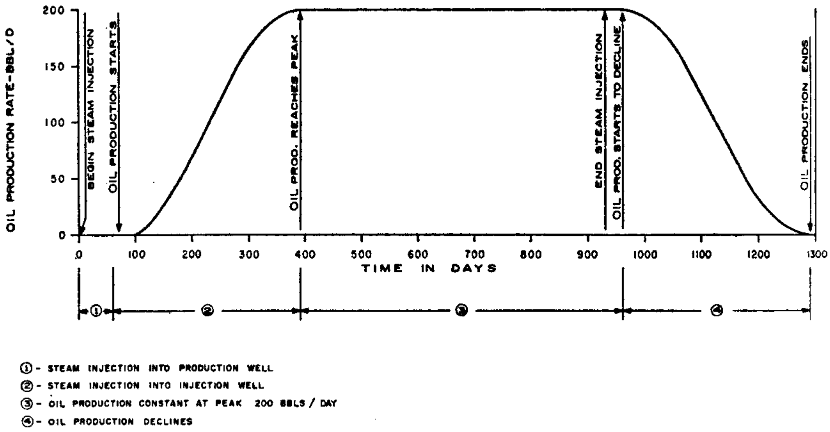


FIGURE 8. Predicted injection and production performance of a single five-spot in a steam-drive project.

At the end of steam injection, production will be continued by pressure reduction. It is assumed that production decline will be exactly the reverse of production build-up*.

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*Because of the relatively rapid build-up and the long period at constant peak-rate the effect of this assumption has little significance on the overall operating performance.

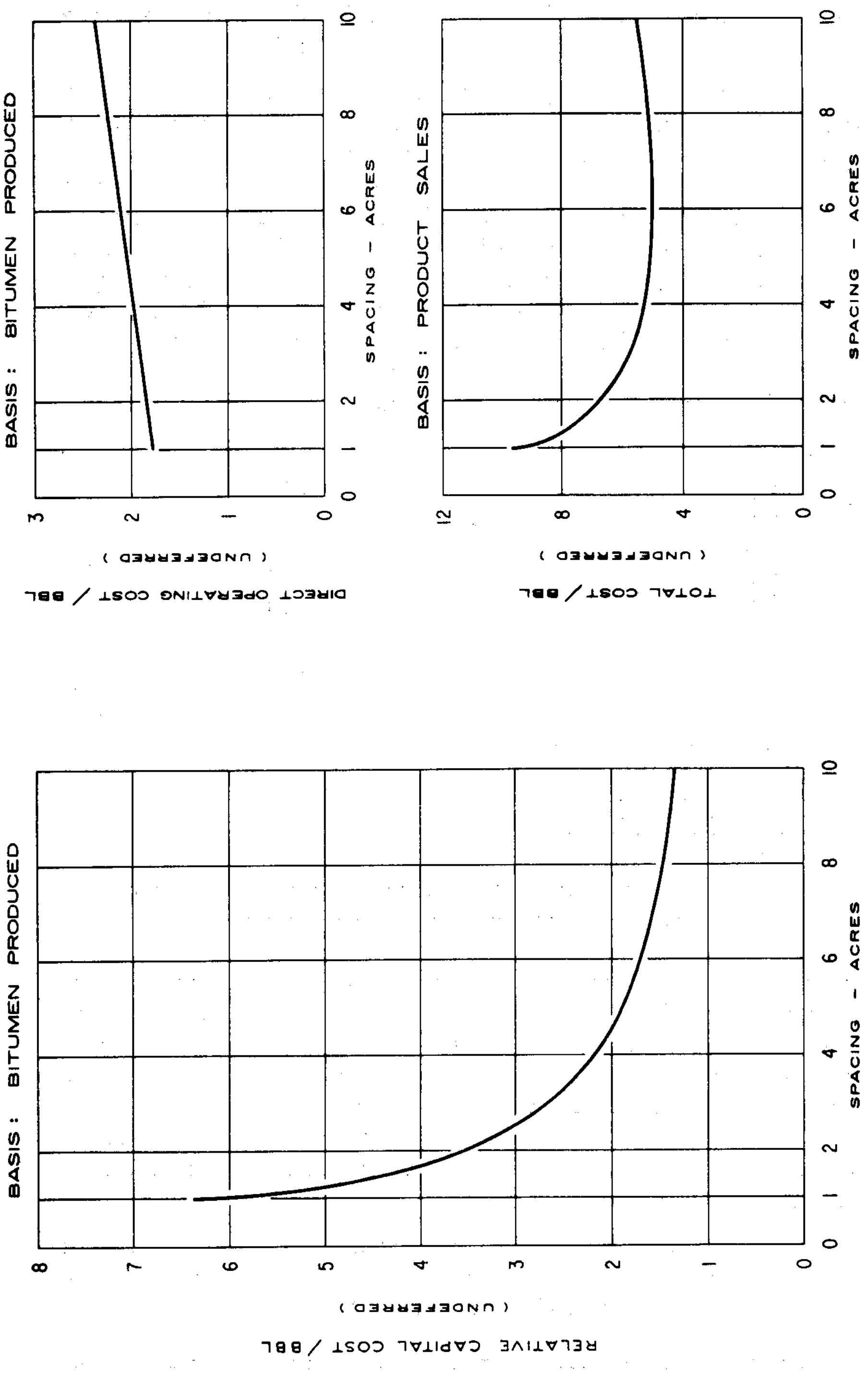

FIGURE 9. *Effect of spacing on operating and capital cost.*

From the above, the production history of a well can be estimated for any given spacing. The production history of a project is the effect of summing many individual patterns, and replacing depleted patterns with others as required. It is therefore possible to determine optimum spacing for a commercial-sized operation by giving consideration to the economics of the process. During the life of the project, capital and operating costs will be incurred by the drilling and completion of additional patterns of wells, and by the installation and operation of surface facilities for injecting steam and auxiliary fluids and for handling produced fluids. Feasibility studies were conducted to ascertain the effect, on the cost of a barrel of produced material, of variations in spacing for the 100 tons per well per day injection rate with the corresponding production rate of 200 barrels of bitumen per well per day. The relative changes in capital, operating and total costs are shown on figure 9. These plots indicate that the change in cost per barrel of liquid product will not be significantly different when the operation is carried out on spacings larger than 4 and smaller than 8 acres. Since the net amount of product is higher at the lower spacings (because of decreased thermal losses and hence less oil burned as fuel) the decision as to spacing should be made on conservation grounds and hence would be 4 acres per producer. Over the range of spacings dictated by the indicated injectivity and economic evaluation, there is no reason to expect any effect of spacing on the overall recovery efficiency of the process.

CONCLUSIONS

Through extensive experimentation in the laboratory and the field, a process has been developed that can successfully produce oil by an *in-situ* process.

The 1960-62 field experiment showed the steam-drive process to have a high efficiency and good productivity.

Reconnaissance drilling and geoolgical studies have shown the existence of oil-in-place of sufficient continuity and volume to support a commercial-sized operation.

ACKNOWLEDGMENTS

The authors wish to thank Shell Canada Limited for granting permission for the publication of this paper. Also, we wish to acknowledge the work done by the many Shell people who contributed to the development of this process.

PROJECT OILSAND

M. L. NATLAND*

ABSTRACT

Project Oilsand is a proposed experiment in the peaceful use of nuclear energy as an aid in producing oil from the McMurray oil sands buried too deeply to permit economic extraction of oil by mining methods. A 9-kiloton nuclear device would be detonated 20 feet below the McMurray oil sand at a depth of 1250 feet. The proposed test site is in a remote undeveloped area about 64 miles south of Fort McMurray.

Since the explosion will be completely contained underground, there will be no fallout. Most of the fission products will be effectively entrapped in a vitreous slag concentrated at the bottom of the blast cavity while the remainder will be confined to the immediate vicinity of the explosion by a process of ion exchange whereby they are retained by the rock minerals, thereby becoming unavailable to contaminate oil or water.

The seismic effects will cause no damage to surface installations of which there is only one, a railroad siding 6 miles away, and Fort McMurray will feel no shock whatsoever.

The contained explosion will create a cavity about 230 feet in diameter into which several million cubic feet of oil sand will fall and be heated to temperatures at which the oil may be recovered by conventional oil-field methods. Additional oil will be made recoverable by the shock energy from the explosion.

Safety measures have been developed which will prevent any accidental detonation, and the test site will be monitored for radioactivity before and after the test.

After the explosion, as soon as conditions permit, a hole will be drilled to the vicinity of the detonation point to determine the effect of the blast on the oil. Any oil produced will be carefully inspected for radioactivity.

INTRODUCTION

Project Oilsand is a proposal made by Richfield Oil Corporation and its associates, Imperial Oil Limited and Cities Service Athabasca, Inc., to use the tremendous energy released by a completely contained underground nuclear explosion in an experimental effort to produce oil from the vast reserves of the deeply buried McMurray oil sands in the Athabasca area of Alberta. It was estimated by the Alberta Technical Committee that only about 2 per cent of these oil sands has a sufficiently small overburden to permit the use of surface-mining methods. Although many schemes using well-known techniques to recover the oil from the deep sands have been tried, none so far has succeeded. Project Oilsand represents a new technological approach to an old problem.

The basic idea of Project Oilsand first occurred to me in the desert of southern Arabia where I was on a geological expedition.

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*Richfield Oil Corporation, Los Angeles

One evening as I sat watching a spectacular sunset from a small hill overlooking a flat, endless sea of sand, the sun looked like a huge orange-red fireball sinking gradually into the earth. It is well recognized that the sun's energy is produced by the same thermo-nuclear reaction which occurs when a hydrogen bomb is exploded; namely, that at temperatures on the order of one million degrees, hydrogen isotopes fuse to form helium, liberating extra mass as energy (Glasstone, 1957). As a synthesis of these thoughts took place, it became so apparent to me that the energy released by a nuclear explosion deep underground could be used to produce oil that I made a sketch in my field notebook of how this might be done.

Careful scientific study of this nuclear-explosive recovery method indicated that it might very well overcome the major obstacles which in the past have blocked oil recovery from the deeply buried McMurray sands. The major production problem is the natural viscosity of the oil which is hundreds of times greater than that of most other oils, a fact which makes it virtually immobile in its underground state. It has long been recognized that the most promising way of freeing the oil from the sand would be to heat it. This is easily done in a laboratory, but deep underground the impermeable nature of the sand when plugged with viscous oil prevents the effective circulation of heat necessary to make commercial oil production possible. However, the tremendous heat and shock energy released by an underground nuclear explosion would be distributed so as to raise the temperature of a large quantity of oil and reduce its viscosity sufficiently to permit its recovery by conventional oil-field methods.

GENERAL PHENOMENOLOGY OF UNDERGROUND NUCLEAR EXPLOSIONS

As the result of an extensive testing program for the U.S. Atomic Energy Commission conducted by the University of California Lawrence Radiation Laboratory*, the general phenomenology (Johnson, 1959; Johnson and Violet, 1958; Sanders, 1962) of an underground nuclear explosion is well known. A few millionths of a second after detonation, temperatures rise exponentially to millions of degrees, vapourizing and melting the surrounding rock, and the superheated gases at pressures of several million atmospheres radially expand to create a cavity. The cavity is lined with molten rock containing most of the radioactive fission products. The shock progressing beyond the cavity continues to crush rock for some distance and finally degrades into a seismic wave. While the cavity is still standing, some of the molten rock lining drips from the ceiling and runs down the sides to collect at the bottom. As the gases cool, the cavity pressure drops, and

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*Hereafter referred to by the initials UCLRL the molten rock solidifies into a vitreous slag which effectively entraps most of the radioactivity. When further cooling causes the pressure to decline below that required to support the overburden, the cavity collapses and fills with a large quantity of crushed and broken rock under which most of the slag lining is buried. In this way the radioactive material is concentrated at the bottom of the cavity. The configuration of the collapsed zone depends upon the type of overburden. The practical result of the explosion is that a large quantity of rock has been crushed, broken and heated to high temperatures with the bulk of this rock relatively uncontaminated by radioactivity.

THE PROPOSED PROJECT OILSAND EXPERIMENT

There is in the Athabasca area of Alberta an excellent site (Fig. 1) for the proposed detonation of a completely confined 9-kiloton nuclear explosive in an experimental effort to produce oil from the deep McMurray sands (Fig. 2). Here the country is remote, undeveloped and devoid of surface culture, while the amount of oil in place is large enough to warrant the test and buried deeply enough

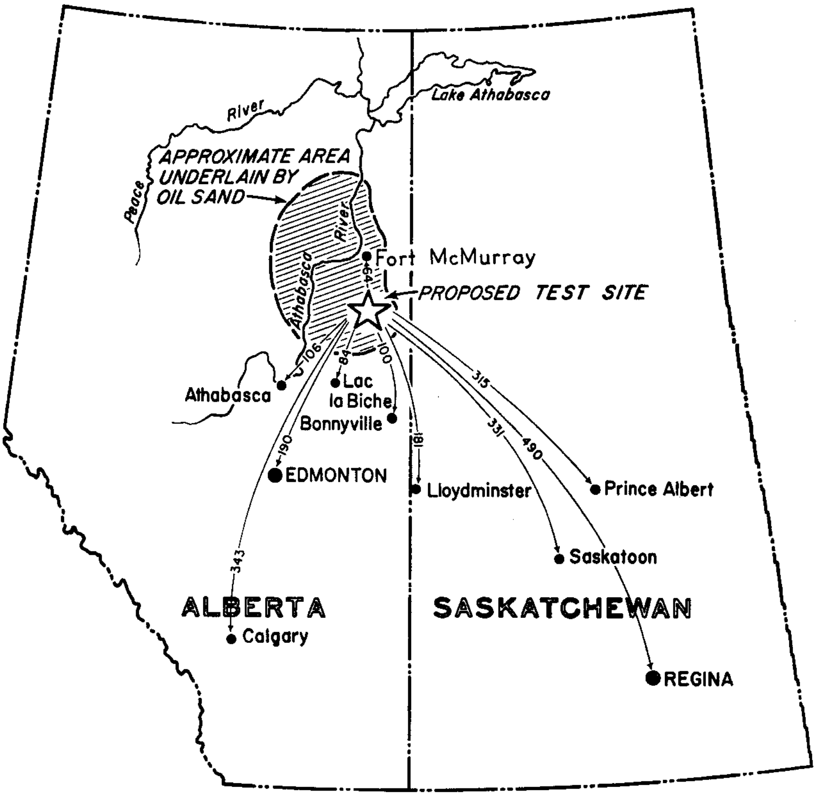

FIGURE 1. Distances in miles of some cities and towns in Alberta and Saskatchewan from the proposed nuclear test site.

so that the explosion would be fully contained. For Project Oilsand a nuclear device with a yield rated at 9 kilotons (9 trillion calories) would be detonated in silty limestone (Beaverhill Lake Formation) 1250 feet underground and 20 feet below the base of the McMurray oil-sand formation (Fig. 3). Data resulting from extensive testing and research by the UCLRL for the USAEC* indicate that the nuclear-explosive method of oil recovery proposed in the Project Oilsand experiment would be safe, that there would be no damage from seismic effects, and that there would be no detrimental contamination of the oil or of water by radioactive fission products. It is hoped that this method will point the way to producing oil from the deep McMurray sands on a commercial scale.

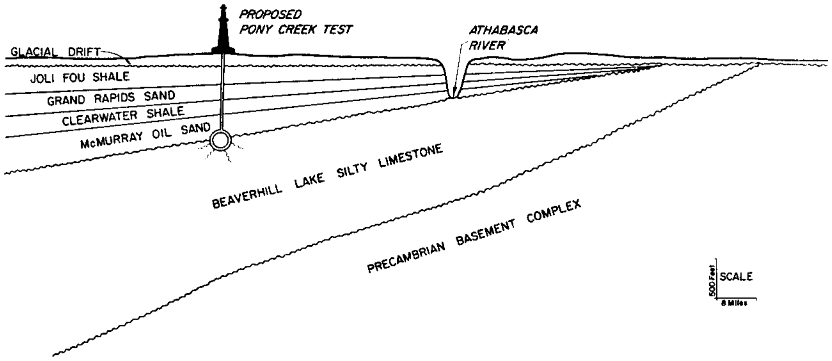

FIGURE 2. Diagrammatic geologic cross section through proposed test site at Pony Creek.

TEST SITE

A tentative location for the Project Oilsand test site has been selected 6 miles northwest of the Chard railroad siding which provides the only surface access to the general area (Fig. 1). The country here is mostly swamp and muskeg during summer and a frozen wilderness in winter. After considerable investigation, this location was selected as a desirable test site for the following reasons:

(1) The area has no surface culture, such as farms, dwellings, towns, etc.

(2) There are no developed oil fields which could be disturbed by seismic effects of an underground detonation.

(3) The mineral and surface rights are held under one ownership, that of the Provincial Government of Alberta, thus simplifying operational and legal matters.

(4) The estimated amount of oil in place is sufficiently large to warrant the experiment.

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*United States Atomic Energy Commission

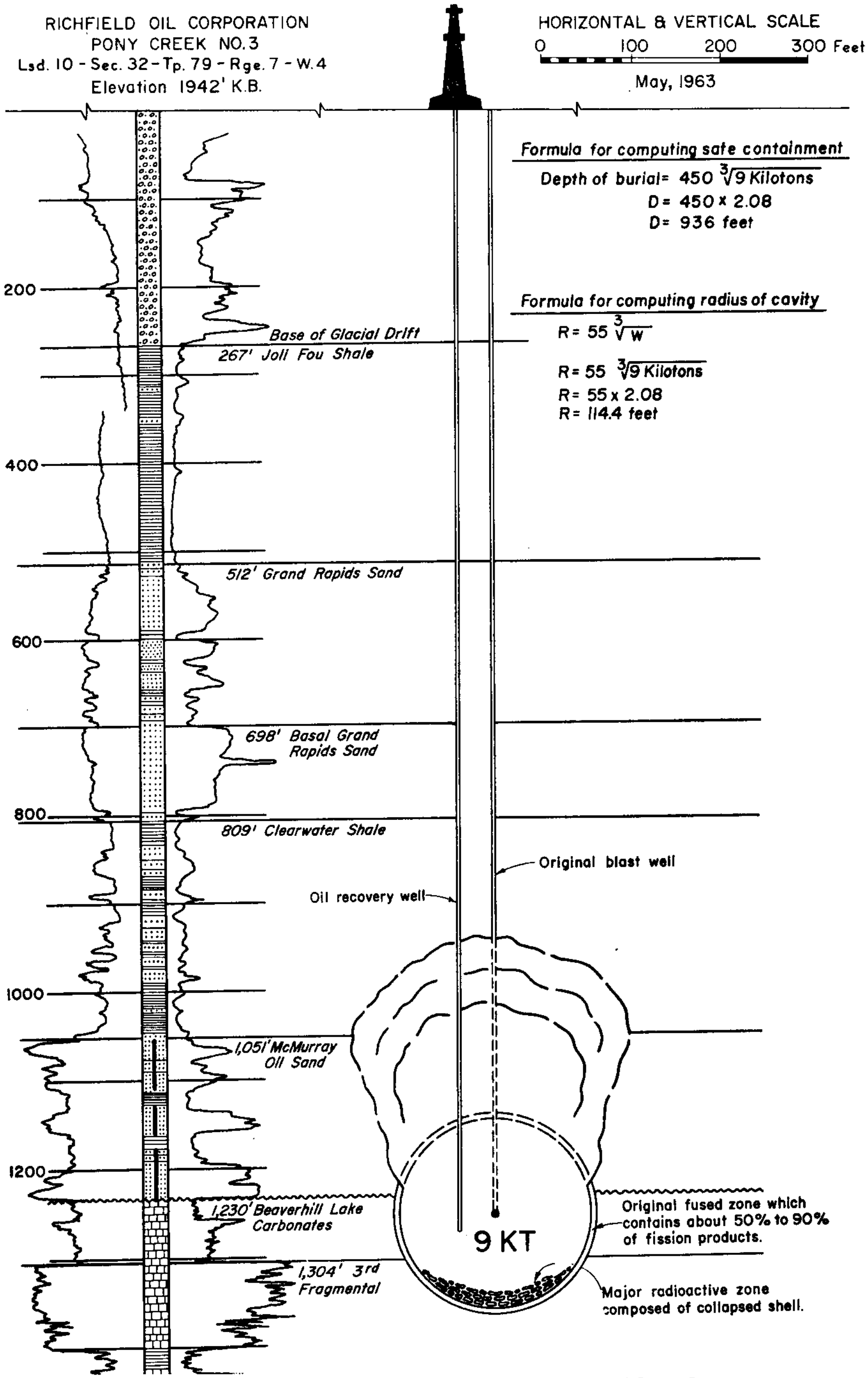

FIGURE 3. *Diagram showing, a log of Richfield Oil Corporation Pony Creek No. 3 well, the proposed stratigraphic location of the 9-kiloton nuclear device below the oil sands, the estimated size of the cavity after detonation, the collapse zone and proposed location of oil-recovery well.*

(5) The sands are buried deeply enough to contain completely a nuclear explosion with an energy yield sufficient to heat a large volume of the viscous oil to temperatures which may permit recovery.

(6) The oil is of good quality, and by processing, a wide range of petroleum products can be derived.

CONTAINMENT

A 9-kiloton device has been proposed because it is large enough to provide the data required to evaluate the test but would be completely contained at 1250 feet, allowing a very generous safety factor. Since the explosion will be completely contained and no radioactive debris will escape into the atmosphere, no question of fallout is involved in the proposed experiment.

The safe containment formula developed by the UCLRL from the Rainier test is: depth $(D)=450\ W^{1/3}$ feet, where W is expressed in kilotons (Johnson and Violet, 1958). For a 9-kiloton device, this formula predicts that $D=450 \times 2.08=936$ feet for complete containment. In fact, a 21-kiloton device would be completely contained at 1250 feet ($D=450 \times 2.76=1242$ feet). However, subsequent testing by the UCLRL has shown this factor of 450 to be extremely conservative. Johnson (1959) in discussing Project Oilsand states, "The sands occur in beds 100 to 300 feet thick at depths up to 1200 to 1500 feet. At such depths, 30 to 40 kilotons could be fired without disrupting the surface, and perhaps as much as 100 kilotons could be used."

In addition, it is anticipated that the resilient nature of the McMurray oil sand and of overlying Clearwater plastic shale beds will tend to reduce the upward transmission of energy from the blast. From the foregoing, it is quite evident that the safety factor allowed for the proposed 9-kiloton explosion is at least more than 2 and may be as great as 10.

CAVITY SIZE

The UCLRL formula, radius$=55\ W^{1/3}$ feet (Higgins, 1959b), applied to a 9-kiloton device, indicates that a cavity about 230 feet in diameter would be formed. The size of the cavity produced in the silty limestone of the Beaverhill Lake Formation is expected to be about the same proportionately as those created in the volcanic rock of the Nevada Test Site because the force of the explosion is so enormous that cavity size will probably be controlled more by factors such as formation water saturation and the weight of overburden than by the range in the compressive strength of the rock.

CAVITY COLLAPSE

After a time ranging from a few seconds to a few minutes, the roof of the cavity formed by the 9-kiloton explosion is expected to collapse from the weight of the overburden. When this occurs, several million cubic feet of oil sand will fall into the cavity where the oil will be heated sufficiently to be recovered by conventional methods.

ENERGY RELEASE

According to calculations, a 9-kiloton device will relase one half of its energy, or 4.5 trillion calories, as heat in ranges capable of reducing the viscosity of the oil so that it can flow or be pumped through recovery wells to the surface. The remaining energy is released in the form of shock waves which pass with supersonic speed through the oil sand and other formations. Recent experiments with this type of high-pressure shock applied to McMurray oil sand indicate that pressures above the 20-kilobar range will crack oil. Therefore, the shock-cracked oil will be a substantial addition to that made recoverable by purely thermal effects.

FISSION PRODUCTS

As the result of extensive testing and research by the UCLRL, the disposition of radioactive debris produced by a fully contained underground nuclear explosion is quite well understood (Higgins, 1959a, b). Following is a description (Johnson *et al.* 1959) of this disposition as it applies to the 9-kiloton explosion proposed for Project Oilsand.

The only place where fissioning occurs to create radioactive isotopes is within the explosive device where neutrons split the fissionable atoms of uranium. The few delayed neutrons which escape the explosive capsule are absorbed before they can travel more than a few feet. At this stage of the explosion, there is still sufficient limestone between the area of the neutron generation and the nearest oil sand to prevent contamination of the oil by neutron activation.

The radioactive fission products formed at the instant of explosion mix with the material within the rapidly expanding gaseous fireball. When the sphere reaches its maximum diameter, between 50 and 90 per cent of the fission products are dissolved in the cavity lining formed by the cooling of the molten rock. The exact amount expected to be dissolved is uncertain since it will depend to a large extent on the temperature of the molten rock and the time that the cavity exists before collapse.

While the cavity is still standing, some of the molten lining containing fission products rains down in the form of glass droplets from the roof area, and some runs down the sides to collect in a pool at the bottom. Further cooling causes much of the lining which remains to fall so that most of the slag with the fission products is confined at the bottom of the cavity in a localized area. Consequently, when a general collapse of the cavity roof occurs, only a small amount of the oil sand tumbling down ever comes into contact with the slag containing radioactive particles. Furthermore, the vitreous nature of the slag will reduce the possibility of introducing objectionable levels of radioactivity into the oil.

Tests have been made to determine whether or not the silty limestone in the Athabasca area would be melted into a slag capable of reducing the availability of fission products to oil or water. Cores of silty limestone recovered from test holes drilled in the area were ground to fine powder. This powder was then pelletized under high pressure in a hydraulic press to make small pellets about 1 inch long. The limestone pellets were then heated in an arc furnace to a temperature of about 2500°C. This temperature was sufficient to fuse the pellets into glassy material which could be tested for solubility. It has been found that this silty limestone does form a slag capable of retaining fission products and thereby reducing their availability to contaminate oil or water.

The fission products not dissolved at the time of cavity collapse take the form of gases, chiefly radioactive isotopes of krypton and xenon. These isotopes have half lives of less than a few minutes, but this is long enough to prevent them from being dissolved in the slag. The radioactive krypton and xenon rapidly decay to longer-lived radioactive elements, Sr^{90} and Ce^{137}. Fortunately, by a process of ion exchange, the Sr^{90} and Ce^{137} take the place of other elements in the medium and are retained by the rock minerals in the vicinity of the explosion, thereby becoming unavailable to contaminate oil or water.

The rate of groundwater flow was determined by recording pressures at several wells drilled in the general area of the proposed experiment. Through the use of standard pressure-recording equipment, water pressures were recorded on charts and were found to be quite uniform over a wide area. From this and other data, the rate of water movement through the McMurray and Grand Rapids Formations was calculated to be of the order of a few feet per year. This slow rate of water movement together with the adsorption characteristics of the earth minerals effectively limits the transfer of radioactive fission products from the immediate vicinity of the detonation cavity through aquifers. Therefore, it is evident that there will be no water contamination detectable outside of the immediate area of the explosion.

The net effect of all these factors is that there will be no contamination of the oil or groundwater above acceptable levels.

OAK RIDGE REACTOR RESEARCH

When Project Oilsand was first being considered and before the extensive UCLRL data on the disposition of radioactive debris, particularly in regard to ion exchange, were available, it was considered necessary to determine what effect high-level irradiation might have on oil from the McMurray sands. Therefore, six samples were placed in the experimental reactor at Oak Ridge, Tennessee. It is important to note that a sample irradiated in this reactor for one-half hour would receive the approximate equivalent of exposure to all neutrons from an unshielded 10-kiloton device at a distance of 100 feet. These six samples were irradiated for times varying from 5 hours to 2 weeks. The maximum milliroentgen count was 6000 per hour after 44 hours of irradiation. Seven and one-half days later the milliroentgen count had dropped to 5 per hour, which is about the same as that emitted from a luminous watch.

This rapid drop in emissions clearly indicates that the oil received no long-lived detrimental effects even when exposed to radiation at levels vastly higher than those which will be available from the radioactive debris of the proposed 9-kiloton explosion. In any event, no oil will be transported or marketed unless it is uncontaminated or can be decontaminated to meet public health standards.

SEISMIC EFFECTS

The seismic effects will cause no damage because the surface motion will be of a low order of magnitude. Also, there are no installations in the immediate area to be damaged. Guided by data from both underground nuclear and high explosive shots, Griggs (1959) stated, "I have carefully read the report of damage from the Rainier, Logan and Blanca shots. Damage was limited to less than one-half mile from the shot point—even in the case of Blanca (20 kiloton)."

Since the roadbed of the Northern Alberta Railroad will be at least 6 miles from the test site of the proposed 9-kiloton detonation, it is realistic in view of the foregoing to expect no damage to the railroad. The nearest town, Fort McMurray, 64 miles away will feel no seismic shock whatsoever.

OPERATIONAL PLAN

If the Project Oilsand experiment is permitted by Provincial and Federal authorities, the following operational plan will be enacted.

Pre-shot

A permanent road will be built from the railroad siding at Chard to the proposed test site located 6 miles to the northwest. A hole will be drilled capable of receiving a 38-inch outside diameter casing which will be cemented in place to accommodate the 34-inch 9-kiloton nuclear device. The UCLRL advises that the nuclear device will be transported from the United States to Canada in such a way as to prevent an accidental detonation and that safety measures will be taken to assure that no nuclear explosion is possible except at the bottom of the hole. The well will be conditioned to accommodate the stemming plug designed and recommended by Dr. G. T. Pelsor of the

UCLRL. After installation of seismic and other necessary scientific instrumentation, the shot will be fired. Measures will be taken to assure that appropriate radiological observers will monitor the test site before and after the detonation. Co-operation is planned with various university seismology laboratories and other interested scientific groups to monitor the Project Oilsand seismic signal because such data should provide much valuable information on the crustal structure of the Canadian Shield and surrounding area.

Post-shot

As soon after the detonation as conditions permit, an oil-recovery hole will be drilled to the McMurray sand level about 500 feet from the detonation center. Here the bottom of the recovery hole will be about 385 feet from the cavity boundary, a distance which is far enough to ensure that any residual gas pressure will be controllable. The hole will be monitored for heat, radiation, gas pressure and fluid oil. When conditions warrant, wells will be drilled progressively closer to the cavity area. All rock fragments, oil, water and gas will be monitored for radiation during all post-shot operations. If at any time radiation levels are found to be above safe limits, operations will cease until either the radioactivity has decayed to safe levels or proper means for isolating the contaminants can be installed.

Oil Refining Study

Safe and adequate tank facilities will be provided to store oil at the test site. The recovered oil will be subjected to extensive chemical and pilot-plant refining tests.

Logistics

Camp and transportation facilities will be provided for all personnel directly connected with all phases of the drilling, detonation and oil recovery program. Richfield and its associates will reimburse the USAEC for the cost of the nuclear device, including its transportation, installation and detonation, to the extent required.

HISTORICAL BACKGROUND

On May 9, 1958, Richfield representatives met with Commissioner Willard F. Libby of the USAEC and his aides in Washington, D.C., to discuss the proposal to use the energy released by an underground nuclear explosion in an experimental effort to produce oil from the deeply buried McMurray oil sands. At Dr. Libby's suggestion, this proposal was discussed with Dr. Edward Teller, Dr. Harold Brown and Dr. Gerald W. Johnson at the UCLRL in Livermore. As a result of this and other discussion, the USAEC agreed that the proposal had merit and that they would support Richfield's request for a nuclear device when made to the proper U.S. authorities. As part of the "Frontiers in Atomic Energy Research" hearings, on March 22, 1960, Mr. W. J. Travers, Vice-President of Richfield, described the Project Oilsand for the Subcommittee on Research and Development of the Joint Committee on Atomic Energy of the U.S. Congress in Washington, D.C.

After tentative USAEC support had been secured, meetings were scheduled with the Provincial Government in Alberta. The first meeting was with Mr. H. H. Somerville, Deputy Minister of Mines and Minerals, on June 5, 1958, in Edmonton. On the following day, the Project Oilsand proposal was presented to Dr. George W. Govier of the Oil and Gas Conservation Board of Alberta. On January 13, 1959, the proposal was formally presented to Premier E. C. Manning and the Executive Council of Alberta. A press conference under the chairmanship of the Hon. A. R. Patrick, Minister of Economic Affairs, was held in Edmonton on February 13, 1959.

The first presentation to the Federal Government was made in Ottawa on June 26, 1958, to Dr. John Convey, Director of Mines Branch, Department of Mines and Technical Surveys, and a group of technical people. After several meetings with this group, a conference was arranged with ministers, deputy ministers and other interested Federal Government officials on January 26, 1959.

These meetings in Canada resulted in the establishment of two technical committees to study the proposal, one for the Province of Alberta and the other for the Government of Canada.

In February 1959, the following were appointed by Premier Manning to serve on the Alberta Technical Committee:

Dr. G. W. Govier (Chairman), Oil and Gas Conservation Board of Alberta, University of Alberta, chemical engineer.

Mr. D. R. Craig, Oil and Gas Conservation Board of Alberta, petroleum engineer.

Dr. D. A. L. Dick, Department of Public Health, therapeutic radiologist.

Dr. G. Garland, University of Alberta, geophysicist.

Dr. C. P. Gravenor, Research Council of Alberta, geologist.

Dr. H. E. Gunning, University of Alberta, chemist.

Mr. A. F. Manyluk, Oil and Gas Conservation Board of Alberta, petroleum engineer.

Mr. Grant MacEwan, M.L.A., lawyer. (Mr. MacEwan resigned from the Committee July 8, 1959.)

The Hon. A. R. Patrick, Minister of Economic Affairs, Government of Alberta.

Mr. H. H. Somerville, Deputy Minister of Mines and Minerals, Government of Alberta.

After considering all aspects of Project Oilsand, the Alberta Technical Committee submitted a report, dated August 1959, to the Minister of Mines and Minerals and the Oil and Gas Conservation Board recommending that the Lieutenant Governor in Council approve the project and that the necessary well licenses and approvals be issued subject to the terms set forth in the report.

However, in addition to Provincial approval, the Project Oilsand experiment must also be approved by the Federal Government which controls the use of nuclear materials. In March 1959, the following were appointed by the Hon. Paul Comtois, Minister of Mines and Technical Surveys, to serve on the Federal technical committee, called the Joint Technical Feasibility Committee:

Dr. John Convey, Chairman), Director, Mines Branch, Department of Mines and Technical Surveys, chemist.

Dr. A. H. Booth, Occupational Health Division, Department of National Health and Welfare, physical chemist (radiation safety officer).

Dr. G. W. Govier, Oil and Gas Conservation Board of Alberta, University of Alberta, chemical engineer.

Dr. W. E. Grummitt, Atomic Energy of Canada, Limited, chemist.

Dr. J. H. Harrison, Director, Geological Survey of Canada, Department of Mines and Technical Surveys, geologist.

Mr. A. Ignatieff, Chief, Fuels Division, Mines Branch, Department of Mines and Technical Surveys, mining engineer.

Mr. A. F. Manyluk, Oil and Gas Conservation Board of Alberta, petroleum engineer.

Dr. M. L. Natland, Manager of Production Research, Richfield Oil Corporation, geologist.

Dr. Robert J. Uffen, University College, University of Western Ontario, geophysicist.

The first meetings of the Joint Committee were held at the USAEC Test Site at Mercury, Nevada, and at San Francisco, California, on May 10-15, 1959. All members of the Committee were conducted through the labyrinth of tunnels driven through the cavity areas created by Rainier (1.7 kiloton) and Blanca (20 kiloton) underground nuclear explosions. At San Francisco the Committee attended the

USAEC Second Plowshare Symposium devoted entirely to the peaceful uses of nuclear explosions and their phenomenology which were discussed in detail. The Committee also prepared a list of "Pre-Approval Requirements" for Project Oilsand which included a request by the Alberta Technical Committee for additional technical data together with details of the proposed drilling and operational programs.

A report (Natland, 1959) containing the requested information was completed. It was submitted to the Alberta Technical Committee and also to the Joint Committee which reviewed it in detail on July 20-21, 1959, in Ottawa. At the close of the meeting on July 21, the Joint Committee agreed that the "Pre-Approval Requirements" had been satisfied and the Committee would proceed with the writing of a report recommending to the proper authorities that Project Oilsand be approved. Completion of the Joint Committee's report has been held in abeyance.

The international moratorium on atomic testing and subsequent Canadian Government policies have prevented the experiment (Bellows and Bohme, 1963).

It is hoped that obstacles blocking the progress of Project Oilsand can be removed and that this experiment in the peaceful use of nuclear energy can take place.

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SOME ASPECTS OF HIGH-CAPACITY PRODUCTION WITH BUCKET-WHEEL EXCAVATORS IN OPEN PITS

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ABSTRACT

This paper discusses several design aspects of open-pit mining employing large bucket-wheel excavators capable of production rates up to 130,000 bank cubic yards per day. An analysis of the service factor is used to predict the excess production capacity which must be allowed for in both the excavator and the conveyors, as well as the necessary storage capacity. Operational applications of bucket-wheel excavators and causes for the periodic fluctuations in machine output are described. Bucket-wheel excavators are well suited to oil-sand mining in areas subject to extreme weather conditions and heavy frost.

INTRODUCTION

This paper compares the anticipated conditions of oil-sand mining in the Athabasca oil sands by means of bucket-wheel excavators with the known conditions encountered during many years of open-pit mining of brown coal in Germany with the same equipment. Excavators which have been used in Germany for several years are capable of producing quantities of the order of 130,000 bank cubic yards per day of either overburden or brown coal. Surface mining of oil sand could be an excellent application of these wheels of large production capacity. The brown coal mined in Germany is used for power production and is derived from several pits employing many bucket-wheel excavators. Storage of the coal is practical and is carried out in this operation. However, it appears that storage of oil sand may be impractical, thus constant production in the mining operation is necessary.

The Athabasca oil-sands mining operation will consist of overburden stripping plus oil-sand mining and, in this aspect, it is analogous to the coal mining in Germany. However, it may be necessary to excavate some material in the ore body which may be undesirable for plant feedstock.

In any mining scheme, the ore transportation system is a vital link and must be carefully designed. Although many forms of transport have been employed in German coal mining, currently conveyor belts seem most economical. The pits now using large bucket-wheel excavators are being converted to belt conveying. It is assumed that these belt conveyors would show the same advantage in oil-sand mining and are proposed as the transportation system.

An open-pit mine should have two separate excavating and transporting systems; the primary one for the plant feedstock, which in this instance is sand, and the secondary one for overburden. The

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primary system must be designed and operated with respect to the feed requirements of the beneficiation plant, whereas the secondary system is usually independent of the primary system. The degree of freedom of the second system is somewhat limited, in that sufficient overburden must be removed so that it is not in any way a limiting factor on the production of the primary system. This paper discusses the primary system; the equipment for mining and transporting oil sand, and it is assumed that most of the comments can also be applied to the equipment for handling overburden.

One of the most important premises from an economic standpoint is that the mine layout should incorporate minimum equipment operating at maximum production. At first glance, a simple solution would be to operate one wheel-excavator continuously at the desired production rate, in conjunction with one belt-conveyor. However, experience gained in Germany on large-scale installations has shown that a more complex scheme is necessary.

SERVICE FACTOR

It is impossible to operate any mechanical equipment one hundred per cent of the time for any extended period. If the beneficiation plant requires continuous feed, it is necessary to predict how much time the excavators and transport system will operate. The ratio of actual working time of the equipment to total working time is generally defined as the service factor. As an example, if the equipment could be operated only 18 hours a day, the service factor for that day would be 75 per cent; although service factors are generally quoted on a yearly basis. The service factor is inversely proportional to the down time of the equipment. Common causes of down time in the coal-mining applications of bucket-wheel excavators in Germany during the past several years are: (a) mechanical and electrical breakdowns of excavators and transport facilities; (b) natural phenomena causing interruptions such as weather, major variations in the ore body, slides or failures in the quarry walls; (c) shut-downs of the beneficiation plant. Shut-downs of the beneficiation plant will not be considered for the oil-sand application, since it is assumed that it would be more advantageous to increase the service factor of the beneficiation plant rather than provide over-capacity in the excavating and transporting facilities. However, it is apparent that the first cause of stoppage (Item "a") will apply in the tar sands and in all probability the second (Item "b") will cause some stoppage. For example, in Germany, during high winds the large bucket-wheel excavators have to be shut down.

The best way for predicting the shut-downs due to mechanical failures is to review the performance of similar large equipment which has been in operation for several years. A statistical evaluation of performance data recorded over one year's operation of power shovels, draglines (Geissel *et al.*, 1961) and wheel excavators has shown that the interruptions caused by maintenance work represent a minimum of 15 per cent of the working time, an average of 21 per cent and, in some instances, up to 30 per cent. With the average figure of 21 per cent, it is conceivable that it could take place in two different ways, the first being a shut-down occurring regularly each day of approximately 5 hours duration, and the second being one extended shut-down during the year, of 77 continuous days. It is highly unlikely that the down time will take place in exactly either form but will, for both unforeseen reasons and planned maintenance considerations, be somewhere in between. These considerations, therefore, demonstrate why an excavator cannot provide design production for 100 per cent of the time. The belt conveyors which transport the material from the bucket-wheel excavator to the beneficiation plant are also mechanical equipment and are subject to breakdowns independently of the excavating equipment.

As previously discussed, large, high-capacity, high-speed movable conveyor belts have been installed recently in conjunction with bucket-wheel excavators in the coal mines in Germany. It is obvious that the conveyors must have high capacity to handle the output of the bucket-wheel excavators. German experience has shown that operational difficulties increase with increased belt capacity and haul distance. These conveyors are markedly different from the conventional type for which service factor can be fairly accurately predicted on the basis of long experience.

Conveyor	Belt Width	Handling Capacity
Standard type	47 inches	3,300 bulk cu. yds. per hour
High-capacity	78 inches	9,500 bulk cu. yds. per hour

Although experience with standard conveyors has shown down times of 5 to 10 per cent over long operating periods (Scharf, 1954), it appears unlikely that this low amount of down time could be realized with high-capacity types in light of operational experience to date.

An obvious approach to minimize down time is preventive maintenance—scheduled servicing, inspections and overhauls. Schedules should be set up for all maintenance jobs, for routine inspection of specific electrical, mechanical, and structural parts, and regular replacement of high-wearing parts. This type of maintenance might reasonably take 24 hours per week, more or less, depending upon the amount of high-capacity operation of the equipment. This preventive maintenance time then results in 52 days or 1,248 hours per year. On the basis of an average of 21 per cent maintenance time, this leaves 25 days or 600 hours per year for repairing unforeseen breakdowns. The author is not aware of any data dealing with the practical effects of a preventive maintenance program, but such a high-cost program should result in considerably less than 600 hours per year of lost time due to breakdowns. No doubt a careful study of a particular installation for many years could result in the most realistic preventive maintenance policy to balance its cost and efficiency. Preventive maintenance on the conveyor belts could be carried out concurrently with similar work on the excavators.

Most shutdowns due to unforeseen circumstances last 24 hours or less. However, in some instances, the period may be somewhat longer, as for example, in a breakdown of essential machinery such as gearings, drive pulleys, motors, etc., which cannot be replaced or repaired within 24 hours. Interruptions due to natural causes could have fairly extended periods, although it is hoped that their frequency would be very low.

SYSTEM SERVICE FACTOR FOR THE MINING AND CONVEYING COMPLEX

With the assumption that continuous feed to a beneficiation plant is a process requirement, the mining and conveying system could be designed to ensure plant feedstock requirements. The following measures could be used to accomplish this: (a) stockpiling of material in so-called surge capacity; (b) multiple trains of excavators and conveying systems; (c) surge capacity in excavator output and conveyor transport capacity plus allowing delivery of feedstock in excess of normal feedstock rate. Surge-equipment capacity means that the beneficiation plant would receive a variable feed rate resulting in a costly and complicated plant, if the feedstock rates were highly variable. On the other hand, if material were stockpiled, surge-equipment capacity must be provided to ensure steady plant feedstock while stockpiles are replaced. Perhaps a combination of these measures would be most desirable. However, an analysis of the anticipated production interruptions can be used to evaluate the proper system-design features. Comparison of the respective investment and operational costs could be used to arrive at the correct system.

It is common engineering practice to provide storage capacity in many feedstock streams. However, few feedstocks are delivered in the quantities which the large bucket-wheel excavators are capable of supplying. If the feed rate were 130,000 bank cubic yards per day of material or more, only a few hours' production rate would require an immense storage volume. Let us assume that it is feasible to stockpile 130,000 bank cubic yards of material, or one day's production, and further, that this stockpiling is more economical than an additional excavator. This means that one excavator plus storage would be installed and that the excavator output would be increased by 1/24, so that a completely depleted stockpile could be replenished during a 24-hour period while still maintaining normal plant feedstock production. Thus a shut-down of up to 24 hours in the mining and conveying systems could be tolerated without feedstock interruption. Subsequent to the 24-hour shutdown and stockpile depletion, sufficient continuous operation must replenish the stockpile before another major shut-down can be tolerated. As previously stated, most shut-downs will last 24 hours or less, and therefore this one excavator, conveyor, and storage system should allow for such interruptions.

If, however, analysis of the expected down time shows significant periods greater than 24 hours, an alternative to providing even more surge capacity than for 24 hours in a stockpile would be to provide a standby excavator with its own conveying system. If the standby equipment is provided, is the need for surge capacity in the stockpile obviated? As a demonstration of surging that can be expected, in the event of a breakdown, a production loss of 1,400 cubic yards can take place before the standby train can be started up. With two full-capacity excavator and conveying systems, practice should be to divide production between both trains as equally as possible so that each train produces 100 per cent of the feedstock during half the total working time. Normally, one train should be operated for from one to six days, depending upon the local mining conditions and other operating requirements. This procedure will be particularly helpful in the preventive maintenance program, the minimizing of operational personnel, the more uniform wear on mechanical equipment, a simplified mining procedure, and the possibility of adapting operations to varying conditions of the ore body.

Experience has shown that down times of approximately two hours per day can also be expected for the conveyor systems. If storage capacity is available in stockpile, it is more advantageous to take plant feed from the storage rather than start up the standby train of equipment. As the mining wheels advance into the ore body, there must be regular periodic conveyor shifts to accommodate the wheel movements. To facilitate wheel operation, German practice has been to assign each wheel its own individual pit conveyor. Some individual conveyors converge where the material is fed on to one single continuing conveyor; other individual conveyors are maintained to the final delivery point. In German coal-mining, individual pit conveyors have given the planning engineer more scope to work out the most advantageous mining operations for the varying conditions within the ore body.

The discussion thus far has been based on the assumption that a wheel excavator will produce a continous, uniform flow of material during its operating time. This in fact has not been the experience of wheel operators in the German coal fields. Fluctuations do take place with varying frequency and duration, such fluctuations normally lasting minutes or, at most, hours. The mining procedure which the wheel employs, as discussed in the next section, will give rise to periodic fluctuations which cannot be eliminated. Despite the extensive experience with large bucket-wheel excavators, the author knows of no accurate method of predicting the magnitude and frequency of the fluctuations. The wheel manufacturers recognize and consider this problem when calculating the average capacity of the excavator. The real difficulty arises when attempting to design the related conveyor system, since stoppages will result unless the conveyor can successfully handle peak flows. In the mining of brown coal in Germany, a great effort is made to know what size conveyors are best to handle the production from large excavators. It is anticipated that surging of oil sand will be somewhat different. Stockpiling of material can be used to even out these production fluctuations.

The foregoing analysis has brought out several points: shutdowns of 24 hours or less in duration could conceivably be handled by stockpiling of material; stoppages of greater than 24 hours' duration must be handled by providing duplicate trains for excavating and conveying; experience has shown that the best operational procedure is to operate one train at full capacity for short periods, while doing maintenance work on the other train; daily conveyor stoppages are best overcome by stockpiling material rather than by starting up the second train of equipment; inherent fluctuations in feed rate from the bucket-wheel excavators, which can only be overcome by using storage.

To achieve a high service factor in the proposed mining of oil sand by bucket-wheel excavators, it is therefore felt that the system should consist of two bucket-wheel excavators of 100 per cent capacity, each with its own conveyor train, and surge storage for production of approximately 24 hours. This conclusion is based on the assumption that a nearly constant feed rate to the beneficiation plant is necessary, and on experience in the operation of large bucket-wheel excavators in German coal mines. Mining of oil sand may be somewhat different.

DIGGING METHODS EMPLOYED BY BUCKET-WHEEL EXCAVATORS

Figure 1 shows a wheel excavator in an established cut. It is positioned to travel on the pit floor in line with the top edge of the high wall at which it is digging. As it advances, it leaves a new high wall on its right, in the direction of mining. Digging is done by rotating the wheel, swinging it from side to side in long arcs, and advancing it into the bank by travel of the whole machine (bench cut) or by lowering the boom (falling cut). Both methods are discussed below.

Modern mining machinery generally excavates in blocks. A block is measured by the width of the cut, its height from the floor to the top, and the horizontal depth of cut behind the face. This depth depends on the reach of the wheel boom and the slope of the face. For a big wheel excavator, a block may consist of five benches containing a total of 82,000 cubic yards. Excavating time for each block is about 15 hours.

BENCH CUT

Figures 2 and 3 show the progress of the digging wheel by curved lines, each one representing the face, or the bench, after one full swing of the wheel boom. Figure 2 illustrates a bench cut. This cut is started by moving the machine forward to a position in which the wheel can cut a thin vertical slice as the boom is swung in an arc between the old high wall and the new one. As the wheel reaches the end of its cutting arc, the whole machine is moved forward on its crawlers so that fresh material can be reached during the return swing. A new advance is made at the end of each swing in either direction, without stopping the wheel or changing boom height, until the tracks reach the toe of the bank or the bottom of the boom almost touches its edge. The machine is then backed so that the buckets can dig the bank directly below the bench it has just cut. Swinging and advancing are resumed and continued until the bench is completed. Successive benches are cut in the same manner until the floor is reached, at which time the wheel is raised and the excavator moved forward to start the top bench in the next series. The arc of swing moves toward the pit teach time the wheel is lowered, due to the slope of the high walls. The swing must be shortened to the right to give the new wall a slope, but it is lengthened to the left by the old slope (Fig. 1). The slices are thinnest at the bottom, where the teeth enter the soil, and thickest at the top where

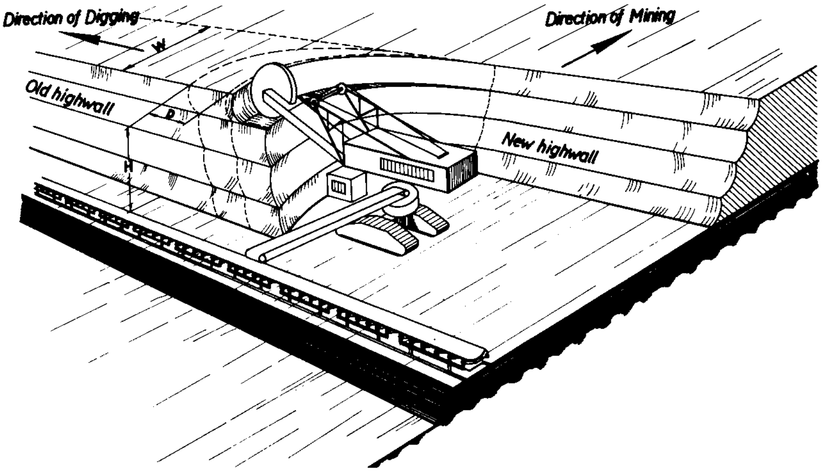


FIGURE 1. Block-digging operation with wheel excavator feeding onto a movable conveyor belt.
H=Height of face; D=Depth of block; W=Width of block.

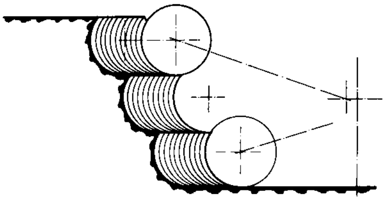


FIGURE 2. Digging method by bench cut.

they break out. With certain classes of hard material the shallow cutting-angle may cause the teeth to ride the face without penetrating it. However, this difficulty can be overcome by using a falling cut.

FALLING CUT

In the falling-cut method, shown in figure 3, the teeth of the wheel enter the bank almost at right angles. Thus, the full wheel-drive is used to achieve penetration. This method is chiefly employed to improve cutting efficiency in hard materials where a steep slope can be used. For a vertical face, as shown, it requires much less crawler travel than does the bench cut.

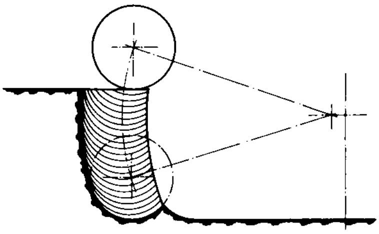


FIGURE 3. Digging method by falling cut.

DIGGING FACTORS

The operator controls digging action by setting the wheel speed and adjusting the swing when necessary. Either crawler travel or wheel hoist, depending on whether a benching or a falling cut is employed, determines the depth of the cut. The big machines work without an independent crowd mechanism: the crawlers are used in the event that crowd is necessary. Wheel rotation and wheel swing may be regulated for variable speeds. It is common practice to select the cutting speed for a given formation on the basis of trial runs, and then to make most adjustments for digging conditions by varying the rate of swing. In either of the digging arrangements described, the cut is deepest on the pit side and thins out to zero at the new wall. If swing speed were uniform, production would vary over the arc of swing. However, output is kept as steady as possible by increasing the rate of swing where the cut is thinner. This can be done manually or automatically.

Swing speed can also be varied to adjust to local changes in the hardness of the formation.

The required digging force in any one formation is largely determined by the cross-sectional areas of the slices cut by each bucket. Figure 4 shows how doubling of the wheel speed halves the cutting section needed for a given production rate. High rotary and low swing speeds are therefore well suited to hard formations. The operator will usually obtain maximum production when the wheel is turning at the highest speed at which the buckets can be filled without breaking out and spilling chunks. He can be guided in his swing speed by the current readings of the wheel-drive motors.

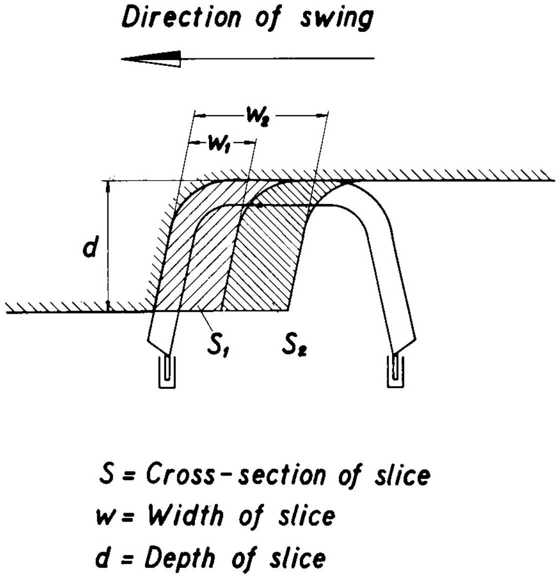


FIGURE 4. Cross section of wheel bucket when cutting a slice.

APPLICATION OF THE BUCKET-WHEEL EXCAVATOR TO OIL-SAND MINING

There are several unique features of the bucket-wheel excavator which make it particularly attractive as a tool for oil-sand mining. The very high production rate of this equipment is most advantageous, particularly when considering operating labor force. However, other important features become apparent when considering the method with which the wheel excavator mines as opposed to other large-capacity mining equipment such as power shovels or draglines.

HARD DIGGING CONDITIONS

The McMurray Formation is an oil-impregnated sandstone which will require high digging forces. These high digging forces must be applied at all points in the formation from top to bottom. In general, the McMurray Formation is 200 feet or less in thickness, with an average of approximately 150 feet. These overall dimensions are roughly similar to those in the coal fields where large bucket-wheel excavators are currently being used. Since the machine can reach any point on the face while standing at the base of the high wall (Fig. 5), and the main gearing is mounted on the bucket-wheel shaft, it is possible to utilize the total installed drive power in any working position, so that the maximum cutting force will be available at any point on the face. If the maximum permissible torque is exceeded, the safety clutch interrupts the connection between the motor and the gearing.

Although the McMurray Formation is a hard, abrasive material, proven techniques developed on bucket-wheel excavators overcome this difficulty. Teeth can be fitted to the bucket for the hard digging. The arrangement, shape and material of these teeth are critical from the standpoint both of tooth life and excavator output. Rasper and Ritter (1961) have shown that average tooth life has been increased from 8 hours to 350 hours in one specific installation. However, it has been shown (Linden, 1957) that the cross-sectional area of the excavated slice is more critical in cutting than is the shape of the teeth and thus it is important to determine the proper slice cross section to minimize the specific digging force. It is known that for large-slice sections the specific digging force may, in fact, be lower than for small ones, although this observation has not been completely evaluated over the total range of cross-sectional slice areas which may be found in practice (from 5 to 620 square inches). This indicates that high-capacity excavators will result in better performance than small ones with respect to power consumption per cubic yard of production.

The bucket-wheel excavator operates in a manner quite different from other large excavating equipment such as a power shovel or dragline. In the operation of power shovels or draglines, very large impact forces are more significant than continuous loads. It is difficult to compare these different types of equipment with bucket-wheel excavators since at present there are no models of each type in a similar size-range and production capacity. However, taking data from Sebold (1962) for the power shovel and from Weis (1962) for the dragline, a comparison of the swing machinery speeds indicates the difference that can be expected in cyclic impact loads:

	Wheel Excavator	**Power Shovel**	**Dragline**
Swing speed, ft./min.	130	2,200	similar to shovel
Swing drive, hp.	220	3,000	2,250

The lower impact loading of the bucket-wheel excavators means smaller dynamic stresses and results in longer life of parts which are subject to high stresses, such as wire ropes. The slower swinging and hoisting movement of the bucket-wheel excavator results in a gradual shifting of load on the crawler assemblies (Habermaas and Krumrey, 1963). This means that bearing stresses on the supporting ground are lower and has resulted in wheel excavators being employed at sites where power shovels could not be used.

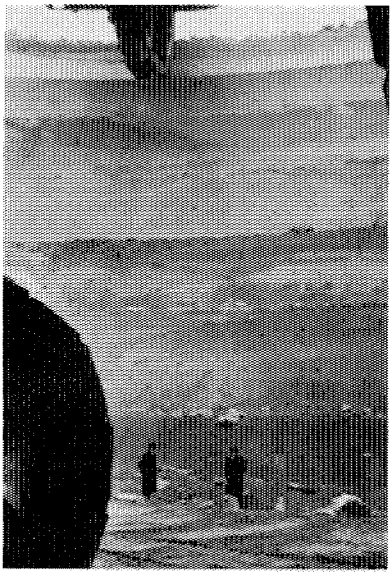
CONTINUOUS FEED

Perhaps the most important feature of a bucket-wheel excavator is that it digs material continuously, of a suitable size to be transferred directly on to belt conveyors, without hoppers. The size of the individual lumps of material is limited by the wheel buckets, although the large excavators are able to pick up lumps of considerable size. Cleavages and natural shearing planes in non-homogeneous material result in large lumps as digging proceeds. The lump size can be decreased by using high rotating speed with a slow swing speed, and if conditions warrant it, the insertion of ripper blades between the buckets. As a precautionary measure, wheel conveyors are equipped with very sturdy idlers in the transfer zones, and before the material is taken from the wheel and transferred to the pit conveyor large lumps are eliminated by a grizzly.

WINTER CONDITIONS

The Athabasca oil sands are in an area subject to long, cold winters, which present difficulties to be overcome in open-pit mining. Of particular concern is the penetration of frost into ground which must be excavated. The lower the temperature and the longer the freezing period, the deeper the frost will penetrate, although the rate of frost penetration decreases with depth. For example, the rate of penetration of frost for ground which is not covered by snow or vegetation, at an average ambient temperature of —5°F, may be as high as 2 to 2.5 inches in 24 hours (Beljakov, 1960). After approximately one month this rate decreases to about half of its initial value and declines steadily in subsequent weeks. The new and old high walls, the digging face, the top of each block, and the quarry floor are all susceptible to frost penetration. The freezing of the quarry floor will have no particular influence on the digging process, but the other three surfaces must ultimately be dug with the excavating equipment. Since the digging face has a fresh cut approximately every 12 to 24 hours, the frost penetration should only be of the order of 1 to 2½ inches. However, the high walls and top of the blocks may stand for periods of over a month, resulting in penetration of frost to depths up to 7 feet.

The thin frost layer on the digging face does not constitute any digging difficulty for the large bucket-wheel excavators, since the buckets bite up to 54 inches into the face. However, difficulties do arise in that the surface material at the top comes out as large, frozen chunks which, if caught in the buckets, represent a hazard for the



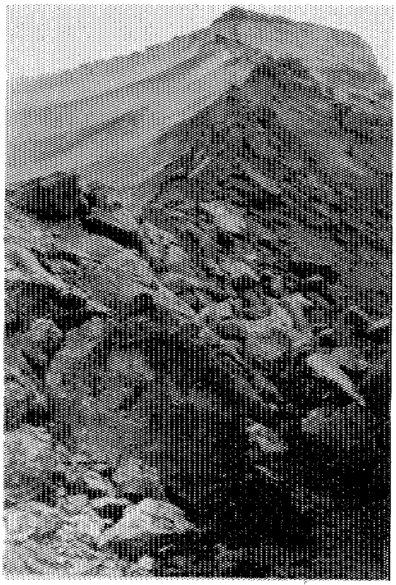


FIGURE 5. (*Left*) *Bucket-wheel excavator digging a 150-foot face of sand overburden. Boulders are frozen sand.*

FIGURE 6. (*Right*) *Digging face and old high wall showing large chunks of frozen sand.*

FIGURE 7. *Further extension of old high wall shown in figure 6 showing movable conveyor for feed from wheel excavator.*

conveyor belts and, if not, roll down the face resulting in debris on the quarry floor. Several techniques can be employed to minimize this effect of large chunks of frozen material. Since the excavator is standing at the foot of the digging face, there is complete access to the top of the block to carry out blasting with closely spaced drill-holes. Heat has been used at the top of the block to thaw the top surface. Ripping of the surface impedes the frost penetration rate and is now commonly employed, resulting in about half the amount of frozen material.

The excavator must advance into the frozen material at the high wall. A great deal of this frozen material will roll down the slope as large chunks and come to rest at the toe of the wall (Figs. 6 and 7). The proximity of the conveyor shown to the right of the picture indicates that precautions must be taken in blasting. Material that is still too large for wheel pick-up after it has rolled down the slope must be sized so that the wheel can pick it up while levelling and clearing the quarry floor.

The mining plan can be altered during the winter to reduce the frost problem. Decreased block widths and shortened face lengths will result in more rapid excavation of the face and some degree of frost penetration control (Beljakov, 1960). A shorter bench length would mean less frost penertation in the high wall: for example, if the total bench could be excavated in ten days it is anticipated that frost penetration on the high wall would be approximately 22 inches and that the frozen chunks would be smaller. However, decreasing of the block width and face lengths is limited since the excavator capacity will drop off, thus offsetting the advantage of a slower rate of frost penetration. Experience has shown that the high wall should be at the southern side of the pit during the winter if possible, to eliminate cyclic thawing and freezing which could result in slides. Although a northern exposure would retard freezing in winter and speed up thawing in spring, this is not particularly advantageous.

The operating conditions in the pit during winter would be vastly improved if a good drainage scheme were installed.

SUMMARY

The preceding remarks demonstrate that the bucket-wheel excavators have operational advantages when mining oil sand, since these wheels have been operated under conditions analogous in some respects to those expected in the Athabasca oil sands. The experience and detailed knowledge now available can be used to design new features into the large machines so that they can be modified to operate successfully in the Athabasca oil sands.

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SEPARATION OF OIL FROM THE ATHABASCA OIL SANDS BY SAND REDUCTION

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ABSTRACT

A process of sand reduction reduces the solids content of the Athabasca oil sand to the extent where direct distillation and coking in a fluidized-solids bed may be economically practical. In this process, oil sand is mixed with water at low temperatures, clean sand separates, and the oil particles released from the structure agglomerate to form an oil phase. The agglomerate is then separated from the solids. The enriched-oil phase is processed directly, and water is recycled in the process.

Most of the process variables of sand reduction have been evaluated in batch and continuous bench scale studies. The results indicate that the process has the following attractive features: (1) low operating temperature, (2) good oil recoveries, (3) tolerance to fines in feed and recycle water, (4) no solvent required, (5) no sludge produced. Agglomeration of poor-quality oil sands, methods of separation and, distillation and coking require additional large-scale study before sand reduction could be considered a fully developed process.

INTRODUCTION

Studies by Peterson and Gishler (1951) have shown that Athabasca oil sands can be readily distilled and coked directly in a fluidized-sand bed. However, the large volumes of sand to be handled in this process result in high capital investments and excessive heat requirements. In the present study an attempt has been made to devise a simple process for removing enough sand to make direct distillation and coking economically attractive. This contrasts with the usual separation approach where the objectives are to recover the maximum amount of oil with minimum contamination of the product by solids and water.

Experiments in these laboratories have shown that mixing and shearing oil sands with water at ambient temperatures will produce oil agglomerates, leaving the bulk of the solids in a clean water slurry. After removal of the clean sand it is visualized that the oil agglomerate might be distilled and coked directly in a fluidized bed (Fig. 1). This paper describes the process variables affecting the formation and separation of oil agglomerates in what has been termed the Sand Reduction Process.

PROCESS VARIABLES

A series of small-scale laboratory experiments were carried out to investigate process variables. A typical experiment involved mixing oil sands with water in a mortar and pestle, followed by screening through a 20-mesh (U.S.) screen submerged in water to remove

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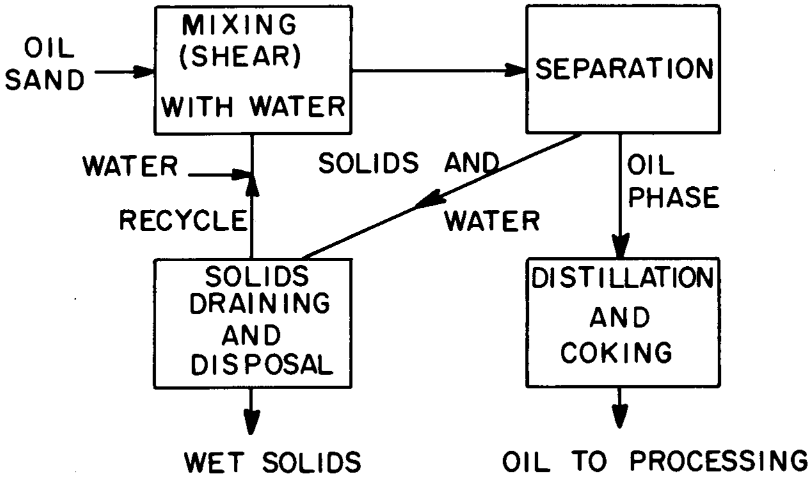

FIGURE 1. *Flow diagram of a sand reduction process.*

the oil agglomerate. The water and the solids which passed through the 20 mesh screen were then screened through a 325-mesh (U.S.) screen to separate water and fines from the sand. The three phases were then analyzed for oil, water, and solids. The sand reduction or solids removed expressed as weight per cent of the total solids originally present in the oil sands, and oil recovery in the agglomerate were computed.

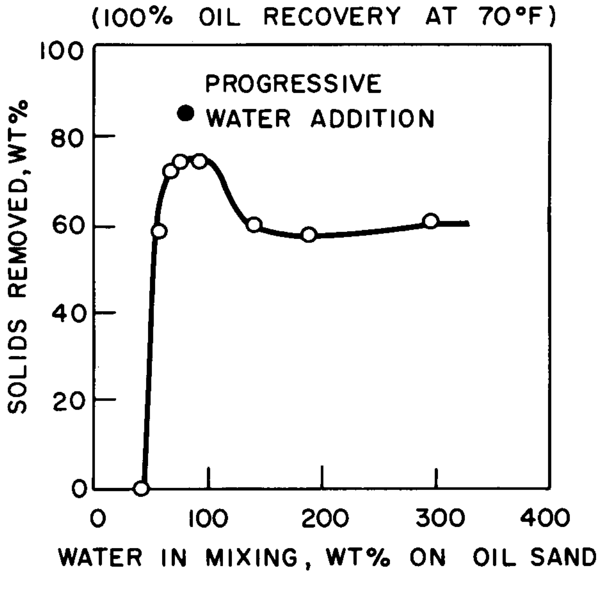

FIGURE 2. *Effect of water used in mixing on solids removed.*

EFFECT OF WATER ON MIXING

The amounts of sand removed by mixing an oil sand with water at room temperature have been plotted against the total water used in figure 2. When less than 30 weight per cent of water was added to the oil sand in the mixing operation, no separation was achieved even after extended mixing times. Water in amounts less than 30 per cent appeared to be taken up in the structure, presumably swelling the films of water around the sand; the result was a porridge-like mass. Additions of water between 40 and 60 weight per cent resulted in a substantial release of clean sand. It appears that more than 30 weight per cent water is enough for the existence of both dispersed oil and sand phases, while permitting agglomeration of oil particles. However, less than 30 per cent water is required at higher temperatures than 70°F for sand to start to separate. Increasing the total water in mixing to the range 60 to 110 weight per cent resulted in a maximum sand removal of 74 weight per cent; virtually no oil was lost in the sand product. In this range, sand reduction and oil agglomeration were maximums. Increasing the total water from 150 to 300 weight per cent appeared to have a beneficial washing effect on the oil phase, and good agglomeration was achieved at about constant sand-reduction. Separations with higher water additions than 300 weight per cent water in mixing, which are not shown in figure 2, gave lower oil recoveries. For example, at 400 weight per cent the oil recovery was 92 weight per cent. The loss of oil to the solids phase resulted from some small widely dispersed oil particles not adhering to the larger agglomerates. In general, these data show the existence of an optimum amount of water in mixing to effect good sand-reduction and oil agglomeration. In one experiment shown by the closed point in figure 2, the water was added in small increments during the mixing operation; this resulted in a markedly higher level of sand removal.

EFFECT OF WATER CONTENT OF OIL SANDS

The water content of the oil sands varies from zero to about 20 weight per cent. Most recorded values are from 1 to 7 weight per cent. In any commercial process, mined oil sands of different connate water and oil contents would have to be processed.

A set of experiments similar to those discussed above was carried out on oil sands with different water contents. Two main types of mechanism of sand reduction appeared on mixing oil sands with water. The optimum amount of water appeared to be about the same in each case. One mechanism occurs with oil sands having a connate water content greater than 5 weight per cent. On mixing this type of oil sand with 30 to 300 weight per cent water, the structure breaks down in about 5 to 25 seconds under very gentle mixing. The resulting mixture is a dispersion in water of sand and very small oil particles, presumably from the voids between the wet sand particles in the original structure. On further mixing, these oil particles coalesce, together with some sand, to form an oil phase. The higher the amount of connate water in the oil sand, the longer is the mixing time required to complete agglomeration for total oil-recovery. The mixing operation for these types of oil sand should be specifically for agglomeration.

Oil sands containing less than 5 weight per cent connate water, when mixed with water for less than one minute form small lumps and not discrete particles of sand and oil. Little or no free sand separates at this time. Further mixing results in release of sand from these aggregates. The net result was similar to that of the first mechanism, after the same mixing time, i.e., clean wet sand and an agglomerate were obtained. However, the sand removal is less by this mechanism. A better mixing operation for these types of oil sands is beating or kneading with the critical amount of water.

When the oil sands contained about 5 weight per cent water, then an intermediate mechanism was observed. The aggregates from which sand was lost also behaved as sites on which the very small oil particles adhered readily; this resulted in very good agglomeration.

Curves of similar shape to that in figure 2 were obtained with these oil sands of different water contents. The optimum sand-reductions were in the same range of total water and mixing time. These optimum results are plotted in figure 3 with open circles. As

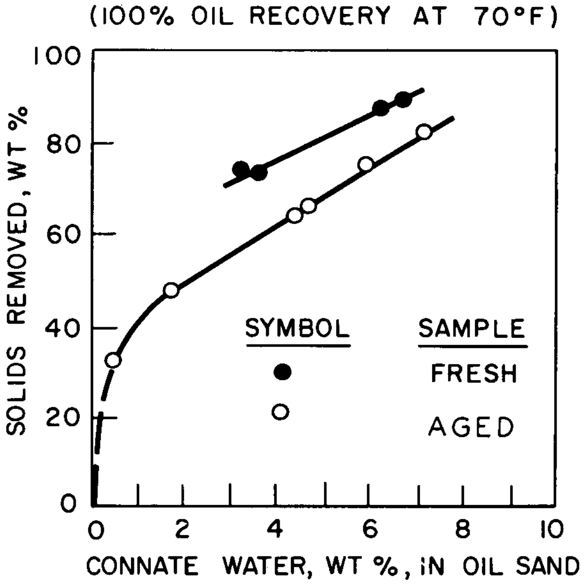

FIGURE 3. Effect of connate water content of oil sand on sand reduction.

these initial oil-sand samples had been in storage for several months, separations were carried out with fresh samples. The results are plotted by solid circles in figure 3, and it can be seen that a significant improvement in sand reduction was obtained. This suggests that oil sands age or deteriorate with time, on exposure to air.

EFFECT OF TEMPERATURE ON SAND REDUCTION

The results of studies on the effect of temperature are plotted in figure 4. With oil sands containing 6.0 per cent connate water above 32°F, the breakdown of the structure occurred readily on mixing with added water. Agglomeration was very poor up to 40°F and some oil was lost to the sand. At higher temperatures up to about 60°F agglomeration occurred, but oil recovery was not complete: 98 per cent. Complete oil-recoveries (good agglomeration) and good sand-reductions were obtained at temperatures of 60 to 90°F. At temperatures above about 100°F, reduced viscosity of the oil phase resulted in more fluid agglomerates of poorer stability.

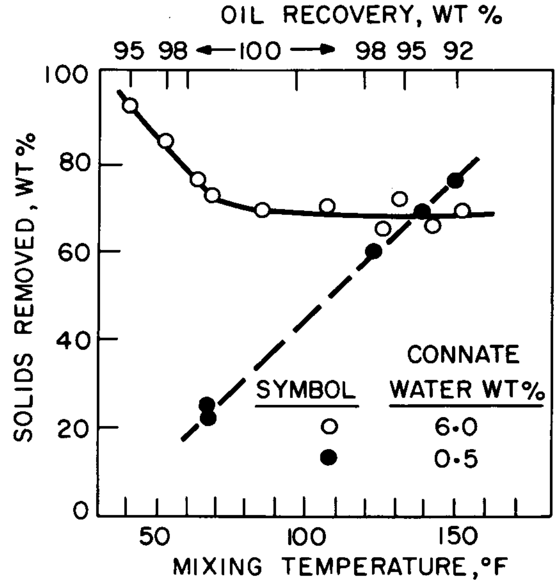

FIGURE 4. Effect of mixing temperature on sand reduction.

Sand reduction from oil sands containing 0.5 per cent connate water increased about linearly with temperature (Fig. 4). The decrease in viscosity of the oil with increasing temperatures caused more sand to be released from the structure at higher temperatures. Equivalent results were obtained with both types of oil sands at about 140°F.

The agglomerate became tacky in the temperature range 90 to 150°F, and separations were difficult as the oil tended to float on the water and to stick to the sides of the mortar above the water line.

Cooling of the mixture by quenching with cold water before screening under water was necessary. Further, if the temperature of screening was less than 70°F, very little or no fouling of the flat submerged screen occurred.

This work shows that mixing oil sands with a critical amount of water at ambient temperatures in an one-stage operation can result in removal of large amounts of clean sand, particularly with oil sands high in water. This approach gives relatively low sand-reduction with oil sands low in water. However, increasing the temperature of mixing with oil sands low in connate water has been found to increase sand removal.

The comparison shown in table 1 illustrates how the effect of temperature provides flexibility in the process to handle oil sands low in water content. It can be seen from the table that by the continuous lowering of temperature a small improvement was obtained. The best sand reduction of 80 weight per cent was obtained in experiments with a two-stage mixing operation, i.e. at a high temperature and then at a low temperature. Similar results, not shown in table 1, were obtained when only 30 to 40 weight per cent was used in the first warm mixing stage, and the balance added in the second colder stage, i.e. progressive addition of water in a two staged temperature mixing operation.

TABLE 1. EFFECT OF PROGRESSIVE LOWERING OF TEMPERATURE ON AGGLOMERATION AND SOLIDS REMOVAL
(Oil-sand feed contained 2 weight per cent connate water. Total water in mixing was 100 weight per cent)

Number of mixing stages	1	1	2
Temperature operation	Constant	Decreasing continuously*	Decreasing in stages†
Temperature (°F)	70	140 to 80	140 and 70
Mixing time (minutes)	5	5	2½ and 2½
Solids removed (wt. %)	45	54	80

*At a rate of 12°F per minute by external cooling
†Rapid external cooling between stages

EFFECT OF COKE

After the initial studies of separation using a shear force with water at ambient temperatures, it was thought that the addition of coke, having a hydrophobic surface, might improve agglomeration and displace more sand from the agglomerate. Some experiments were carried out in which the oil sands were shaken and mixed with water and different types of coke in a glass bottle, although shaking is probably not the best method of mixing. Good sand-reduction and agglomeration were achieved. The longer the shaking the larger the agglomerates became; the final shape of the agglomerates was roughly spherical. The added carbon goes into the oil phase or agglomerate, and apparently behaves as a carrier for the oil by increasing the viscosity of the agglomerate and acting as a scavenger for small oil particles, hence aiding agglomeration.

The advantage of adding coke, which is a possible product ot subsequent oil processing, at low temperatures to an oil sand high in water was very small (Table 2). The coke had about the same particle size as the sand.

TABLE 2. EFFECT OF COKE ON SAND REDUCTION
(100 weight per cent water in mixing, 100 weight per cent oil recovery)

Connate water in feed (wt. %)	7	7	2	2	2	2
Coke* added (wt. %)	0	25	0	25	0	25
Temperature (°F)	70	70	70	70	125	125
Oil-phase yield, less coke (wt. %)	39	33	63	51	49	33
Solids removed (wt. %)	76	78	45	60	62	83

*All particle sizes were in the range of 20- to 325-mesh.

Further experiments on adding coke to an oil sand, low in water content, at different temperatures were carried out. The results are compared with those from experiments without coke in table 2. The best results with coke addition were obtained at 125°F.

Use of coke to increase the viscosity of the oil phase was useful at the higher temperatures. Mixing was clean and agglomeration appeared similar to that of the operations at lower temperatures without coke.

The addition of carbon has also been tried by Weingaertner (Weingaertner *et al.*, 1956; Weingaertner, 1957 and 1960a) who proposed a phase-exchange mechanism. More recent work by Weingaertner (1960b) showed that the addition of carbon was not needed with the oil sands that he used.

EFFECT OF FINES

Studies with fines in the feed have indicated that amounts of clay and silt up to 25 weight per cent of the oil sands may be tolerated in the feed without significant effect on oil recovery in the sand-reduction process (Fig. 5). However, with amounts greater than 25 weight per cent poor agglomeration was obtained. The oil became dispersed, and at these higher clay and silt contents and the oil spheres tended to float. The effect of fines in causing poor agglomeration was physical. The clay and silt formed a skin around the oil phase, hence, the more fines the greater the surface area of oil-clay

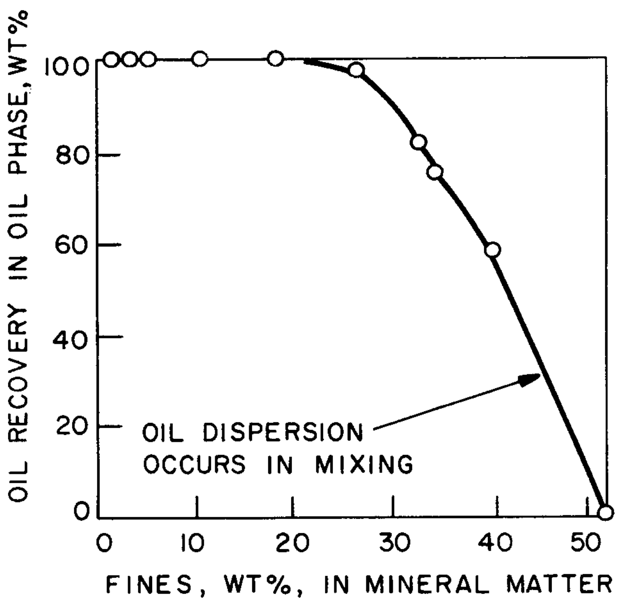

FIGURE 5. Effect of fines on oil recovery.

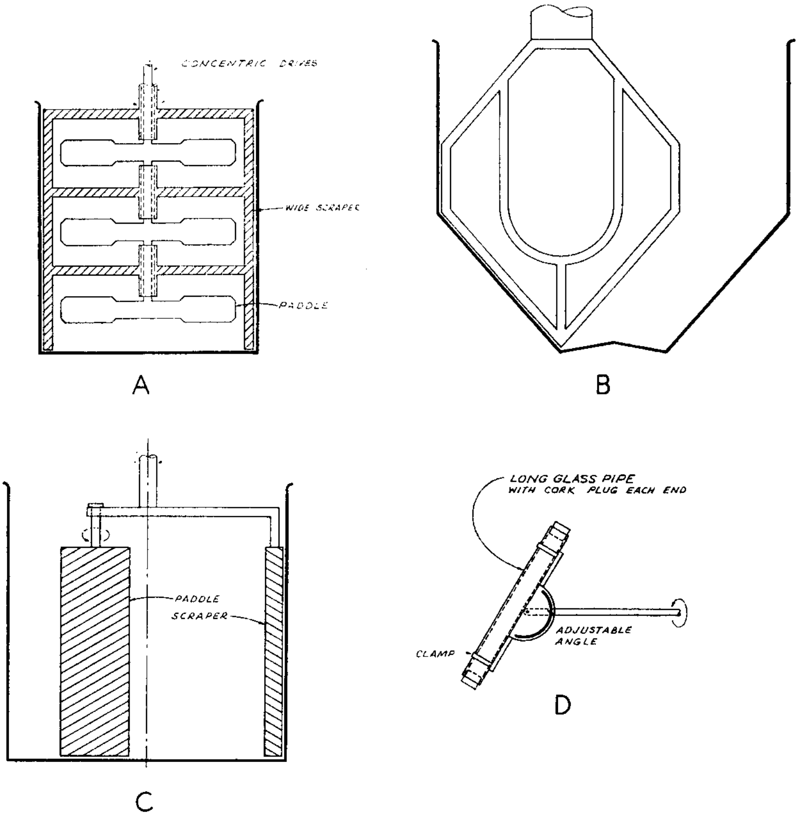

FIGURE 6. Mixing equipment producing poor oil agglomeration. A. Grease mixer, B. Hobart beater, C. Hobart mixer, D. Tumbler.

and the smaller the oil-particle size. Continued mixing also gave smaller oil particles.

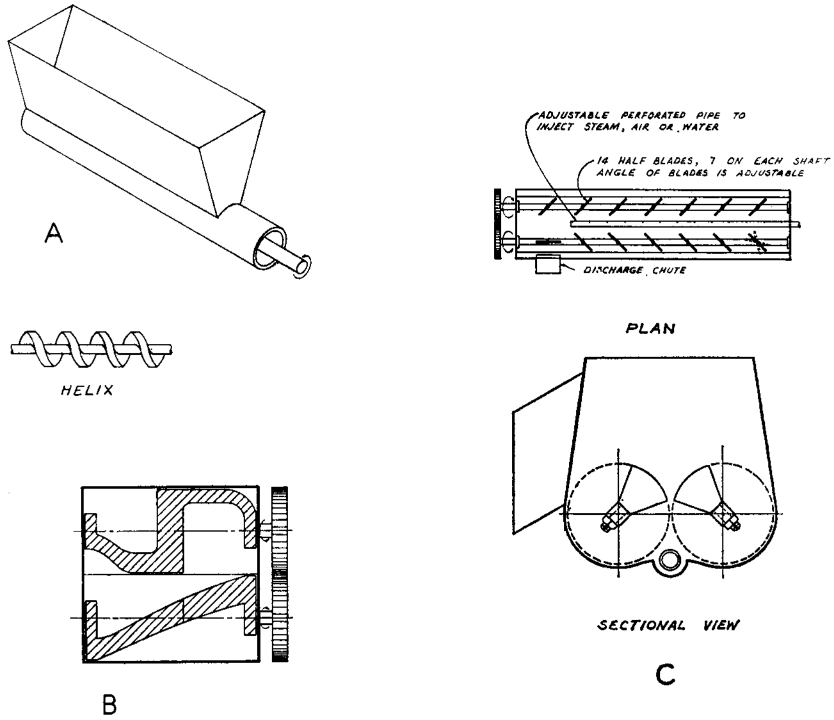

FIGURE 7. Mixing equipment producing good oil agglomeration. A. Trough mixer, B. Kneader, C. Pug mill.

TABLE 3. EFFECTIVENESS OF MECHANICAL BATCH MIXERS

(Oil sand contained 4.5 per cent water. Conditions were 3 to 8 minutes mixing, 65 to 140 weight per cent total water, 70°F temperature. The product was screened from sand with a 20-mesh sieve in a Ro-tap sieve shaker.)

A. Mixers that gave poor agglomeration	Results (wt. %)	
Grease mixer	Solids removed	61 to 68
Hobart mixer	Oil in oil phase	26 to 39
Tumbler	Water in oil phase	12 to 13
	Oil recovered in oil phase	75 to 92

B. Mixers that gave good agglomeration	Results (wt. %)	
Pestle and mortar		
Trough mixer		
(a) Square helix	Solids removed	59 to 69
(b) Ribbon	Oil in oil phase	30 to 36
Kneader	Water in oil phase	14 to 20
Pug mill	Oil recovered in oil phase	94 to 100

EFFECT OF BATCH-MIXING ACTION

A series of experiments was carried out to evaluate the effect of different types of mixers on sand reduction. The technique used was similar to that of the earlier laboratory studies. A batch mixer was substituted for the mortar and pestle, and a Ro-Tap sieve shaker (Fisher) with 20-mesh and 325-mesh screens submerged in water was used for separation. The agglomerates recovered were egg-shape in form and of different sizes.

The results summarized in table 3 are, in general, similar except for oil recovery. On this basis of comparison, the mixers formed two groups that gave poor agglomeration and good agglomeration. The designs of some of these mixers are shown in figues 6 and 7. A square helix essentially duplicated the action of the mortar and pestle. However, it was also found that a kneading action, such as in a dough kneader or pug mill, gave good results. The mixer parts tended to become covered with a layer of oil. This appeared both to aid agglomeration and to protect the equipment from the abrasive action of the separated clean sand.

WATER CONTENT OF OIL PHASE

The average water contents of the agglomerate from the laboratory experiments and in the batch-mixing operations are shown in table 4. Water contents were lower in the batch-mixing operations, which may have resulted from the larger scale of the experiments.

TABLE 4. AVERAGE WATER CONTENT OF OIL PHASE

Mixing operation	Oil sand charge grams	Number of experiments	Average weight per cent water in oil phase
Mortar and pestle	25 to 100	23	22.2
Batch mixers	400 to 600	45	13.3

SEPARATION METHODS

SUBMERGED SCREENING

The technique of screening sand from agglomerates under water has proved to be surprisingly effective, provided the temperature is less than 70°F and care is first taken to clean and water-wet the screen, then little or no fouling of the screen occurs. The screen was cleaned with (1) toluene, (2) acetone, (3) hot soapy water, and (4) cold water.

The screen size of 20-mesh (U.S.) was chosen to enable most of the sand to pass readily through and to minimize oil loss to the sand. In general, changing the mesh size from 14 to 40 did not change the results significantly. However, there is probably an optimum screen-size to maximize oil recovery and throughput. A disadvantage of screening is that water-wet solids greater than the mesh size are removed with the agglomerates.

FLOTATION

Some of the gas in the oil sand is hydrocarbon and is dissolved in the oil. Other gas components excluding air are carbon dioxide and nitrogen. Reducing the pressure above oil sand mixed with water liberates the gas as bubbles which adhere to the oil and float it as a froth. This was clearly observed in studies measuring the relative change in volume of small portions of an agglomerate in a graduated tube, immersed in a temperature bath, and connected to a vacuum system controlling the pressure at the desired level.

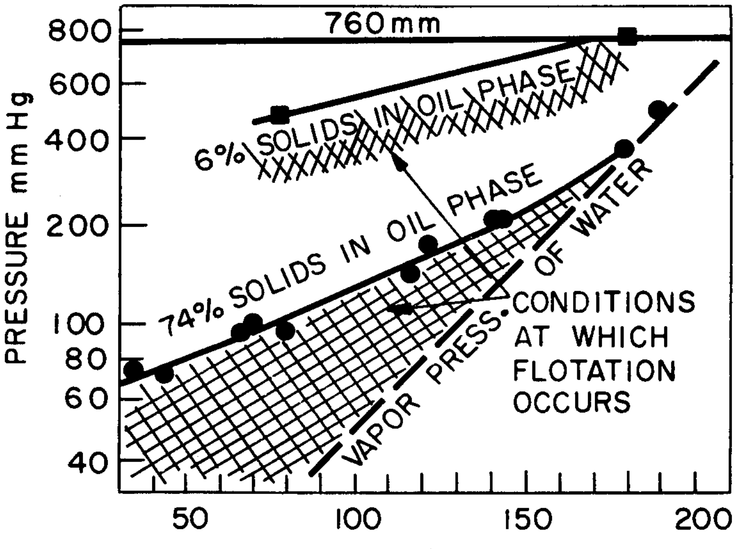

FIGURE 8. Temperature and pressure relations at which oil phase floated on water.

The lower limit of pressure at a given temperature is governed by the boiling point of water. The upper limit of pressure for the oil phase to float is governed approximately by the builk density, i.e., the density of the mixture of oil, gas and solids. The experimental relation of reduced pressure and temperature at which two oil phases of widely different solids content floated in water is shown in figure 8. The shaded area between the curve and the line for the vapour pressure of water represents conditions at which flotation could be carried out. The general patterns illustrated in figure 8 apply only to the two specific oil phases shown. The lower the solids content or the greater the gas content, then the higher the pressure will be for flotation. The dissolved hydrocarbon gases in the oil play a useful

TABLE 5. RESULTS OF OIL-SAND SEPARATION BY REDUCED PRESSURE METHOD (75°F and 41 mm Hg—No Aeration)

	Distilled Water	Freshly boiled distilled water
Solids reduction (wt. %)	89	91
Oil recovery (wt. %)	83	61

role in decreasing the apparent density of the oil froth. However, not all the oil in the oil sand is associated with adequate gas for flotation.

It has been shown that a critical temperature-pressure relationship exists which determines whether an oil particle will float or sink in water. The solids content of the oil particle is an important parameter. A low-temperature, high-vacuum process (75°F, 40mm Hg) is technically feasible, but it is likely that some air would be required to assist flotation. The lower oil recovery when freshly boiled distilled water was used (Table 5) is believed to result from the lesser amount of dissolved gas coming out of solution and attaching to the oil when the pressure is reduced.

An extension of these methods would be to mix oil sand with hydrocarbon gases under pressure to increase the gas content of the oil phase prior to mixing with water to effect agglomeration of oil and separation of clean sand. On release of the pressure, the oil floats under atmospheric conditions. Preliminary experiments using this type of operation were tried in a pressure-tight kneader. The results showed this method was possible and should give good oil-recoveries. However, further study of variables is required to define the potential of this method of separation.

OTHER METHODS

In some mixing operations all the oil adhered to an oil-wet pestle, and complete separation could be made by removing the pestle from the mortar. This suggested the possibility of utilizing the property of the oil to stick to a hydrophobic surface to effect separation.

Also, when oil sand is mixed with a gross amount of water and discrete particles separate, they classify depending on their density, the oil particles forming a layer on the heavier sand. This indicated that the use of a water-fluidized solids bed may effect separation by flotation of the oil particles.

SUMMARY

Two general approaches to the problem of separation were considered in the laboratory studies:

Mechanical

(1) Submerged screening
(2) Oil wetting
(3) Other methods

Flotation

(1) Reduced pressure
(2) Hydrocarbon-gas flotation
(3) Denser media

All of these methods were investigated on a larger scale in small pilot-plant semicontinuous-equipment. These studies are discussed in the next section.

PILOT-PLANT DEMONSTRATIONS

HELIX AND SUBMERGED ROTARY SCREEN

A unit was built to demonstrate the sand-reduction process on a continuous basis. A flow diagram is shown in figure 9. In this equipment, oil sands and water were fed to a hopper and in turn, to a helix mixing element, which had been found to simulate the mortar and pestle operation. Provision was made to vary the temperature of mixing. The mixture passed into a submerged rotating screen, which separated the sand from the oil agglomerates. The product streams were withdrawn through valves. The oil phase was heated to facilitate flow for withdrawal.

An oil scum collected on the surface of the water above the screen. The amount of oil varied with the oil-sand feed. However, provision was made to recover this oil by overflow, settling, and recycle of water. The size and shape of the screen could be changed. Two mixing elements were used: a solid helix shown in figure 9, and a modification in which a stainless steel ribbon was attached to a center rod by studs.

It can be seen from the first comparison in table 6 that the quality of oil sand feed is an important variable. The sand reductions obtained in this unit with fresh and aged oil sand agreed with those obtained in the laboratory studies. The second comparison shows the solid helix to be superior to the ribbon.

TABLE 6. SAND REDUCTION WITH CONTINUOUS SCREW AND ROTARY SCREEN (70°F, 30 lb/hr oil sand and 25 lb/hr of water)

Mixing Element	Oil Sand (% Water)	Oil Sand (Type)	Solids removed (wt. %)	Oil in oil phase (wt. %)
Effect of feed quality				
Solid helix	5	Aged	62(63)	32
	5	Fresh	83(81)	45
	6	Fresh	88(88)	60
Effect of mixing element				
Solid helix	6	Fresh	88	60
Ribbon	6	Fresh	81	48

Mortar and pestle results shown in parentheses

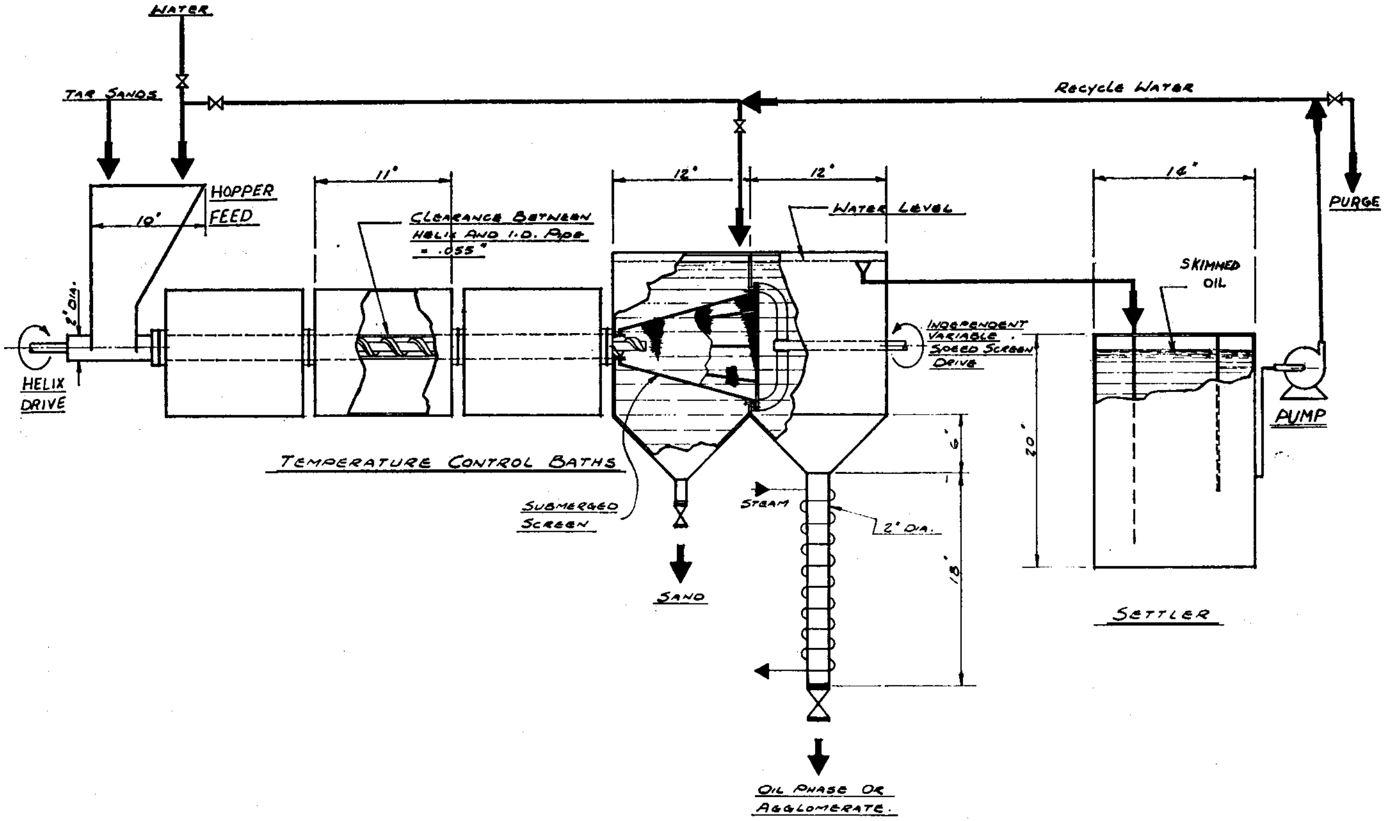

FIGURE 9. Continuous screw and rotary screen equipment used for process demonstration.

Successful demonstration runs were made combining a pug mill with a submerged rotary screen. Although the pug mill is not the best type of mixer for agglomeration, it does represent a more realistic approximation to a commercial type of operation.

SCREEN SEPARATIONS

Three types of screens have been tried: flat, rotary, and vibrating. Flat and vibrating screens have not been successful. The oil phase piles up on the surface throughout screen angles from 20° to 70° and obstructs the sieve. If the screen is first water wet, the accumulation of agglomerate causing the fouling could be readily lifted off leaving a clean water-wet screen. Rotary screens from 14 to 40-mesh with slopes of 1¼ to 2½ inches per foot (length) have given very good performance provided they are initially water wet and maintained submerged in water. No problems with plugging occurred even after continuous operations for many runs at different conditions.

FLUIDIZED-SOLIDS FLOTATION

The idea was to use a sand bed fluidized by water to create a denser medium than oil to effect separation by flotation. This approach is shown diagrammatically in figure 10. The system worked well, however the main difficulty was that a small number of oil particles moved up and down in water channels resulting from uneven fluidization of the sand. On a larger scale this may represent a significant oil loss in the solids. Considerable further investigation and development would be required to evaluate this approach fully.

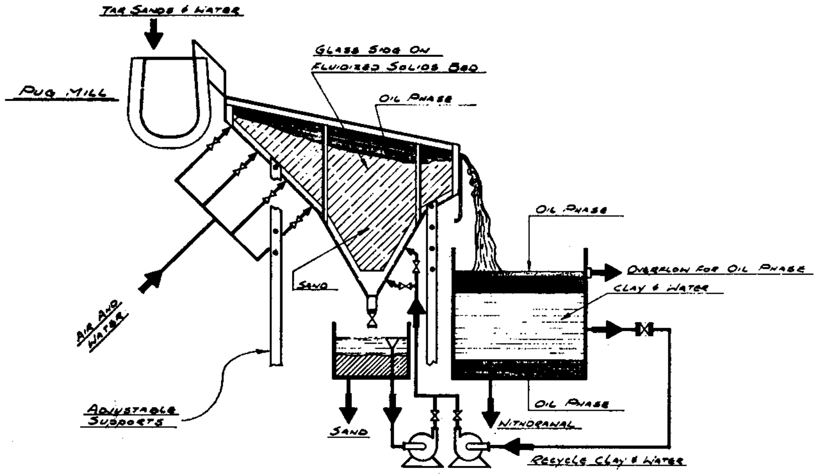

FIGURE 10. Equipment used for fluidized-solids flotation.

Other possible methods of separation along similar lines are riffling and jigging which were not explored.

REDUCED-PRESSURE FLOTATION

Two methods of separation by reduced-pressure flotation were devised on a larger scale (Fig. 11). In method A, a continuous demonstration with the pug mill used in the batch operations and a glass separator was attempted. Some problems were encountered in the separation. These were the disengagement of the oil from the sand in feeding and the pressure due to the height of material between the feed point and the overflow. An improved approach would introduce the feed at the oil-water interface, just above, or just below, with a minimum distance between the interface and the oil-product overflow. A feed-distributing system also appears to be advantageous. A commercial operation would also use barometric legs to withdraw the product streams.

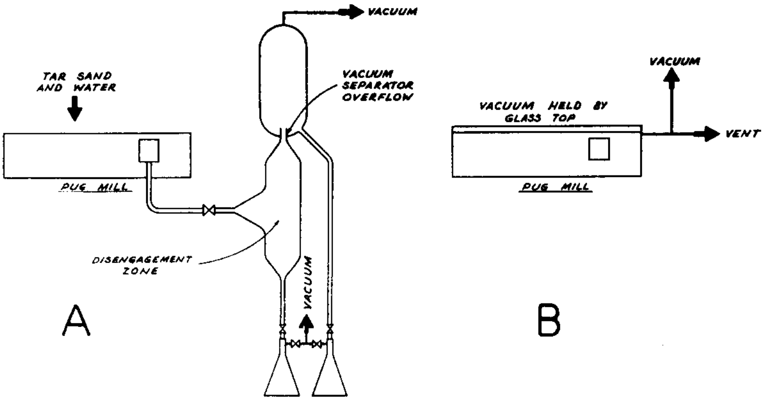

FIGURE 11. *Equipment used for reduced pressure separations in continuous and batch operations.*
A. Continuous, B. Batch.

In method B, figure 11, the mixing action of the paddles in a pug mill helped raise the oil phase to the surface of the mixture and disengage it as a froth. In the experimental technique, most of the oil product was collected and sampled by rapidly breaking the vacuum and skimming the froth from the surface of the mixer. Skimming and pumping out of the froth or withdrawing the product streams through barometric legs would presumably be used on a large scale.

The conclusions that may be drawn from the results of these bench operations are: (1) better oil-recoveries are obtained with longer mixing time to effect agglomeration; and (2) reduced-pressure flotation and submerged screening give essentially equivalent results.

OIL WETTING

The agglomerate-sand mixture from the pug mill was fed to the belt separator shown in figure 12. The oil phases adhered to

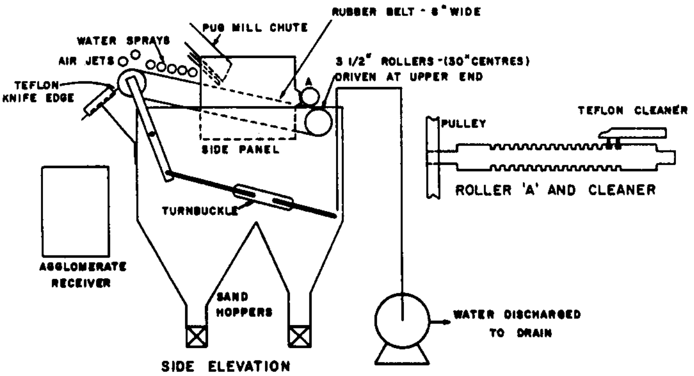

FIGURE 12. Belt separator equipment used for separations by oil wetting.

the belt, which had been precoated with oil, and were removed by a knife blade at the upper edge. The sand-water slurry was washed down the belt and fell into a tank beneath. Considerable mechanical and hydraulic work is required to effect complete separation of oil and solid phases by this method.

COMPARISON OF METHODS OF SEPARATION

Preliminary comparisons of the methods described above are shown in table 7. Essentially equivalent results were obtained with each method. However, further investigation and development of these methods are required before a realistic quantitative comparison could be made.

TABLE 7. COMPARISONS OF SEMI-CONTINUOUS METHODS OF SEPARATION
(Separations were preceded by mixing in a pug mill at 70°F)

Method of Separation	Oil in oil phase (wt. %)	Oil recovery in oil phase (wt. %)	Wt. % sand removed
Oil sand with 6% water			
Rotary screen	60	100	88
Fluidized-solid bed	55	99	85
Reduced pressure (A)	60	100	88
Oil sand with 5% water			
Rotary screen	45*	99	83
Belt conveyor with water washing	45†	99+	83

*Oil phase contained 15 wt. % >20-mesh solids
†Oil phase contained 2 wt. % >20-mesh solids

A general appraisal of these methods can be based on the average size of the agglomerates produced in mixing (Fig. 13). Of the methods tried, submerged rotary screening appears to be the most

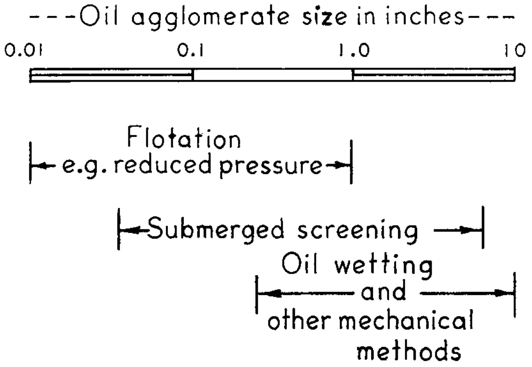

FIGURE 13. *Diagram showing the application of separation techniques to various particle sizes of oil agglomerates.*

successful and most positive method of separation with different types of oil sands. However, only a part of the total screen is being used, and although immersion in water significantly increases the rate of screening, capacities must be based on effective area in a given design.

One of the more important considerations is the size and area of separation equipment that would be required in a commercial operation. Only preliminary relative numbers for scale up can be obtained from these studies. Larger-scale operations would be required to determine these factors.

RESULTS OF STUDIES OF PILOT-PLANT INVESTIGATIONS

Process variable studies with a pug mill-rotary screen assembly gave the following results:

(1) The addition of 25 weight per cent sand or fines in the feed had surprisingly little effect on sand reduction or oil recovery with a good-quality oil sand.

(2) Clay and silt (minus 325-mesh fines) in the recycle water, although starting at 25 weight per cent quickly decreased to an equilibrium value of 13 weight per cent. Solids removal was approximately 80 per cent throughout the entire range of clay and silt levels studied. Oil recoveries were more than 96 per cent for all tests. This essentially substantiated the results of the small-scale laboratory studies.

(3) In some pug-mill operations with special paddle design, angle, lower weir height, and increased water circulation, improved agglomeration was obtained. The larger agglomerate-size appeared to result from reducing the ratio of sand to oil in mixer hold-up. Effectively, the clean sand that separated sank to the bottom of the mixing zone and was washed out quickly while the oil phase had a longer residence time. Hence, improved mixer design can effect better oil agglomeration.

(4) Some poor-quality oil sands gave 62 to 81 weight per cent solids removal, and lower oil-recoveries of 60 to 95 weight per cent. The oil particles from initial mixing did not readily adhere to each other. The oil phase separated by submerged screening was in the form of small agglomerates. Essentially complete oil recoveries were obtained for blends of one part by weight of poor-quality oil sand and three parts of good-quality oil sand. Processing of some poor-quality oil sands is a problem and requires further study.

DISTILLATION AND COKING OF OIL AGGLOMERATE

In sand reduction the oil sand is mixed with a specific amount of water to effect sand separation and oil agglomeration. The agglomerate is then separated and presumably could be fed directly to a fluidized-solids bed for distillation and coking (Fig. 1).

The heat requirement for distillation and coking of the agglomerate is compared in table 8 with those for the oil sand as feed, and froth from hot-water separation from pilot-plant operations reported by Clark (1950). The heat requirements are based on values reported by Peterson and Gishler (1951) from their studies on the fluidized-solids techique applied to the recovery of oil directly from oil sands. It can be seen from the comparison in table 8 that the agglomerate requires less coke to be burnt and would be in heat balance if the

TABLE 8. COMPARISON OF HEAT REQUIREMENTS FOR DIRECT DISTILLATION AND COKING OF OIL SAND AND SEPARATED OIL PHASES

	Oil Sand	Froth from hot water separation†	Agglomerate from sand reduction
A. Composition weight per cent lb/lb of oil			
Oil	14(1.00)	61(1.00)	58(1.00)
Solids	80(5.71)	4(0.07)	27(0.47)
Water	6(0.43)	35(0.58)	15(0.26)
B. BTUs* required to heat feed containing 1 lb. of oil			
Oil	717	717	717
Solids	1216	15	100
Water	667	892	403
Total	2600	1624	1220
water separation† of oil to produce heat requirement**	0.282	0.176	0.132

*1 lb oil=717 BTU, 1 lb sand=213 BTU, 1 lb water=1550 BTU. Peterson and Gishler (1951)

**At 9230 BTU/lb coke net heat release

†Average froth quality for seven different oil sands from pilot-plant separations reported by Clark (1950)

coke produced was about 13 weight per cent based on oil. Therefore, the agglomerate from sand reduction could be fed directly to a fluidized-solids bed for distillation and coking without excessive heat requirement.

The heat requirements for different water and solids contents in the feed are shown in figure 14. The data from table 8 have also been plotted. As the heat requirement for water is about seven times that for solids, there is considerable incentive to reduce the water content of the feed to a fluidized-solids bed for distillation and coking.

CONCLUSIONS

The results of studies in small-scale laboratory apparatus and pilot-plant equipment on sand reduction have indicated that this process has the following attractive features. (1) low operating temperature, (2) good oil-recoveries, (3) tolerance to clay and silt in feed and in recycle water, (4) relatively dry oil-phase, hence less

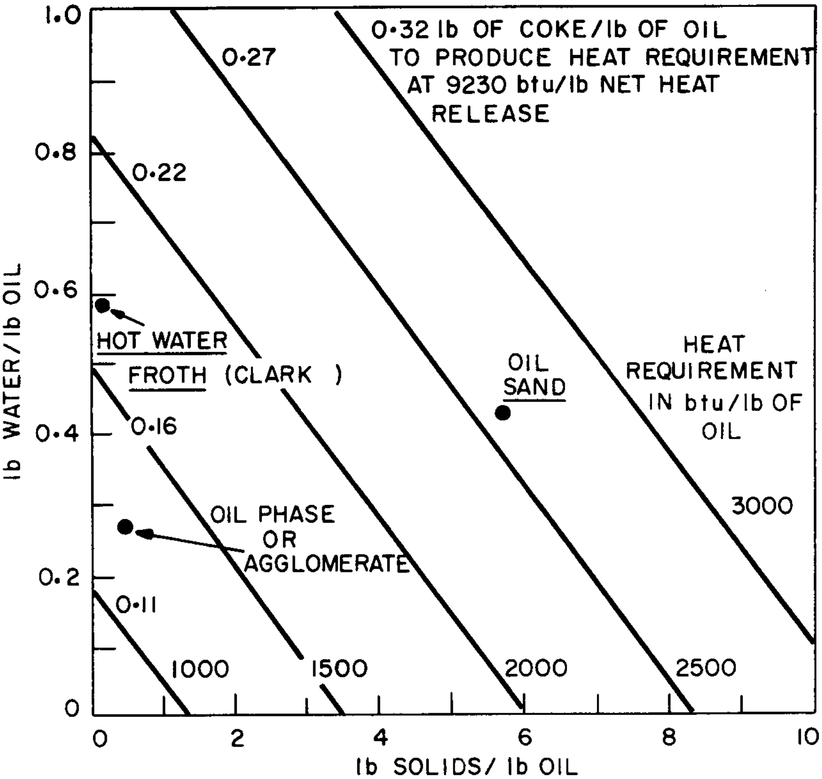

FIGURE 14. *Heat required in coking still for oily materials having different amounts of water and solids: Based on feed at 32°F, still products at 932°F, burner at 1,300°F and no heat recovery.*

heat requirement in direct distillation and coking, (5) no solvent required, (6) no sludge produced. Further large-scale experimental work is required before sand reduction can be considered a fully developed process. Agglomeration of poor-quality sands, methods of agglomerate separation, and distillation and coking all require additional study.

Application for patents covering all aspects of sand reduction have been filed.

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DEVELOPMENT OF AN ANHYDROUS PROCESS FOR OIL-SAND EXTRACTION

J. H. COTTRELL*

ABSTRACT

An anhydrous process for oil-sand extraction would as presently conceived result in a commercial plant in which a slurry of oil and sand plus hydrocarbon solvent would be placed on a moving belt passing over three drain chambers and a solvent-recovery chamber. The produced oil would go to a fractionation tower for solvent recovery and the raw bitumen to refining from whence make-up solvent would be realized.

INTRODUCTION

The search for a workable and economically sound process for extracting oil from oil sands and shales has long challenged man's ingenuity. Deposits of tar sands and shales occur at many localities throughout the world, but the Athabasca oil-sand deposits of northern Alberta represent an estimated reserve which exceeds the combined proven oil reserves of the rest of the world.

Many schemes for oil-sand development have been proposed over the past few decades. These proposals have included direct combustion, solvent extraction, water flotation and many variations thereof. Several schemes for extracting the bitumen *in-situ* have received consideration; however, this paper deals with extraction of oil sand recovered by open-pit mining only. The process for extraction of oil sand from open-pit mining can be separated into two general categories: (a) processes using water as separation media; (b) others, such as direct combustion, and solvent extraction.

The classic water processes are represented by the hot-water process of Clark and Pasternack (Pasternack *et al.*, 1951), the cold-water process of Djingheuzian (1951) and the more recent dense-phase process developed by the group composed of Cities Service Athabasca Inc., Imperial Oil Limited, Richfield Oil Corporation and Royalite Oil Company Limited. This process has rectified some of the recovery problems inherent in the previous water approaches. However, in order to provide an alternate non-water process, research work was carried out by the Cities Service Research and Development Company in 1959-61 which resulted in the anhydrous process of Cottrell and Leary (1962). This process is essentially a counter-current solvent extraction scheme.

Early experimental work on the anhydrous approach to extraction of bitumen from Athabasca oil sands led to the postulation of an hypothesis as to the physical makeup of the oil sands. The major

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*Cities Service Athabasca Inc., Edmonton portion of the solids contained in the normal oil sand is water-wet (Ball, 1935), and it is visualized that the individual grains of sand are surrounded by a thin film of water. This film of water is further encased by bitumen, with the bitumen also partially filling the voids between the individual sand grains (Fig. 1).

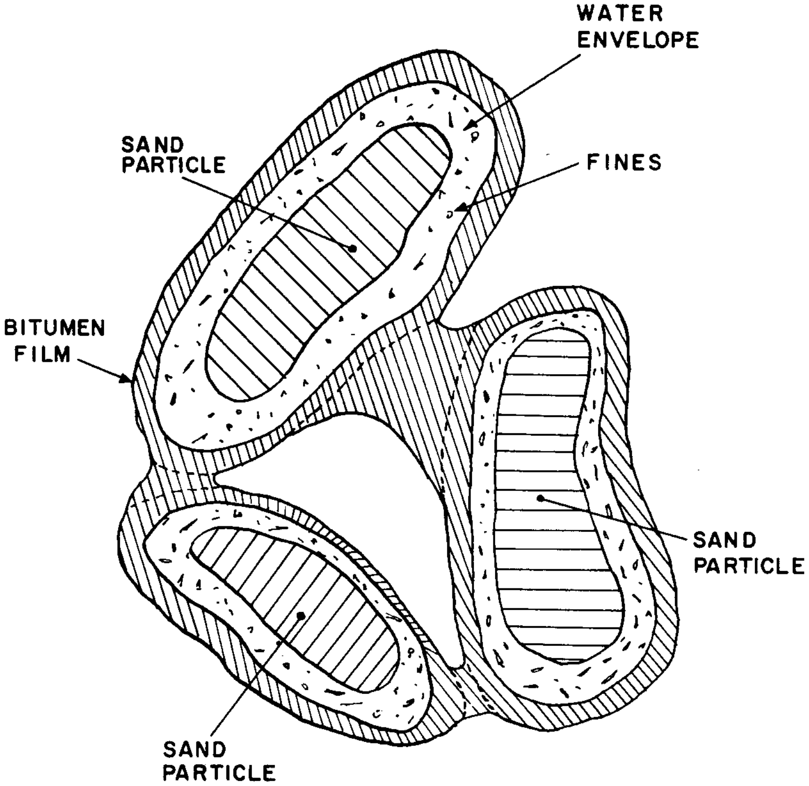

FIGURE 1. Typical arrangement of oil-sand particles.

A proportion of the mineral matter is in the form of clay and other materials of minus 44-micron size. The aforementioned hypothesis postulates that the majority of these materials are contained in the water envelope surrounding the sand grains or in the clay lenses throughout the oil-sand deposits which are generally water saturated. Alteration of the physical configuration tends to result in some oil-wetting of solids which in turn results in oil-rich sludges and solids-stabilized emulsions. This condition also leads to contamination of the oil phase with solids and water. It would then seem advantageous to develop a process that would remove the most oil from the sands before rupturing the connate-water envelope.

The interstices in the oil sands are partially filled with bitumen which agglomerates the *solid-water* particles into a cohesive mass. The problem of separation, therefore, is simply that of dispersing the *solid-water* particles and extracting with solvents the enveloping bitumen films in a water-immiscible-solvent phase. The resultant solvent-bitumen mixture can then be drained from the *solid-water* particles and the drained bed subjected to repeated solvent washes to displace the bitumen mixture.

The majority of the hydrocarbons filmed around the *solid-water* particles in the exit stream from the extraction step would then consist of a relatively low-boiling solvent. These *solid-water* particles would be processed for reclamation of the residual solvent before discarding.

The occurrence of fine mineral matter in the virgin sands has been mentioned and is widely discussed in the literature (Clark and Pasternack, 1949). When considering the factors that affect filtration or draining functions it is readily apparent that if the solids detected by screen analysis of the oil sands were uniformly dispersed throughout a solvent-oil-sand mixture, draining would be a slow, if not impossble, job. However, the observed drain-rates were high, ranging to 50 gpm/ft.^{2} through a 3-inch solids bed at a drain-liquor viscosity of 1.0 centistoke and pressure differentials of less than 15 inches of mercury. These high observed drain-rates can readily be explained by the *solid-water theory* stated above. The apparent diameters of the *solid-water* particles randomly laid down in the draining step were quite uniform and were larger than those of most dry solids existing within a given oil-sand sample. As long as the film strength of the hydrocarbon encasing the *solid-water* particle was maintained, one could expect a free flow of hydrocarbon through the solids bed.

Several methods were used to test the *solid-water* theory. The screen analyses of the mineral matter before solvent extraction and that of the drained mineral matter were not significantly different. In the same manner the ratio of water to solids was not affected. These results were obtained while removing in excess of 90 per cent of the bitumen in the form of a low bottom-sediment and water-drain liquid. Water was then dropped on the drained solids beds. This ruptured the films, and the beds blinded almost immediately. Drained solids were also slurried with water, and the resultant slurry was dropped on to the draining surface. Only about one half of the water drained before the bed plugged and would not drain under a pressure differential in excess of 20 inches of mercury. The water that did pass was clouded by fine suspended solids. It was readily apparent that the connate water and solids were physically associated in a manner which rendered them readily separable from a continuous hydrocarbon phase. In addition to these experimental tests, many microscopic observations were made.

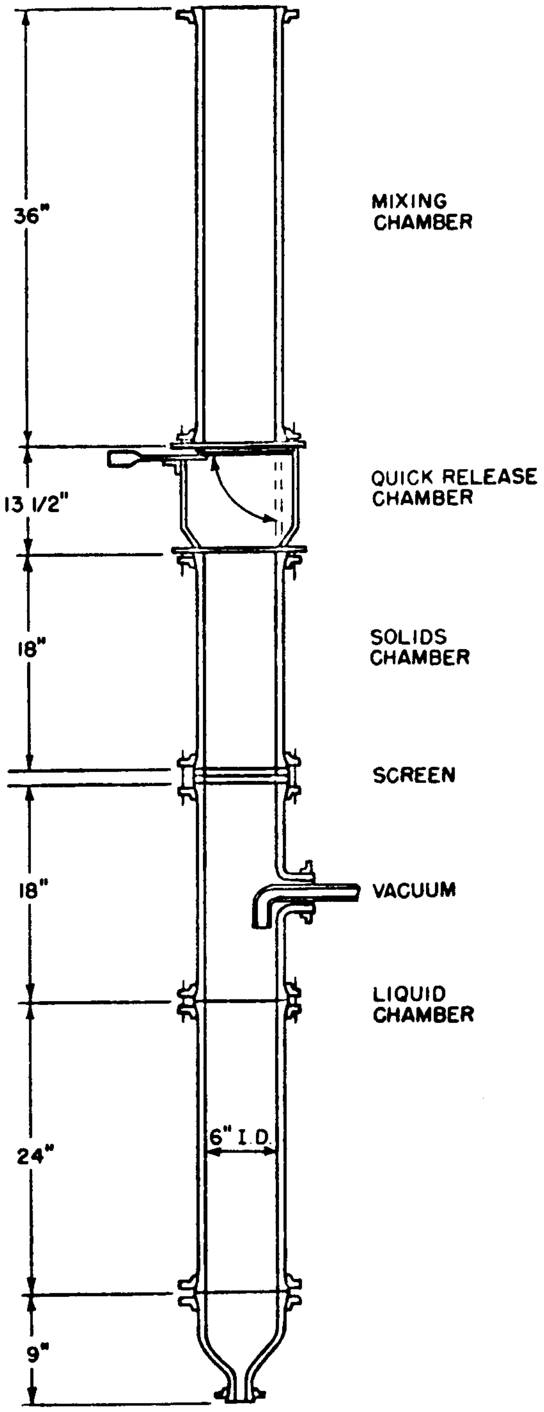

FIGURE 2. Experimental anhydrous test unit.

EXPERIMENTAL DATA

The experimental studies were carried out in a batch system which simulated a dynamic three-stage drain circuit. The apparatus (Fig. 2) consisted of:

1. a mixing chamber for slurrying the oil sand with recycle solvent-bitumen mixture;
2. a quick-release chamber to allow discharge of the slurry onto the draining surface at the completion of the specified mixing residence time;
3. a solids chamber to contain the *solid-water-particle* bed;
4. a draining surface;
5. a drain-liquid chamber to receive drain liquor;
6. a vacuum surge system.

The experimental program initially consisted of a series of tests to establish procedures and to accumulate a supply of recycle oil for each solvent type to be tested. There followed a statistically designed study to establish the effect in the extraction step on recovery, drain rates, and product quality, of the following variables: (1) solvent type (four levels); (2) solvent amount (three levels); (3) sand-bed depth (three levels).

With replicates based on a partial factorial design, this study consisted of a total of forty-two separate runs. Subsequent studies were carried out to test the effect of temperature and pressure differential on the extraction phase. A series of runs was carried out at the optimum conditions established from the above studies to establish reproducibility. Finally, a series of runs at final conditions made to produce drained solids for solvent-recovery studies. The solvent recovery approaches evaluated were as follows: (1) vacuum flashing; (2) steam stripping; (3) water displacement.

A typical test-procedure is illustrated in figure 3 in the form of a flow diagram. The oil sand was slurried with recycle oil and solvent to yield the specified solvent to bitumen ratio and a slurry of approximately 55 per cent solids. The resultant slurry was mixed for 5 minutes by a turbine-type mixer. The temperature of the entire test column was maintained by a hot-air jacket (generally 100°F). A vacuum was produced in the lower column and surge system sufficient to yield the desired pressure drop across the *solid-water* bed (generally 12 to 15 inches of mercury). The slurry was dropped on the drain surface. A high-speed absolute-pressure recorder-timer was used to time the draining to the nearest one hundredth of a second and to actuate the pressure-equalizer valve when vacuum loss indicated liquid breakthrough. The drain liquid was removed, weighed, and sampled, for: (1) water; (2) solids; (3) gravity; (4) viscosity; (5) solvent to bitumen ratio.

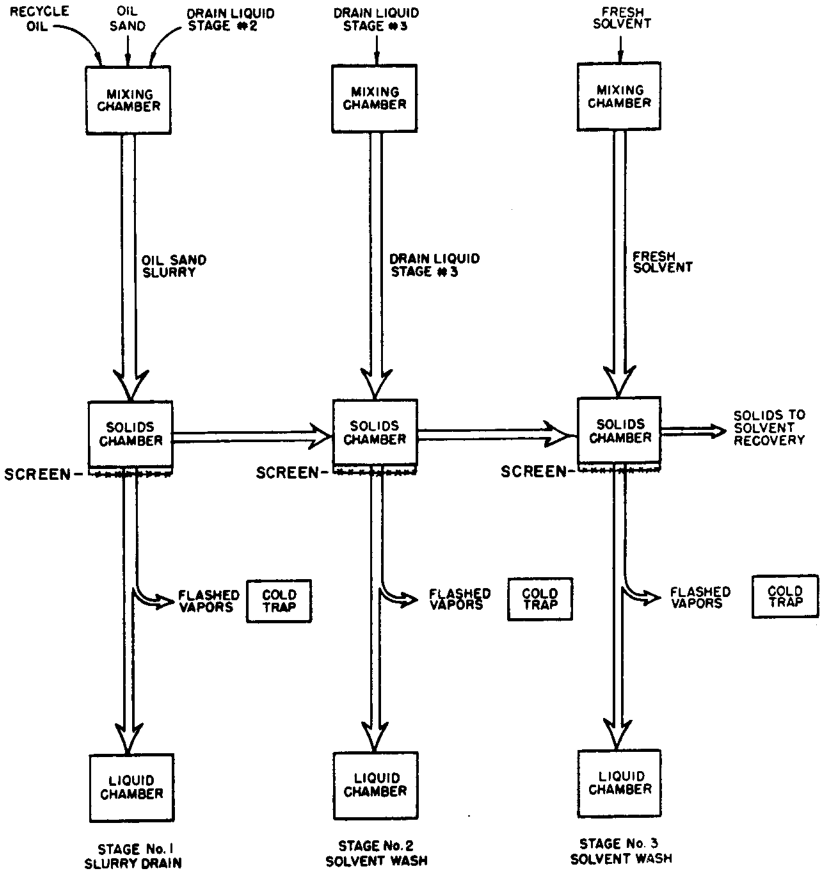

FIGURE 3. Flow diagram of test operation.

The solids chamber with screen was removed, weighed, and replaced. The first solvent wash consisted of placing third-stage drain liquid in the mixing chamber and dropping it onto the first-drain solids. The procedure for weighing and sampling was repeated as for above. The third stage was similar to the second stage, but fresh solvent was used. After the third stage, the entire *solid-water* bed was placed in a 5-litre kettle and vacuum-distilled to remove solvent and water. The resultant dry solids were ground in a catalyst grinder, blended, and sampled for bitumen content.

For evaluating solvent recovery, the solids chamber was removed and weighed without disturbing the *solid-water* bed. Water displacement was evaluated both upward and downward. The downward problem has been previously discussed: the bed blinded. Upward displacement was not effective, particularly in recovered-solvent quality. Vacuum flashing showed promise but was limited in bed depth. Steam stripping was employed by introducing steam above the *solid-water* bed and establishing a pressure drop of 5 inches of mercury by creating a vacuum below the bed. This yielded good results in recovery and solvent quality. Further bitumen recovery was also achieved by an initial displacement of diluted bitumen when steam was heating the bed to flash temperature prior to steam stripping of solvent.

DISCUSSION

The results of the research project yielded overall bitumen recoveries in excess of 90 per cent at established conditions. The first-drain liquid (product oil) averaged 1.5 per cent solids and 2.0 per cent water. However, these samples included occasional *solid-water* particles which passed the coarse-drain medium and short settling (30 minutes) at ambient temperature yielded a 95 per cent supernatant of less than 0.5 per cent bottom sediment and water. Temperatures to 130°F were tested and could be tolerated. It is to be expected that higher temperatures could cause film rupture and destruction of the vital *solid-water* structure. The drain rates for each stage are present in graphic form. (Figs. 4, 5 and 6). These graphs were established by computer correlation of the experimental data. Drain-rate equations of high significance, statistically, were developed: multiple correlation coefficients were greater than 0.9. The viscosity of the drain liquid and the depth of the bed were the most dominant variables. Pressure differential only remained in the first-drain equation, but this was not a designed variable and varied only over a narrow range. Indication was that pressure drops in excess of 15 inches of mercury compressed the bed and thereby substantially reduced drain rates. The drain-rate equations were as follows:

First drain

$$\text{Log drain rate} = 1.31 + 1.00 \log \Delta P - 1.10 \log \text{Vis.} - 1.45 \log \text{bed depth}$$

Second drain

$$\text{Log drain rate} = 2.23 - 1.32 \log \text{Vis} - 1.48 \log \text{bed depth}$$

Third drain

$$\text{Log drain rate} = 1.91 - 1.14 \log \text{Vis} - 1.40 \log \text{bed depth}$$

where:

Drain rate is expressed in gallons per minute per square foot of drain area

ΔP=Pressure differential in inches of mercury

Vis=Viscosity in centistokes

Bed depth is in inches.

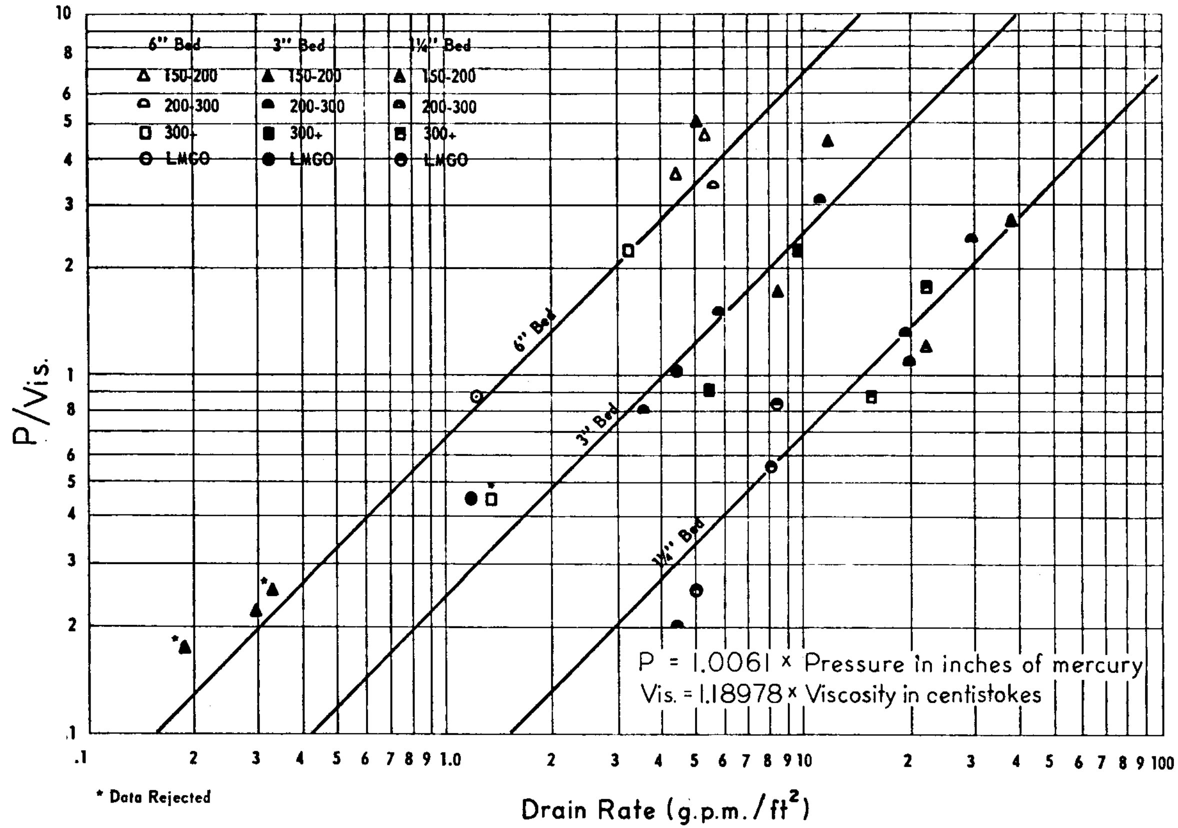

FIGURE 4. Effect of viscosity on drain rates: Viscosity versus drain rate (First drain).

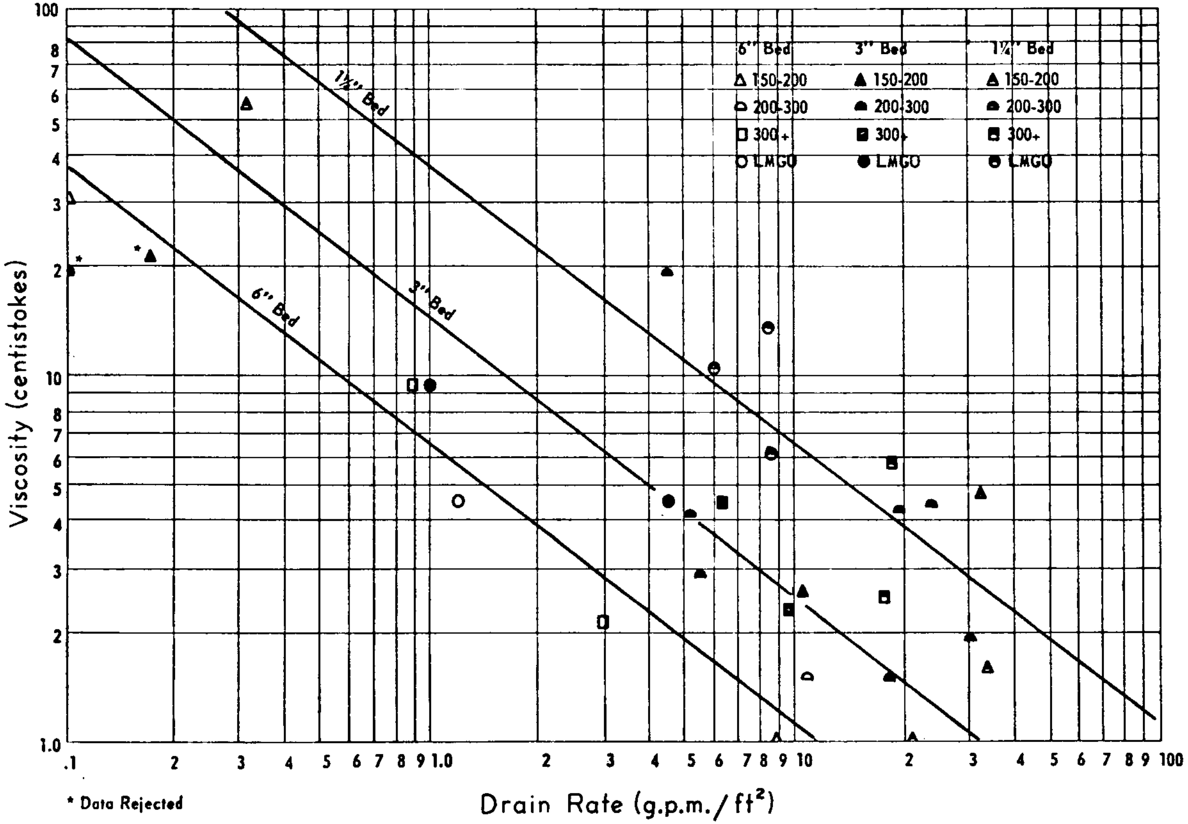

FIGURE 5. *Effect of viscosity on drain rates: Viscosity versus drain rate (Second drain).*

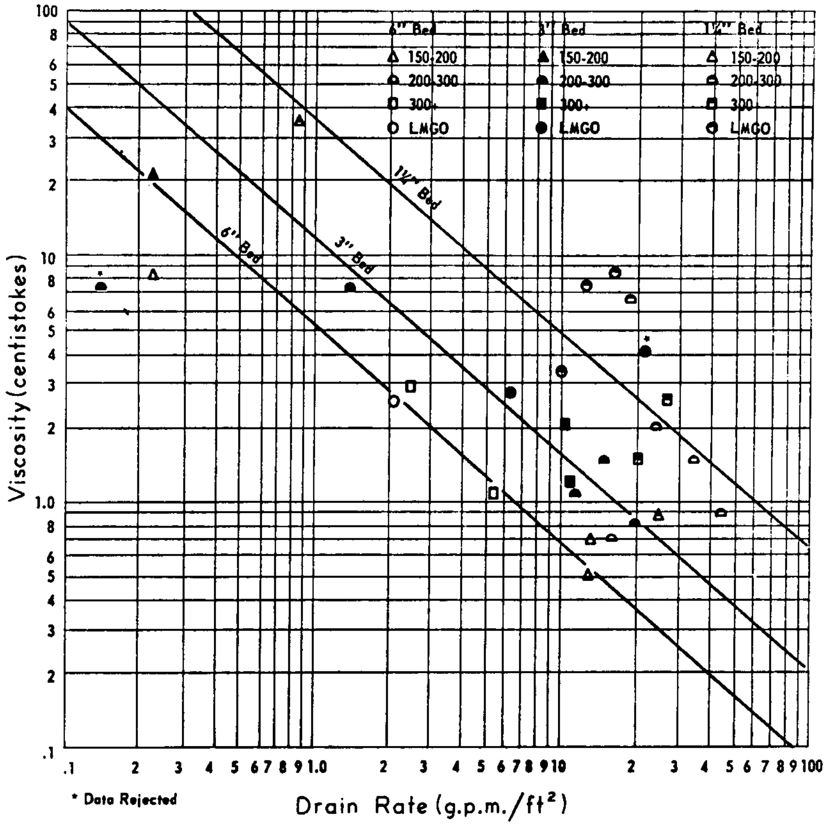

FIGURE 6. Effect of viscosity on drain rates: Viscosity versus drain rate (Third drain).

As presently conceived, the commercial plant would include a slurrying step with the slurry placed on a moving belt which would pass over three drain chambers and a solvent-recovery chamber. The product oil would go to a fractionation tower for solvent recovery and raw bitumen product to refining from whence make-up solvent would be realized. Figure 7 illustrates a flow diagram of a commercial process scheme with accompanying material balance (Table 1) for 1,000 barrels per hour of raw bitumen product based on average experimental results. It would be expected that efficient large-scale steam stripping of the drained bed would leave substantially less solvent with the discard solids.

CONCLUSIONS

The work reported herein has resulted in development of an anhydrous process which can be used as an alternate to one of the water processes for extraction of bitumen from Athabasca oil sand.

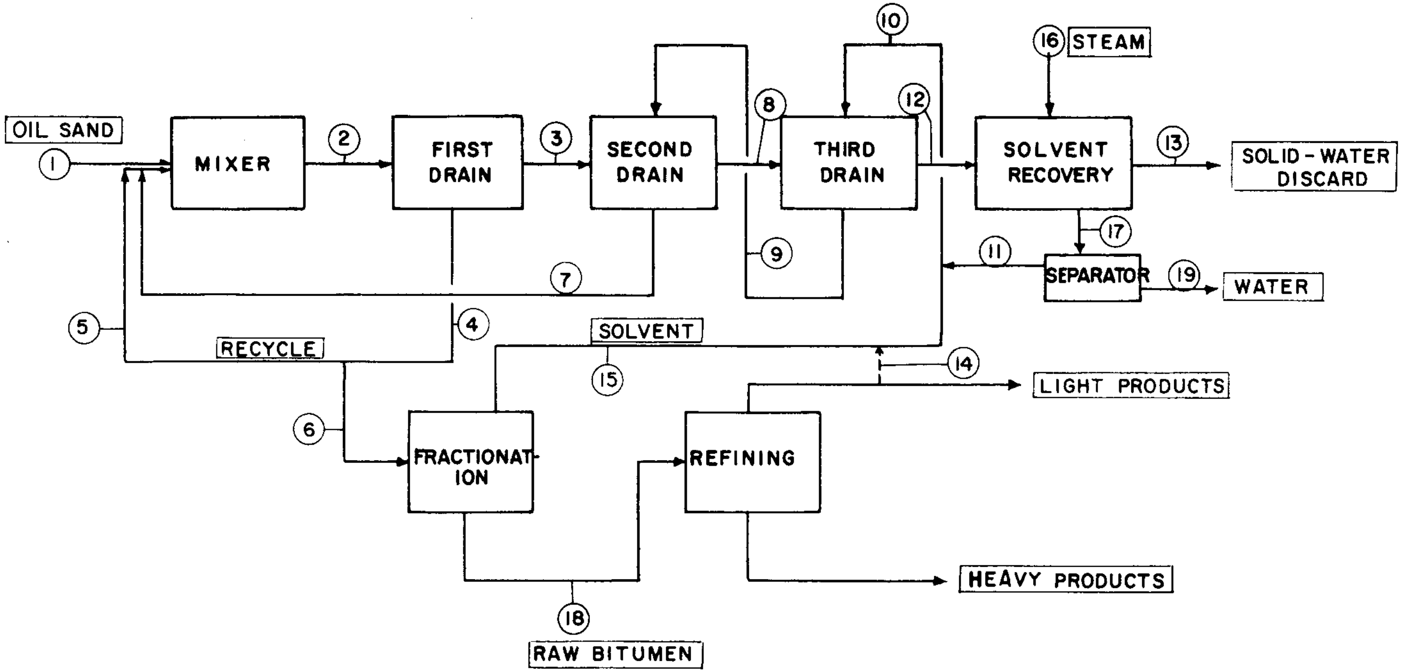

FIGURE 7. *Flow diagram of typical commercial scheme.*

TABLE 1. TYPICAL COMMERCIAL MATERIAL BALANCE AT 1,000 BARRELS PER HOUR THROUGHPUT
(SEE FIGURE 7)

	Stream Number									
	1		2		3		4		5	
	A*	B†	A	B	A	B	A	B	A	B
Bitumen	380.8	11.7	1142.3	23.2	146.3	4.7	996.1	55.3	642.0	55.3
Solvent	—	—	860.5	17.5	110.2	3.5	750.3	41.6	483.6	41.6
Water	234.3	7.2	252.6	5.1	231.0	7.4	21.6	1.2	13.7	1.2
Solids	2639.4	81.1	2668.7	54.2	2634.4	84.4	34.2	1.9	21.7	1.9
Total	3254.5	100.0	4924.1	100.0	3121.9	100.0	1802.3	100.0	1161.0	100.0
	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%
Bitumen	752.8	100.0	2258.5	31.9	289.2	9.4	1969.3	49.1	1269.3	49.1
Solvent	—	—	2258.5	31.9	289.2	9.4	1969.3	49.1	1269.3	49.1

	Stream Number									
	6		7		8		9		10	
	A	B	A	B	A	B	A	B	A	B
Bitumen	354.1	55.3	119.5	23.5	53.3	1.7	26.6	5.5	11.4	2.4
Solvent	266.7	41.6	376.9	74.1	185.3	6.0	452.0	93.5	469.4	97.5
Water	7.9	1.2	4.6	0.9	228.3	7.4	1.9	0.4	0.7	0.1
Solids	12.6	1.9	7.6	1.5	2629.7	84.9	2.9	0.6	0.1	—
Total	641.3	100.0	508.6	100.0	3096.6	100.0	483.4	100.0	481.6	100.0
	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%
Bitumen	700.0	49.1	236.3	19.1	105.3	3.4	52.5	4.2	22.5	1.8
Solvent	700.0	49.1	989.2	79.7	486.3	15.8	1186.3	95.3	1232.0	98.1

	Stream Number									
	11		12		13		14		15	
	A	B	A	B	A	B	A	B	A	B
Bitumen	11.4	5.5	38.1	1.3	26.7	0.9	—	—	—	—
Solvent	192.3	93.9	202.7	6.5	10.3	0.3	10.3	100.0	266.7	100.0
Water	0.7	0.5	227.0	7.3	363.3	12.0	—	—	—	—
Solids	0.1	0.1	2627.0	84.0	2627.0	86.8	—	—	—	—
Total	204.5	100.0	3094.8	100.0	3027.3	100.0	10.3	100.0	266.7	100.0
	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%
Bitumen	22.5	4.2	75.3	2.4	52.8	1.9	—	—	—	—
Solvent	504.8	95.5	532.0	17.3	27.2	1.0	27.2	100.0	700.0	100.0

	Stream Number							
	16		17		18		19	
	A	B	A	B	A	B	A	B
Bitumen	—	—	11.4	5.3	354.1	96.6	—	—
Solvent	—	—	192.3	88.8	—	—	—	—
Water	149.1	100.0	12.9	5.9	—	—	12.2	100.0
Solids	—	—	0.1	—	12.5	3.4	—	—
Total	149.1	100.0	216.7	100.0	366.6	100.0	12.2	100.0
	gpm	vol%	gpm	vol%	gpm	vol%	gpm	vol%
Bitumen	—	—	—	—	22.5	4.1	—	—
Solvent	—	—	700.0	100.0	504.8	91.2	—	—

*A. Thousands of pounds per hour

†B. Weight per cent

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LOW-ASH ASPHALT AND COKE FROM ATHABASCA OIL-SANDS OIL*

D. S. PASTERNACK**

ABSTRACT

Dehydrated Athabasca oil-sands oil, produced either by the hot-water-washing process or the cold-water-washing process, on normal coking produces a coke which has too high an ash content to be of value for metallurgical purposes. Two simple methods are outlined to obviate this, each representing a controlled partial-deasphalting based on partial thermal cracking of the oil. The extraneous finely divided solids present in the dehydrated oil apparently serve as nuclei for the coke which is produced during thermal cracking, an association which facilitates subsequent removal of the solids from the residue by filtration. The filtrates represent a relatively low-ash asphalt, satisfactory for production of metallurgical coke, carbon for electrodes, bituminous paints, or for other uses in which a low ash is a prerequisite or desirable.

The first method consists of thermally cracking the oil, followed by filtration of the residue. The ash content of the coke produced from the filtrate depends upon the rate and extent of the thermal cracking, but is generally less than 0.5 per cent.

In the second method, the residue (or the filtrate) from the thermal cracking is diluted with distillate, and then filtered or centrifuged to remove the insolubles. Prior removal from the oil of 40 per cent by weight of distillate by slow thermal cracking results in the recovery of an extract with about 40 per cent of the original asphaltenes and which provides a coke with an ash content of about 0.07 per cent.

INTRODUCTION

In the recovery of the oil from the Athabasca oil sands by means of the hot-water-washing process (Pasternack *et al.*, 1951) or the cold-water-washing process (Djingheuzian, 1951), the oil products after dehydration contain varying percentages of mineral matter depending on the processing conditions. Since much of this mineral matter consists of clay and very fine sand which will not settle out of the hot dry oil, coke produced from the oil contains much too high an ash content—2.8 to 3.8 per cent when using settled diluted wet oil for coke production (Bowles and Booth, 1947; Sterba, 1951)—to be satisfactory for metallurgical purposes or for electrode carbon. Similarly, the dry oil is unsatisfactory for the preparation of bituminous paints. Consequently, interest heretofore has centred on the yield and quality of the distillates produced from the oil.

Sterba (1951) reported on the thermal cracking of the oil both on a batch and continuous basis, and stated that the true density, apparent density, and cell space of the bitumen coke are typical of

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*Contribution No. 223

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those observed for petroleum coke produced from high sulphur feed stocks. Peterson and Gishler (1951) used a fluidized bed for their thermal and catalytic cracking investigations. Boomer and Saddington (1931) and Warren *et al.* (1951) studied the hydrogenation of the oil. Peterson and Boomer (1944) were interested in the viscosity-breaking of the oil, that is, in reducing its viscosity and pour point by mild thermal cracking under pressure, without removal of potential distillate, in order to make it easier to handle.

The present investigation was undertaken to study changes which take place in the residues during thermal decomposition of the oil at atmospheric pressure, with simultaneous removal of distillate, and to determine whether any useful products—aside from the production of additional distillate and an impure coke valuable only as a fuel—might be obtained from the residues. Specifically, it was considered desirable to develop a simple practical method for obtaining, from the residues, a low-ash asphalt which might be utilized for the production of low-ash coke for metallurgical purposes, the specifications generally requiring that the ash content of the coke be less than about 0.5 per cent.

The oil used had been separated from the sand at Bitumount by the hot-water-washing process, and then dehydrated at 300 to 310°F with a back pressure of 15 to 20 p.s.i.g. (Clark and Pasternack, 1948). The dehydration removed about 3 weight per cent of light ends, which were not returned to the dry oil. The dehydrated oil still contained 0.5 weight per cent of water and 3.3 weight per cent of finely-divided solids, a portion of which was clay. At no time had diluent been added to the oil during its recovery or dehydration, which accounts for the relatively high solids content of the dry oil. The coarse solids had settled out while the wet oil was kept warm as a feed stock for the dehydration. Since the Ramsbottom carbon residue of the dehydrated oil is 12.0 weight per cent, coke produced from the oil in a normal manner would have an ash content of approximately 20 per cent.

EXPERIMENTAL PROCEDURE

The distillations (thermal cracking with simultaneous removal of distillate) were performed at atmospheric pressure in 1-litre short-necked flasks (Brown-Duvel moisture test flasks with straightened side-arm). A Cenco giant adjustable 500-watt heater was used for the heating and provided satisfactory control, the portion of the flask above the heater being adequately insulated with asbestos. A charge of 500 grams of the dehydrated oil containing 481 grams of pure oil was used for each distillation.

Both rapid and slow distillations of the oil were made. The slow distillations were carried on at a rate which, generally, provided a distillate recovery of approximately 50 weight per cent in 4 hours as compared to 0.8 hour for the rapid distillations. To introduce variations, one slow distillation was performed at a rate which was 50 per cent slower than the other slow distillations, while two of the rapid distillations were 50 per cent faster than the other rapid distillations. However, even these were slow as compared to the instantaneous and more complete distillation which takes place in a fluidized bed (Peterson and Gishler, 1951).

The very short neck on the flask permitted adequate control of the rate of refluxing during the slow distillations, and minimized refluxing during the rapid distillations. Throughout the rapid distillations, both the liquid and vapour temperatures rose continuously. In the slow distillations, the vapour temperature was made the controlling factor and the liquid temperatures reached the maxima shown in table 1.

Two methods were used to obtain a low-ash product. In the first method, the residue from the thermal cracking was filtered through a No. 1 Whatman filter paper on a Büchner funnel (127 mm o.d.). The Büchner, well insulated with asbestos pipe-covering, was heated in a gas oven to about 550°F immediately prior to the filtration, and the filter paper was moistened with about 3 ml. of warm high-ends of kerosene. The residue from the thermal cracking was poured into the Büchner at a temperature of about 650 to 700°F and a watchglass, and inverted heater on low heat, placed over the top. In this way the contents of the Büchner were kept reasonably hot until the end of the filtration. The ash and Ramsbottom carbon residue contents of the filtrates were then determined. Extraction with toluene of a portion of the filtrate and of the entire residue exclusive of filtrate, permitted a close approximation of the amount of coke produced during the thermal cracking.

In the second method, the residue (or the filtrate) from the thermal cracking was diluted with one or more volumes of the distillate, and this was followed by filtration or centrifuging to remove the insolubles. The diluent was then removed by distillation, and the ash and Ramsbottom carbon residue contents of the extracts determined.

In order to follow, to some extent, the complex changes which take place in the Athabasca oil-sands oil during thermal cracking, especially with respect to the reduction which shows up in the ash content of the filtrates due to coke formation, determinations were made of the percentages of insolubles in the filtrates and in extracts from the filtrates (or residues). For this purpose toluene, varsol or kerosene, and *n*-pentane were used as solvents, the dilution generally being 30:1. The ash and Ramsbottom carbon residue contents of the insolubles and of the extracts were determined.

RESULTS AND DISCUSSION

Decomposition of the oil became rapid when the liquid temperature of the oil reached 665°F, although signs of cracking were evident at about 650°F. However, up to a liquid temperature of 665°F only 1 to 2 weight per cent of distillate had been obtained, exclusive of water. Consequently, Bitumount oil-sands oil *in situ* contains, in total, about 4 weight per cent of distillate obtainable at atmospheric pressure without cracking (including the 3 weight per cent removed during the dehydration).

YIELD OF FILTRATE

Figure 1 shows that the maximum yield of filtrate from both rapid and slow distillations was obtained when 35 to 40 weight per cent of distillate had been recovered. Under the filtering conditions employed, the maximum amount of filtrate possible would be about 95 weight per cent for the slow distillations and about 90 weight per cent for the rapid distillations. In all cases, at least 80 to 90 per cent of the recovered filtrate was obtained in less than one-half hour.

Until slightly more than 30 weight per cent of distillate has been produced, all filtrates flow at room temperature. This applies to both the rapid and slow distillations. Actually, the consistency of the filtrates at room temperature up to this point is only slightly greater than that of the original dehydrated oil, but some distillate from the cracking is always present in the residue when it is poured into the funnel (more from the slow distillations).

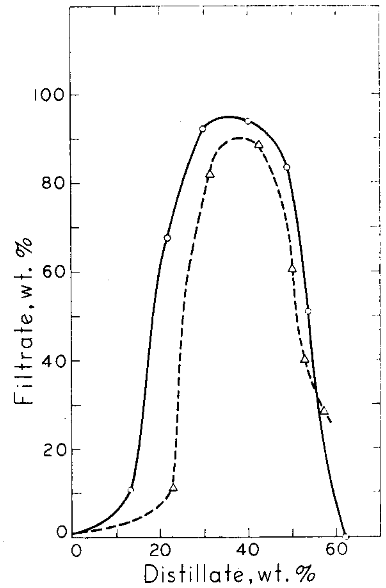

FIGURE 1. Filtrate, as weight per cent of residue on mineral-matter-free and coke-free basis, versus distillate recovery. Circles are results from slow distillations. Triangles are results from rapid distillations.

Even when more than 50 weight per cent of distillate has been produced, the residues start off filtering well. Later, under the experimental conditions of the filtrations, sufficient cooling takes place with time to slow down the filtrations and eventually bring them to a stop. When the amount of distillate approached or exceeded 50 weight per cent, the filtrations ceased within 18 to 30 minutes due to congealing of the filtrate inside the Büchner. This resulted in plugging of the outlet and a decreased recovery of filtrate as shown in figure 1. Nevertheless these viscous residues will filter readily, if the filtering medium and the residues are hot enough. It should not be difficult to arrange this in a commercial operation, and the filtration could be made continuous by utilizing some method for scraping off the filter cake. Probably the filtering medium could be somewhat more porous than the No. 1 Whatman filter paper without decreasing the purity of the filtrate. Each of these three improvements should have a pronounced effect in increasing the rate of filtration and the total recovery of filtrate.

When the dehydrated oil is heated to 625°F, that is, below the cracking point, and poured into the hot Büchner, only a trace of filtrate is obtained (Table 1, col. 9); presumably, the finely divided mineral matter in the oil immediately plugs the pores of the filter paper. At some point during the distillation coke formation starts, and apparently tends to become associated with the mineral particles, the latter probably serving as nuclei. In the early stages of the distillations very little coke is produced; the amount which is formed presumably adheres to a portion of the mineral particles enabling about 11 weight per cent of filtrate as shown in figure 1, to be obtained before the filter becomes plugged. As the distillations progress more coke is formed, and, eventually, a sufficient proportion of the mineral particles are coated with coke, or have become associated with coke, so that the filtrations are able to proceed to completion if the temperature of the residue is maintained sufficiently high.

COKE PRODUCTION DURING PARTIAL THERMAL-CRACKING

Determination of the toluene-insoluble content of the filtrates and the toluene-insoluble content of the balance of the residues enabled the amount of coke produced during each distillation to be calculated, by making allowance for the quantity of mineral matter in the oil charge. Since, in some instances, the quantity of coke was small compared to the quantity of mineral matter, the values shown in table 1 (col. 10) should be considered as relative rather than absolute amounts.

As illustrated in figure 2, the proportion of coke produced increases with the duration of distillation, and the linear relationships are markedly different for the comparatively rapid and slow distillations.

ASH CONTENT OF COKE FROM FILTRATES

To test the efficacy of the filtrations, portions of the filtrates were ignited in a muffle furnace at 725°C. In addition, Ramsbottom carbon residue determinations at 1022°F were made on the filtrates. From these values, the ash contents of the carbon residues were calculated. Although the Ramsbottom carbon residue material is not identical to normal coke which would be produced from filtrates in a coking operation, it is similar. Consequently, the data in table 2 (col. 6) indicate that the ash content of normal coke produced from the filtrates could be held to less than 0.5 per cent if about 40 weight per cent of distillate were removed during the distillation. This ash

TABLE 1. COKE PRODUCTION DURING THERMAL CRACKING OF ATHABASCA OIL-SANDS OIL

(1) Expt. No.	(2) Duration of dist'n from 650°F (minutes)	(3) Max. liquid temp. (°F)	(4) Max. vapour temp. (°F)	(5) Distillate obtained[a] (wt.%)	(6) Distillate obtained[a] (vol.%)	(7) Gravity of distillate at 60°F (°A.P.I.)	(8) Sulphur content of distillate by Dietert method (wt.%)	(9) Filtrate, (wt. % of total residue on mineral-matter-free and coke-free basis)	(10) Coke produced, based on oil charge pure basis (wt.%)
35 R	23	770	595	22.9	26.1	28.0	2.64	11	not determined
21 R	38	796	615	31.6	36.0	28.1	2.90	82	0.58
20 R	39	820	655	42.5	47.8	26.3	3.04	89	0.61
22 R	49	830	650	50.2	56.7	26.8	3.10	61	0.66
36 R	36	830	696	53.0	59.4	25.5	3.12	40	0.27
37 R	33	845	714	57.2	63.7	24.5	3.12	28	0.33
Dry Oil								<1	
34 S	36	722	531	13.1	14.9	28.0	2.22	11	trace
18 S	94	755	550	21.7	25.3	32.1	2.70	68	trace
19 S	131	769	550	30.0	35.2	32.6	2.75	92	0.33
13† S	209	773	559	40.1	46.7	31.7	2.77	94	0.90
14 S	228	782	568	48.9	56.9	31.6	2.84	84	1.29
16 S	383	794	550	53.5	62.8	32.9	2.73	51	2.62
15 S	320	800	628	62.0	71.0	28.9	2.92	[b]	could not determine
31‡ S	480	794	550	53.4	62.6	32.6			3.60
32 S		850	562	60.0	69.9	31.9			[c]
33 S		859	562	60.3	70.6	32.4			[c]

R Comparatively rapid thermal cracking.

S Slow thermal cracking.

† Experiments 1 to 12 were utilized in working out the experimental procedures.

‡ Experiments 23 to 30 were utilized in obtaining a stock supply of filtrate for experiments 31, 32 and 33.

[a] Does not include the 3 weight per cent of distillate obtained during the rapid dehydration of wet Athabasca oil-sands oil produced by the hot-water-washing process. This distillate was not returned to the dry oil used for the "numbered" distillation experiments. Its sulphur content was 0.94 weight per cent.

[b] No filtrate; congealed too quickly.

[c] Filter paper ruptured.

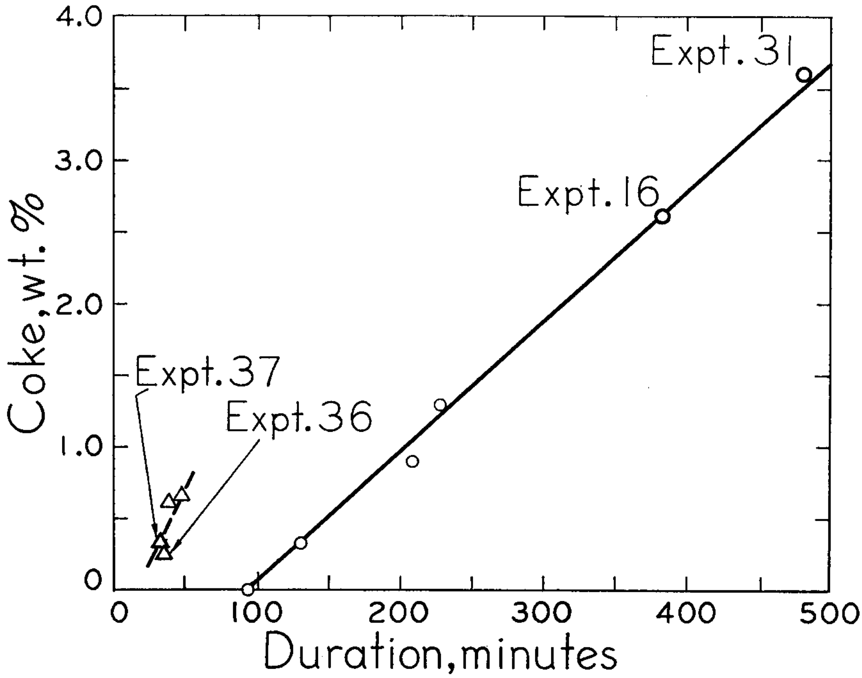

FIGURE 2. Coke production versus duration of thermal cracking, based on oil charge (pure basis).
Circles are results from slow distillations.
Triangles are results from rapid distillations.

content would meet the specification for petroleum coke for metallurgical purposes. The filtrates themselves may meet the ash specification for other uses.

Since the Ramsbottom carbon residue determination provides a measure of the coking tendency and represents a fairly close approximation of the amount of normal coke which could be produced from the filtrate, table 2 (col. 7) shows, approximately, the percentages of low-ash coke which could be obtained from the dehydrated oil by partial thermal cracking, filtration of the residue, and then coking of the filtrates. For example, with 40 weight per cent distillate recovery, the yield of low-ash coke, based on the oil charge (pure basis) would be about 15.0 weight per cent from a slow distillation and about 13.8 weight per cent from a comparatively rapid distillation, the coke having ash contents of 0.38 and 0.45 per cent respectively.

Since only a trace of coke was produced in experiments 18 and 34, it appears from the data in table 2 (and verified by figure 3) that the oil charge (pure basis) would have provided approximately 0.0690 weight per cent or 690 p.p.m. of ash. On making allowance for the 3 weight per cent of light ends removed during the dehydration, the

TABLE 2. ASH CONTENT OF CARBON RESIDUES FROM FILTRATES, AND INCREASE IN CARBON RESIDUE PLUS COKE

(1) Expt. No.	(2) Distillate (wt.%)	(3) Total filtrate possible[a] (wt.%)	(4) Ash content of filtrate at 725°C (wt.%)	(5) Ramsbottom carbon residue of filtrate at 1022°F (wt.%)	(6) Ash content of carbon residue[b] (%)	(7) Carbon residue of filtrate at 1022°F based on oil charge, pure basis[c] (wt.%)	(8) Coke produced during dist'n, based on oil charge, pure basis (wt.%)	(9) Carbon residue plus coke, based on oil charge, pure basis (wt.%)	(10) Increase in carbon residue plus coke, based on oil charge, pure basis (wt.%)
Dehydrated oil (pure basis)			0.069[d]			12.0		12.0	
35 R	22.9	75.2	0.091						
21 R	31.6	65.3	0.097	20.7	0.47	13.5	0.58	14.1	2.1
20 R	42.5	54.0	0.116	25.5	0.45	13.8	0.61	14.4	2.4
22 R	50.2	45.5	0.130	30.8	0.42	14.0	0.66	14.7	2.7
36 R	53.0	42.1	0.147				0.27		
37 R	57.2	37.5	0.169				0.33		
34 S	13.1	85.1	0.081						
18 S	21.7	75.9	0.091	18.2	0.50	13.8	trace	13.8	1.8
19 S	30.0	66.4	0.099	21.3	0.46	14.1	0.33	14.4	2.4
13 S	40.1	54.0	0.105	27.7	0.38	15.0	0.90	15.9	3.9
14 S	48.9	44.2	0.122	35.9	0.34	15.9	1.29	17.2	5.2
16 S	53.5	36.8	0.116	45.8	0.25	16.9	2.62	19.5	7.5

[a] Total filtrate possible: subtract (distillate + gas loss + coke) from oil charge, pure basis.

[b] $\frac{\text{col. 4}}{\text{col. 5}} \times 100.$

[c] $\frac{\text{col. 3} \times \text{col. 5}}{100}$

[d] From figure 3.

N.B. Column 7 provides a fairly close approximation of the amount of normal coke which could be produced from each filtrate, based on the oil charge (pure basis), and column 6 indicates the approximate ash content of the normal coke.

oil on an *in situ* basis would provide 670 p.p.m. of ash in the form of oxides.

Figure 3 shows also that as coke production increases, the total ash content of the total possible filtrate decreases. The linear relationship shown by the upper part of the graph provides a ratio of coke produced to ash reduction of approximately 85:1. The curved portion represented by the dashed line will be dealt with later.

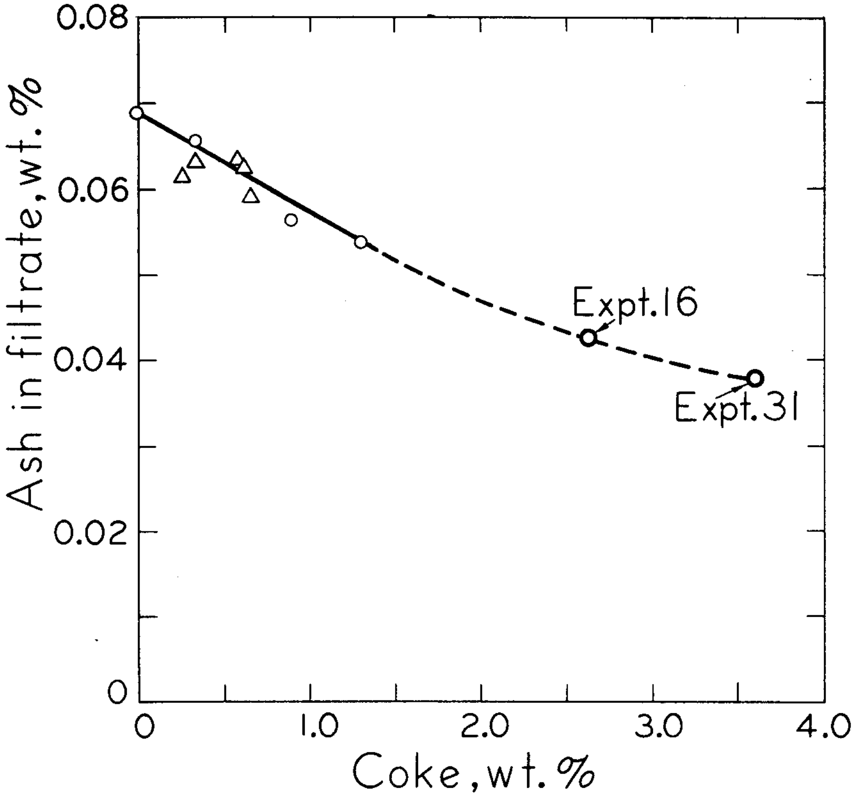

FIGURE 3. Reduction in ash content of filtrate due to coke production, based on oil charge (pure basis).
Circles are results from slow distillations.
Triangles are results from rapid distillations.

Scott *et al.* (1954) stated that in an analysis of a typical Athabasca oil-sands oil, they obtained 360 p.p.m. V_2O_5, 93 p.p.m. NiO and 106 p.p.m. Fe_2O_3, for a total of 559 p.p.m., and that the metals themselves are present in combination with the oil. Since slight amounts of other metals are also present in a typical oil-sands oil (Scott *et al.*, 1954), it appears that the filtration of the residues through a No. 1 Whatman filter paper removes all the free inorganic mineral matter and that, for all practical purposes, only metals actually in combination with the oil pass through the filter.

TABLE 2. ASH

TABLE 3. COMPOSITION OF RESIDUES AT END OF THERMAL CRACKING
(Based on oil charge, pure basis)

(1) Expt. No.	(2) Distillate (wt.%)	(3) Coke (wt.%)	(4) Tl in filtrate (wt.%)	(5) Vl in toluene extract (wt.%)	(6) Asphaltenes in varsol extract (wt.%)	(7)	(8) "Difference resins" in asphaltenes (wt.%)	(9) Coke +Tl (wt.%)	(10) Coke +Tl +Vl (wt.%)	(11) Coke+Tl+Vl +asphaltenes (wt.%)
Dehydrated oil (pure basis)				1.6	18.1†	16.8*	33.3			
21 R	31.6	0.58	trace	4.4				0.6	5.0	
20 R	42.5	0.61	trace	6.7				0.6	7.3	
22 R	50.2	0.66	0.1	8.1				0.8	8.9	
34 S	13.1	trace	trace							
18 S	21.7	trace	trace	3.5				trace	3.5	
19 S	30.0	0.33	trace	6.5				0.3	6.8	
13 S	40.1	0.90	2.7	7.1	7.3	16.5*	33.7	3.6	10.7	18.0
14 S	48.9	1.29	5.8	5.8				7.1	12.9	
16 S	53.5	2.62	10.6	4.4				13.2	17.6	
31 S	53.4	3.60	10.0	4.4	5.4	24.1*	32.6	13.6	18.0	23.4
32 S	60.0					55.0*	69.6			
33 S	60.3				4.4	54.7*	72.3		23.1	27.5

Tl—Toluene-insoluble material.

Vl—Varsol-insoluble material. Varsol is that portion of gasoline boiling, primarily, between 320° to 375°F.

† —Total asphaltenes in the dehydrated oil (pure basis); includes the varsol-insoluble material which, apparently, was produced from the asphaltenes during removal of the toluene from the toluene extract.

* —Wt.% of asphaltenes based on varsol-soluble portion of residue.

Table 2 (col. 10) gives the increase in Ramsbottom carbon residues shown by the distillation residues based on the oil charge (pure basis), the latter yielding a carbon residue of 12.0 weight per cent. The coke produced during the distillations has been included, on the assumption that the coke would show no appreciable loss in weight during a carbon-residue determination. The high carbon residues shown by toluene-insoluble material from the filtrates (Table 5) indicate that the assumption is reasonably realistic. The slow distillations produce substantially more carbon residue material including coke than do the comparatively rapid distillations.

COMPLEX CHANGES DURING THERMAL CRACKING

Obviously, the cracking of the oil, besides providing distillate, causes complex changes to occur in the distillation residues which increase their content of carbon-residue-plus-coke. Figure 4 illustrates that, up to about 20 weight per cent of distillate recovery, time is not a factor in these complex changes since, presumably, the lower-boiling

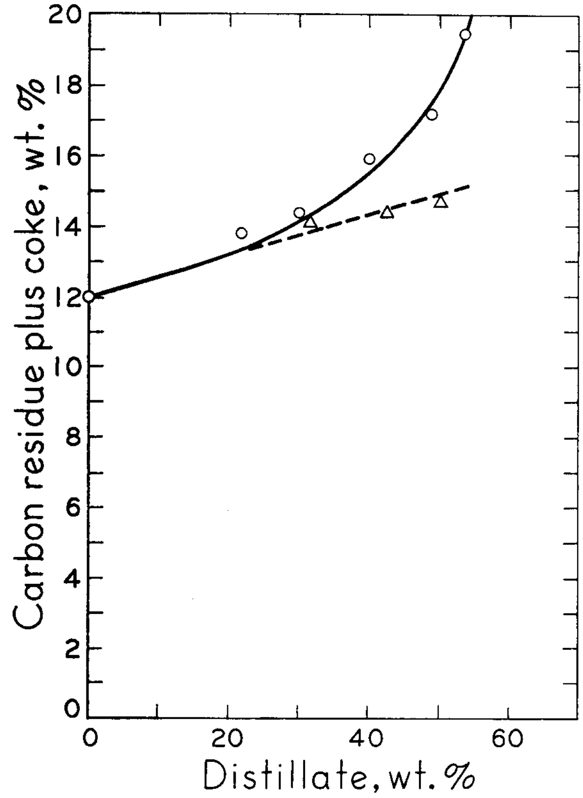

FIGURE 4. Increase of carbon-residue-plus-coke in distillation residue, based on oil charge (pure basis).
Circles are results from slow distillations.
Triangles are results from rapid distillations.

components of the oil are, primarily, those being cracked. However, with increasing distillate recovery, the slow distillations do provide the required time for the complex changes to occur more completely, and the distillation residues then show marked increases in carbon-residue-plus-coke. The temperature of the liquid during the cracking is of much less importance than is duration of cracking in producing these complex changes, because the maximum liquid temperatures during the rapid distillations were substantially higher than for the corresponding slow distillations.

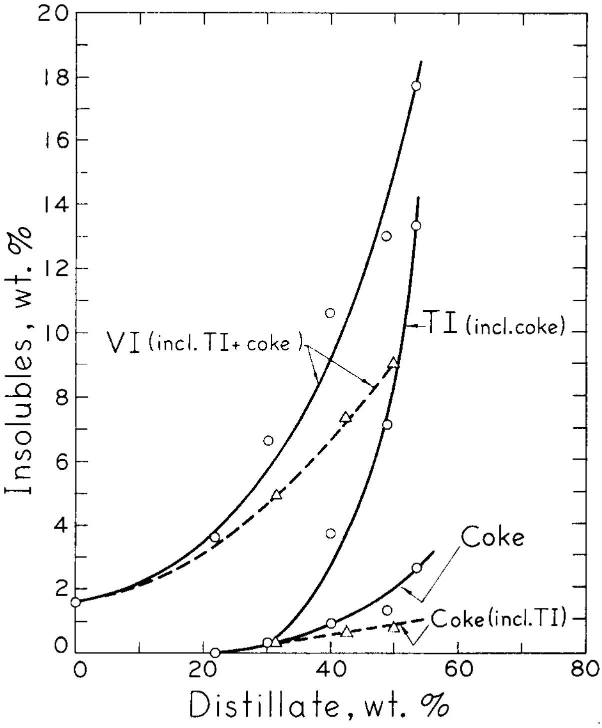

FIGURE 5. Formation of insoluble materials during thermal cracking, based on oil charge (pure basis).
Circles are results from slow distillations.
Triangles are results from rapid distillations.
TI—Toluene insoluble
VI—Varsol insoluble

Table 3 deals with some of the complex changes that occur and shows the composition of the residues at the conclusion of each thermal cracking experiment, based on the oil charge pure basis. It indicates the percentage of coke produced, the amount of toluene-insoluble material present in the filtrate, the amount of varsol-insoluble material contained by the toluene-soluble portion, the asphaltene (*n*-pentane-insoluble) content of the varsol-soluble portion, and the difference-resins content of the asphaltenes (i.e., the portion of the asphaltenes soluble in ethyl ether). Figure 5 illustrates graphically the data for table 3, columns 3, 9, and 10.

Some corresponding data for experiments 31, 32 and 33 are included in table 3 for comparison. For these three experiments a stock supply of filtrate was prepared by repeating the distillation represented by experiment 13. A portion of the stock filtrate was then distilled further until a total of 53.4 weight per cent of distillate (including the initial 40 weight per cent) had been obtained, and the residue filtered. This was called experiment 31, and was comparable to experiment 16 except that the total time for the distillation was 480 minutes as compared to 383 minutes for experiment 16. The maximum liquid and vapour temperatures—794°F and 550°F respectively—were the same for the two experiments. However, the mineral matter present during the distillation represented by experiment 16 was not present during experiment 31.

The same stock filtrate was the starting point for experiments 32 and 33, and a second distillation to maximum liquid and vapour temperatures of 794° and 540°F respectively produced an additional 12 weight per cent of distillate. Then, in experiment 33, the varsol extract of the second distillation residue—which was obtained by extracting the toluene extract with varsol—was cracked slowly up to maximum liquid and vapour temperatures of 859° and 562°F respectively until the total distillate recovery, based on the original oil charge (pure basis), was 60.3 weight per cent. In experiment 32, the *n*-pentane extract of the second distillation residue—which was obtained by extracting the varsol extract with *n*-pentane—was cracked slowly up to maximum liquid and vapour temperatures of 850° and 562°F respectively until the total distillate recovery, based on the original oil charge (pure basis), was 60.0 weight per cent. The purpose of these two experiments was to determine whether the removal of the varsol-insoluble material in experiment 33 and the *n*-pentane-insoluble material in experiment 32 had an appreciable effect on the course of the thermal cracking.

Table 3 shows that in comparatively rapid distillations, with up to 50 weight per cent distillate recovery, practically no toluene-insoluble material is produced except coke, but the amounts of varsol-insoluble material increase. On the other hand, in slow distillations, after a certain amount of varsol-insoluble material is present, the formation of toluene-insoluble material commences and continues quite rapidly, apparently at the expense of the varsol-insoluble material. Even though additional varsol-insoluble material continues to be produced, the net amount of the varsol-insoluble material (exclusive of toluene-insoluble material plus coke) continues to drop. The formation of increasing amounts of toluene-insoluble material in slow distillations also results in increased coke production, presumably

TABLE 4. EFFECT OF REMOVAL OF VARSOL-INSOLUBLE AND *n*-PENTANE-INSOLUBLE MATERIALS ON COURSE OF THERMAL CRACKING

	Expt. 32 Third distillation (based on varsol extract)	Expt. 33 Third distillation (based on varsol extract)	Difference
Asphaltenes present prior to third distillation, wt.%	none	24.0	
Toluene-insoluble material (including coke) produced during third distillation, wt. %	9.2	23.6	14.4
Varsol-insoluble material in toluene-soluble portion, wt. %	6.2	6.6	0.4
Asphaltenes in varsol-soluble portion, wt. %	11.3	16.8	5.5
Distillate produced, wt. %	33.3	33.0	−0.3
Loss, wt. %	4.7	6.1	1.4
n-Pentane extract (by diff.), wt. %	11.3	13.9	2.6
		Total	24.0

at the expense of the toluene-insoluble material; for example, the total coke of 3.6 weight per cent in experiment 31 equals the 3.6 weight per cent of coke plus toluene-insoluble material in experiment 13.

Until about 40 weight per cent of distillate has been obtained in the slow distillations, no new asphaltenes are formed (Table 3, cf. 18.1 weight per cent of asphaltenes in the oil charge, pure basis, with the 18.0 weight per cent of coke plus toluene-insoluble material plus varsol-insoluble material plus asphaltenes in col. 11 for experiment 13). By the time 53.5 weight per cent of distillate has been recovered in a slow distillation, almost all original asphaltenes have been converted to coke plus toluene-insoluble material plus varsol-insoluble material (col. 10, for experiments 16 and 31) and 5.3 weight per cent of new asphaltenes have been formed (col. 11 for experiment 31). These new asphaltenes in experiment 31 have the same difference-resins content as the original asphaltenes. The difference-resins represent that portion of the asphaltenes soluble in ethyl ether (Pasternack and Clark, 1951).

Experiment 33 (Table 3, col. 10) indicates that in slow distillations, the new asphaltenes apparently go through the same cycle of change to varsol-insoluble material, toluene-insoluble material, and coke, as did the original asphaltenes, because the formation of coke plus toluene-insoluble material plus varsol-insoluble material continues to increase with increased distillate recovery while the amount of asphaltenes (Table 3, col. 6) decreases continuously. The formation of varsol-insoluble material from the asphaltenes appears to be only a transitory stage.

In experiments 32 and 33 (Table 3) the asphaltene content of the varsol-soluble portion of the residue has risen to 55 weight per cent and the difference-resins content of these asphaltenes has increased to 70 weight per cent. It may be fortuitous, but both values are greater than the corresponding values shown for experiment 31 by a factor of about 2.2.

Two explanations for these marked increases in the asphaltene and difference-resins contents are the increased maximum liquid temperatures during the slow distillations—850°F in experiment 32 and 859°F in experiment 33—and the increase to 60 weight per cent in the total distillate recovery. The increased maximum liquid temperature seems to be the logical reason, because the asphaltenes in experiment 32 are all new asphaltenes whereas in experiment 33 some of the asphaltenes in the varsol extract which was distilled are still present and yet their difference-resins content has also become 70 weight per cent.

Two interesting speculations arising from the marked increase to 55 weight per cent of the asphaltene content of the varsol extract and to 70 weight per cent of the difference-resins content of the asphaltenes are firstly, whether coke prepared from residues containing substantial proportions of (or comprised entirely of) such asphaltenes would have any special characteristics, and secondly, what is the effect on the physical properties of asphalts and on their usefulness and performance characteristics when substantial proportions of such asphaltenes are present. These speculations arise from the fact that the difference-resins content of these varsol extracts is about five to six times as great as for the other varsol extracts in table 3. However, for asphalts, any countereffects due to variations in the amounts of toluene-insoluble material and varsol-insoluble material would also have to be considered.

TABLE 5. ASH CONTENTS AT 725°C AND RAMSBOTTOM CARBON RESIDUES AT 1022°F OF INSOLUBLES IN FILTRATES AND OF EXTRACTS

	Expt. 13	Expt. 16	Expt. 31
Distillate (based on oil charge, pure basis), wt. %	40.1	53.5	53.4
Total filtrate possible, wt. %	54.0	36.8	
Ash content of filtrate, wt. %	0.105	0.116	
Filtrate:			
Tl in filtrate, wt. %	5.0	27.4	27.1
Ash content of Tl, %	0.975		0.340
Ash content of Tl based on filtrate, wt. %	0.049		0.092
Carbon residue of Tl, %	83.3		89.5
(Ash content of carbon residue from Tl), %	1.17		0.38
Toluene-soluble portion of filtrate:			
Vl in TS (based on filtrate), wt. %	13.1	11.4	11.9
Ash content of Vl, %	0.350		0.182
Ash content of Vl based on filtrate, wt. %	0.046		0.022
Carbon residue of Vl, %	79.0		81.7
(Ash content of carbon residue from Vl), %	0.44		0.22
Varsol-soluble portion of filtrate:			
Ash content of VS, wt. %	0.011		0.006
Ash content of VS based on filtrate, wt. %	0.009		0.004
Carbon residue of VS, wt. %	16.4		27.0
Carbon residue of VS based on filtrate, wt. %	13.4		16.5
Carbon residue of VS based on oil charge pure basis, wt. %	7.2*		6.1
(Ash content of carbon residue from VS), %	0.07*		0.02
Asphaltenes in VS (based on filtrate), wt. %	13.5		14.7
Ash content of asphaltenes, %	0.062		0.022
Ash content of asphaltenes based on filtrate, wt. %	0.009		0.004
Carbon residue of asphaltenes, %	60.0		47.0
(Ash content of carbon residue from asphaltenes), %	0.10		0.05
Ash content of *n*-pentane extract, wt. %	trace		trace
Carbon residue of *n*-pentane extract, wt. %	7.7		6.5

Tl—Toluene-insoluble material.
TS—Toluene-soluble.
Vl—Varsol-insoluble material.
VS—Varsol-soluble.

*Indicates that approximately 7.2 wt. % of normal coke could be produced from the varsol extract, based on the oil charge pure basis, and that the coke would have an ash content of about 0.07%.

Experiment 32 in table 4 shows the production of the various insoluble materials during the thermal cracking of a *n*-pentane extract which contained no insolubles. Comparison of experiment 32 with 33, in which the asphaltenes were not removed prior to the thermal cracking, shows that most of the asphaltenes in the varsol extract have changed to toluene-insoluble material including coke, that a small portion of the original asphaltenes in the varsol extract is still soluble in varsol, and that the formation of varsol-insoluble material (but toluene-soluble) does appear to be a transitory stage in the formation of toluene-insoluble material from the asphaltenes.

ASH CONTENT OF DILUENT EXTRACTS

Table 5 shows the ash and carbon residue contents of the insoluble materials produced during two of the slow distillations, and similar data for the varsol extracts. The latter data shows that, with 40 weight per cent of distillate recovery, the ash content of the varsol extract is reduced to 110 p.p.m., and with 53.4 weight per cent of distillate recovery the ash content of the varsol extract is reduced to 60 p.p.m. Coke produced from these extracts should certainly meet the specifications for good grades of metallurgical coke, electrode carbon and other uses with respect to ash content and metals content.

Extraction with kerosene in place of varsol provides similar results. The entire distillate obtained during the distillation, or portions of it, can also be used. Minor variations show up in the amount of diluent required and in the percentage of precipitate, depending upon which is used, but one or more volumes of diluent per volume of residue (or filtrate) is adequate to remove the bulk of the insolubles. Increased dilution may precipitate relatively small amounts of additional insolubles; however, there appears to be a point beyond which additional dilution results in less insolubles due to their solubility. The insolubles can be removed by filtration or centrifuging. Filtration of the diluent mixture when hot provides slightly less insolubles than does filtration of the same mixture at room temperature.

The diluent extracts of the residues (or filtrates) from comparatively rapid distillations would show similar marked reductions in ash content (Table 3, col. 10). However, for any weight per cent distillate recovery, the reduction following a slow distillation is substantially greater than that from a rapid distillation.

COKE FROM DILUENT EXTRACTS

The amount of coke producible from the extracts depends on the percentage of asphaltenes and asphaltene-forming compounds present in the extracts. The degree of dilution used in removing the insolubles can be adjusted so that the maximum amount of coke meeting ash specifications may be produced from the extracts. As mentioned earlier, the Ramsbottom carbon residue material is similar in amount to normal coke. Thus in a slow distillation, with 40 weight per cent

TABLE 6. APPARENT RELATIVE VARIATIONS IN THE METALS CONTENT OF THE ORIGINAL ASPHALTENES IN ATHABASCA OIL-SANDS OIL, AS INDICATED BY THE ASH CONTENTS OF INSOLUBLES

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9) Ratio, col. 8:col. 4
		Insolubles in residue at end of Expt. 13			Total ash contained by Insolubles			
	(gm.)	(Wt.%, based on oil charge, pure basis)	(as % of original asphaltenes)	Ash content (%)	(Wt.%, based on oil charge, pure basis)	(Wt.%, based on original asphaltenes)	(as % of total ash in oil charge, pure basis)	
Coke in residue	4.3	0.9	5	1.15*	0.010	0.056	15	3:1
Tl in filtrate	13	2.7	15	0.975	0.027	0.146	39	2.6:1
Vl in toluene extract	34	7.1	39	0.350	0.025	0.137	36	0.9:1
Asphaltenes in varsol extract	35	7.3	40	0.062	0.005†	0.025	7	0.2:1
Total	**86.3**	**18.0**	**99**		**0.067**	**0.364**	**97**	
Original asphaltenes	87	18.1	100	0.38	0.069*	0.38	100	1:1

Tl—Toluene-insoluble material.
Vl—Varsol-insoluble material.
* —This value based on figure 3.
† —Represents 110 p.p.m. of ash in varsol extract (see Table 5).

of distillate recovery, the yield of low-ash coke obtainable from the varsol extract of the residue amounts to approximately 7.2 weight per cent, and the coke would have a very low ash content of 0.07 per cent. The carbon residue of this varsol extract, based on the filtrate, is similar to that for the original dehydrated oil (13.4 weight per cent versus 12.0 weight per cent). Removal of the asphaltenes from the varsol extract provides a *n*-pentane extract which still yields 7.7 weight per cent of carbon residue.

VARIATIONS IN METALS CONTENT OF ORIGINAL ASPHALTENES

The difference-resins portion of the asphaltenes in Athabasca oil-sands oil contained only a trace of ash on ignition (Pasternack and Clark, 1951). Consequently, the data in table 5 appear to indicate that the nondifference-resins portion of the original asphaltenes is associated with varying amounts of metals which are responsible for the bulk of the ash. It appears, further, that during these distillations the asphaltenes with the highest percentages of metals are the ones that change most quickly to varsol-insoluble material, then to toluene-insoluble material, and finally to coke. This explains the curved, dashed-line portion of figure 3, in that during the later stages of the distillations increasingly larger percentages of coke production are required to achieve a fixed amount of ash reduction in the filtrate.

Table 6 indicates that, at the end of the distillation in experiment 13, the varsol-soluble portion of the residue or filtrate contains only 7 per cent of the total ash, and that 40 per cent of the original asphaltenes are still present in the varsol extract for use in making good grades of coke since the varsol extract yields only 110 p.p.m. of ash. These remaining asphaltenes show an ash content of 0.062 per cent, which is much lower than the average of 0.38 per cent for the original asphaltenes. Even with 40 weight per cent distillate recovery, as in experiment 13, no new asphaltenes are produced during the distillation.

Table 7 shows that, with 53.5 weight per cent distillate recovery, as in experiment 16, almost all of the original asphaltenes have been converted to varsol-insoluble material, and that the 4.9 weight per cent of newly formed asphaltenes have an ash content of 0.022 per cent. The varsol-soluble portion of the residue or filtrate from experiment 16 yields only about 60 p.p.m. of ash (Table 5), and should be better than the varsol extract of experiment 13 for making good grades of metallurgical coke and electrode carbon (Experiment 16 is similar to 31.).

BY-PRODUCTS

As already indicated, diluent extraction may be used on the entire distillation residue or on the filtrate. Extraction of the latter would be preferable if the diluent-insoluble material proved valuable as a source of vanadium or nickel or for other purposes, as it contains no mineral

TABLE 7. ASH CONTENT AT 725°C OF NEW ASPHALTENES

(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
		Insolubles in residue at end of Expt. 16			Total ash contained by Insolubles			Ratio, col. 8:col. 4
	(gm)	(Wt.%, based on oil charge, pure basis)	(as % of original asphaltenes)	Ash content (%)	Wt.% (based on oil charge, pure basis)	Wt.% (based on original asphaltenes)	(as % of total ash in oil charge, pure basis)	
Coke in residue	12.6	2.6	14	1.0*	0.026	0.145	38	2.7:1
Tl in filtrate	51	10.6	59	0.340	0.036	0.199	52	0.9:1
Vl in toluene extract	21	4.4	24	0.182	0.008	0.043	11	0.5:1
Asphaltenes in varsol extract	26	5.4	30	0.022	0.001†	0.007	2	0.1:1
Total	**110.6**	**23.0**	**127**		**0.071**	**0.394**	**103‡**	
Original asphaltenes	87	18.1	100	0.38	0.069*	0.38	100	1:1
New asphaltenes (by diff.)	23.6	4.9	27	0.022	0.001†			

Tl—Toluene-insoluble material.
Vl—Varsol-insoluble material.
* —This value based on figure 3.
† —Represents 60 p.p.m. of ash in varsol extract (see Table 5).
‡ —Apparent increase probably due to the approximation of 1.0 per cent for the ash content of the coke.

matter or coke. Whether or not the residue should be filtered before extraction by diluent depends on economics. Prior extraction with toluene or similar solvent is not needed.

SUMMARY

(1) Thermal cracking of Athabasca oil-sands oil produces coke, which becomes associated with the mineral matter in the oil and permits filtration of the residues, the filter cake retaining all mineral matter. It is probable that the mineral-matter particles serve as nuclei in the coke formation. The filtrate may be satisfactory for the production of metallurgical coke and bituminous paints.

(2) The residue from the thermal cracking, or the filtrate itself, may be extracted with one or more volumes, or even somewhat less than one volume, of diluent. When 40 weight per cent of distillate has been removed during distillation, the diluent extract yields only about 110 p.p.m. of ash on ignition even though 40 weight per cent of the original asphaltenes are still present. When 53.5 weight per cent of distillate has been removed, the diluent extract yields about 60 p.p.m. of ash on ignition. With respect to ash and metals contents, these extracts are very suitable for the manufacture of good grades of metallurgical coke and electrode carbon, for use in bituminous paints, or for any other uses in which a low ash content is a prerequisite or desirable. Both the filtration and extraction represent simple, controlled, partial-deasphalting operations.

(3) If a pure grade of diluent-insoluble material proves to be economically valuable, it can be obtained readily by extracting the filtrate with diluent rather than extracting the entire residue.

(4) The proportion of coke produced increases with the duration of distillation rather than with distillate recovery, and the linear relationships are markedly different for the comparatively rapid and slow distillations. The ratio of coke produced to ash reduction in the filtrate appears to be approximately 85:1 until over 40 weight per cent of distillate has been recovered; beyond this point the ratio increases.

(5) The Ramsbottom carbon residues of the distillation residues increase with distillate removal, the slow distillations producing substantially more carbon residue material than do the comparatively rapid distillations.

(6) During slow distillations, asphaltenes appear to be converted at a slow rate into diluent-insoluble material, then into toluene-insoluble material, and then into coke. During comparatively rapid distillations, increasing amounts of diluent-insoluble material are produced from the asphaltenes, with very minor amounts subsequently converted to toluene-insoluble material and coke.

(7) It appears that the original asphaltenes in Athabasca oil-sands oil are homogeneous only with respect to their content of difference-resins which yield a trace of ash on ignition. The balance of the original asphaltenes appears to be associated with varying amounts of metals which furnish the bulk of the ash contents on ignition. During the distillations it appears that the asphaltenes which are associated with the highest percentages of metals are the ones which change over most quickly to diluent-insoluble material, then to toluene-insoluble material, and finally to coke. This results in an increase during the distillation in the ratio of coke produced to ash reduction.

(8) No new asphaltenes are produced during a slow distillation until more than 40 weight per cent of distillate has been recovered. These new asphaltenes have very low ash contents. When produced below a liquid temperature of 800°F, they have the same difference-resins content as the original asphaltenes, i.e. 33 weight per cent. However, when the maximum liquid temperature is about 850°F or higher, the content of difference-resins rises to 70 weight per cent and, in addition, the asphaltene content of the diluent extract rises to 55 weight per cent.

(9) After removal of 40 weight per cent of distillate during slow thermal cracking of the Athabasca oil-sands oil, the residue may be filtered and the filtrate coked. The yield of coke, based on the oil charge pure basis, amounts to approximately 15.0 weight per cent, and the coke has an ash content of 0.38 per cent. Or, the residue (or filtrate) may be extracted with diluent and the extract coked. In that case the yield of coke, based on the oil charge pure basis, amounts to approximately 7.2 weight per cent, and the coke has an ash content of about 0.07 per cent.

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OUTLOOK FOR THE ALBERTA SULPHUR INDUSTRY

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ABSTRACT

The Athabasca oil has a sulphur content of approximately 5 per cent by weight. Thus the reserve contains of the order of one billion long tons of sulphur.

Currently four oil-sand development projects have been proposed which would produce up to 1,600 long tons of sulphur per day. Rather than being a boon to a sulphur-starved world as it might heve been at one time, this sulphur could contribute to oversupply and thus depress the industry.

INTRODUCTION

The task assigned to the writer was to comment upon the outlook for the Alberta sulphur industry with particular reference to the significance of sulphur production from the Athabasca oil sands.

The oil contained in the sands has a sulphur content of approximately 5 per cent by weight, and reserves of the order of one billion long tons of sulphur are estimated to be contained in this deposit. This quantity of sulphur is enough to supply double the free world's annual consumption for the next five decades. By comparison sulphur reserves of the sour gas fields of Alberta, which total 80 million long tons, although substantial, are overshadowed by the oil sands deposit.

However, the reserve figures in themselves are simply numerical curiosities since it is the quantity and timing of actual production which is important.

There was a time when as much attention might have been paid to the sulphur produced in association with the extraction of crude petroleum from the Athabasca oil sands as to the primary development itself. However, the sulphur situation has proved to be dynamic and marked changes in the world situation have taken place in recent years. Before elaborating on the current or future sulphur situation, a review of events which have led up to the present situation is a necessary prologue. This perhaps will provide the proper atmosphere of change and uncertainty which is so much a part of the situation at the moment.

SULPHUR PRODUCTION IN ALBERTA

Sulphur has been produced in Alberta for more than a decade, but it is only in recent years that the quantity produced has assumed really significant proportions. Alberta sulphur is produced from hydrogen sulphide which must be removed from sour natural gas in order to make the gas suitable for market. Other products such

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as propane, butane, condensate, and so on, are removed from the gas as well. It is the recent increase in gas sales which has resulted in the upsurge in sulphur production.

Recognition of the fact that hydrogen sulphide gas was available in Alberta in commercial quantities is not a recent development. In the immediate post-war period the Research Council of Alberta studied the possibility of the establishment of sulphur-extraction facilities in the province. Hydrogen sulphide gas was being flared from the Turner Valley field and attention was focused on converting this to elemental sulphur. However, the size of the local market was very small relative to the size of an economical sulphur-recovery unit, and transportation charges precluded moving Alberta sulphur to more distant market areas. Who could have foreseen at that time that in spite of these factors, natural gas development would make Alberta a major sulphur producer?

The first sulphur recovery unit was established at the Jumping Pound field in 1952. Production in that first year of operation amounted to approximately 8,000 long tons. The increase in the volume of sulphur produced in the province over the period of the next few years was modest (Table 1).

TABLE 1. SULPHUR PRODUCTION IN ALBERTA, 1952-1962

Year	Production (long tons)	Sales (long tons)	Sales as Percentage of Production	Inventory* (long tons)
1952	7,974	3,772	47.3	2,832
1953	16,338	14,350	87.8	5,240
1954	19,929	16,665	83.6	7,271
1955	25,976	23,193	89.3	10,183
1956	29,879	31,057	103.9	8,605
1957	89,915	83,328	92.7	15,561
1958	109,220	81,726	74.8	39,628
1959	213,075	86,285	40.5	164,856
1960	348,147	208,486	59.9	300,239
1961	434,396	302,748	69.7	430,120
1962	972,351	560,883	57.7	839,142

*Figures do not necessarily balance with production and sales due to losses and adjustments

Source: Alberta Oil and Gas Conservation Board

By 1961, Alberta sulphur production amounted to just over 400,000 long tons. This year (1963) sulphur recovered from gas processing operations should total approximately 1,200,000 long tons, an increase of approximately 800,000 long tons within 2 years.

TABLE 2. ACTUAL AND ESTIMATED PRODUCTION OF SULPHUR IN ALBERTA, 1960-1972

Gas-processing plants		H_2S in raw gas (%)	Capacity (long tons per day)	Cumulative to Dec. 31, 1959	Annual Production (thousands of long tons)								
					Actual			Estimated					
					1960	1961	1962	1963	1964	1965	1966	1967	1972
Producing plants													
Operating company	Location												
British American Oil Co. Ltd.	Nevis	6	76	—	11.8	15.2	18.5	19	19	19	19	19	19
British American Oil Co. Ltd.	Pincher Creek	11	675	242.2	156.5	162.9	173.2	175	180	190	200	200	200
British American Oil Co. Ltd.	Rimbey	2	250	—	—	36.0	63.9	72	76	80	80	80	80
California Standard Company	Nevis	6	120	1.4	20.8	29.6	37.3	40	40	40	40	40	40
Canadian Fina Oil Ltd.	Wildcat Hills	4	105	—	—	—	21.7	24	25	27	30	30	30
Canadian Oil Companies Ltd.	Innisfail	14	100	—	2.9	30.2	33.7	34	35	35	35	35	35
Home Oil Company Limited	Carstairs	1	40	—	—	0.1	5.7	6	7	8	9	9	9
Imperial Oil Limited	Redwater	3	9	5.6	1.4	2.1	2.2	2	2	2	2	2	2
Jefferson Lake Petrochemicals Ltd.	Coleman	14	377	—	—	—	28.3	40	45	50	50	50	50
Pan American Petroleum Corporation	Windfall	16	650	—	—	—	127.2	190	220	220	230	275	460
Petrogas Processing Ltd.	Calgary	16	863	—	—	2.9	170.2	190	210	220	225	225	230
Royalite Oil Company Limited	Turner Valley	2	30	70.0	8.2	7.7	7.8	8	8	8	7	7	5
Shell Canada Ltd.	Jumping Pound	4	100	140.6	29.2	26.8	28.3	28	28	28	27	26	20
Shell Canada Ltd.	Waterton	25	1,400	—	—	—	134.3	190	235	300	400	400	400
Texas Gulf and Sulphur Company	Okotoks	35	370	52.5	117.4	121.0	120.0	125	125	125	125	125	120
			5,165	512.3	348.2	434.5	972.3	1,143	1,255	1,352	1,479	1,523	1,700
Probable future plants													
Major operator in area	Field or area												
British American Oil Co. Ltd.	Berland River	15	250					—	20	85	195	370	500
British American Oil Co. Ltd.	Lookout Butte	3	45										
British American Oil Co. Ltd.	Wimborne	15	110										
California Standard Company	Moose Mountain	13	25										
Imperial Oil Limited	Waiparous Creek	5	10										
Olds Gas Ltd.	Olds	6	40										
Pan American Petroleum Corporation	Bigstone	12	25										
Pan American Petroleum Corporation	Crossfield East	38	450										
Shell Canada, Ltd.	Burnt Timber	6	40										
Shell Canada, Ltd.	Panther River	87	380										
Shell Canada, Ltd.	Simonette	17	175										
Texas Gulf Sulphur Company	Hunter Valley	10	50										
Texas Gulf Sulphur Company	Wildhorse Creek	6	30										
			1,630	—	—	—	—	—	20	85	195	370	500
TOTALS			6,795	512.3	348.2	434.5	972.3	1,143	1,275	1,437	1,674	1,893	2,200

April, 1963.
Based on existing gas contracts and proven gas reserves not yet under contract.

This soaring increase in production is the direct result of large quantities of natural gas being exported from Alberta. The new area being served by Alberta gas is the West Coast region of the United States supplied by the Alberta California Pipeline. To meet the export requirements, sour-gas fields were tapped with the consequential upsurge in sulphur production.

At present there are fifteen sulphur-recovery plants operating in the province with a daily productive capacity of 5,165 long tons. Plants contemplated for construction in the next few years should boost this productive capacity to 6,800 long tons per day (Table 2).

MARKETS FOR ALBERTA SULPHUR

Very few countries of the world are major sulphur producers. Therefore this nation is joining a rather select group by its sudden increase in sulphur production. The magnitude of this development may be seen in its proper perspective by examining the volume of sulphur produced by the major Free World producers. In 1962 the major Free World sulphur producers in order of importance were: the United States, Mexico and France, with production of 6,000,000 long tons, 1,400,000 long tons, and 1,300,000 long tons, respectively.

Canada's sulphur output this year will be of the order of 2,000,000 long tons, with approximately 70 per cent of the total being produced in Alberta. Thus Canadian sulphur will be second to the United States in terms of total output.

The sudden increase in sulphur production in Alberta has attracted world-wide attention. Both the sulphur producers who will be competing with Alberta sulphur for markets and the possible customers who are viewing Alberta as a source of supply have been interested in this development.

Canada's total requirements of sulphur only amount to approximately 500,000 long tons per year, and a significant proportion of this consumption is located beyond the market area which may be

TABLE 3. ESTIMATED MAGNITUDE OF POSSIBLE MARKETS FOR ALBERTA SULPHUR, 1965

Area	Long tons
Korea, Indonesia, Taiwan, etc.	100,000
New Zealand	180,000
Union of South Africa	200,000
Australia	270,000
U.S.—Northwest States and California	338,000
India	400,000
U.S.—North Central States	872,000

Source: Author's estimate

economically served by Alberta sulphur. Therefore, a large portion of the sulphur produced in Alberta will have to find markets outside of this country. Table 3 shows the magnitude of the markets most likely to be served by Alberta sulphur. The figures are shown for the year 1965, the year in which Alberta sulphur production will reach approximately 1,500,000 long tons on the basis of existing gas contracts. It will be noted that besides the United States, the markets which Alberta sulphur will most likely penetrate are the countries which will be reached via the Port of Vancouver, such as South Africa, India, Korea, Japan, Australia and New Zealand.

Although the consumption of the countries which are likely to be served by Alberta sulphur is sizeable and growing steadily, the competition for these markets is keen. The United States producers, besides being accustomed to supplying their own needs and the needs of most of the major users of the world, have grudgingly had to share these markets with production from Mexico, France and now Canada.

The situation is exactly opposite to the one which prevailed a decade ago. At that time a severe sulphur shortage occurred primarily as a result of the Korean War. Sulphur became a strategic material and the United States, the principal world supplier at that time, had to restrict its sulphur sales. Sulphur users naturally began looking for alternate sources of supply.

Alberta was considered as a source of supply of sulphur and the the Alberta sulphur-recovery industry as presently constituted really had its beginings as a result of this sulphur shortage. However, other sources of sulphur in Alberta were examined as well.

When the term "Frasch" is used, most people with a knowledge of the sulphur industry immediately think of the process used to mine the sulphur deposits located on the Gulf Coast of Texas. The United States material is obtained from underground deposits using a hot-water and compressed-air method developed in the early 1900s by an inventor by the name of Herman Frasch.

This same process was considered for use in Alberta in the early 1950s when the shortage created sudden interest in underground deposits of sulphur located 90 miles northeast of Edmonton. These deposits had been discovered through oil exploration. However, ultimate investigation revealed that the reserves did not warrant this type of development. At that time quite a flurry of excitement was caused by the announced plans.

British sulphur users were particularly hard hit by the sulphur shortage and they became seriously interested in the sulphur of the Athabasca oil sands as a possible source of supply for this material.

Offers were made to establish sulphur-recovery facilities as an adjunct to oil-separation installations. However, in 1951 and 1952, conventional oil production in Alberta was being developed rapidly and the need for utilizing the petroleum reserves of the Athabasca oil sands diminished. From the point of view of sulphur production alone from the Athabasca oil sands in the early 1950s would have been most advantageous since sulphur prices were high and the material was in demand.

As a direct result of this sulphur shortage in the early 1950s sulphur productive capacity of the United States and Mexico was increased substantially. This increase in production came into full capacity by the mid-1950s. There followed an unexpected sour-gas discovery in France which gave rise to sizeable recovery sulphur production there in 1959. Even before the French production reached its peak, output of sulphur in Alberta began to soar and contribute to what had become an oversupply situation.

Thus large quantities of recovery sulphur, produced as they are in association with natural gas which is sold under long-term contract, are adding to the world supply of sulphur despite the unfavourable immediate market outlook. This is in fact a rather peculiar aspect of the recovery sulphur industry in that output is forthcoming despite market conditions.

Up until the end of 1962, only modest sales of Alberta sulphur have been made in foreign markets. Sulphur producers have been concerned primarily with getting their facilities into production. Since storage of sulphur represents no difficulty (this material is simply dyked and allowed to solidify with little or no deterioration taking place), marketing on the whole had not been pushed too hard.

Sulphur stocks in Alberta now total approximately 1,000,000 long tons and with production rising, more attention is being paid to marketing. The year 1963 marks the first time that Alberta sulphur producers have made a concerted effort to enter markets. To the end of June approximately 100,000 long tons were sold in overseas markets as compared with 60,000 long tons in the full year of 1962.

Sales have been averaging approximately 65 per cent of production even as production has been rising. Sulphur prices on the other hand have slumped sharply. Since sulphur is an almost homogeneous material, price becomes a very important factor in the marketing of this material. Sulphur is sold on a bid basis, and in order to get a share of new markets, attractive offers have been made. Some producers have made the mistake of bidding too low on initial shipments in an attempt to obtain an order and have the material tried. Subsequently it was planned to raise the price. However, in the face of oversupply, upward revisions of the sulphur price structure have been difficult to achieve.

Improvements in the selling price may be expected as the world demand for sulphur grows and as Alberta sulphur becomes absorbed into world markets. Happily, sulphur consumption increases at an amazingly stable rate of approximately 4 per cent per annum.

However, improvement in the price structure of the industry is really dependent on the rate which sulphur production increases, since sulphur consumption is so stable. Alberta sulphur recovery production on the basis of present gas contracts is shown in table 2. By 1965 Alberta sulphur-recovery production could amount to 1,437,000 long tons. This would appear to be a fairly firm figure since sizeable additional quantities of gas for export are not indicated before 1966. From what limited information is available at the moment, it would appear that sulphur produced from the Athabasca oil sands would come on the scene around the end of the decade.

TABLE 4. ANTICIPATED SULPHUR PRODUCTION ASSOCIATED WITH CRUDE PETROLEUM PRODUCTION—ATHABASCA OIL SANDS 1966-1970

Project	Sulphur Production long tons/day
Great Canadian Oil Sands Ltd.	260
Cities Service Athabasca Inc.	500
Shell Canada Ltd.	500
Can-Amera Oil Sands Development Ltd.	340
TOTAL	1,600

Source: Press reports plus industry contact.

SULPHUR FROM THE OIL SANDS

Currently, four Athabasca oil sands development projects are being discussed. Table 4 indicates the magnitude of sulphur production which would be associated with these plants. Sulphur production from all of these projects will be discussed even though at the moment only one project has been approved by the Alberta Oil and Gas Conservation Board.

If sulphur production from the oil sands does amount to 1,600 long tons per day, or 584,000 long tons per year, this would only amount to approximately one fifth of total Alberta production. The proportion could be as little as one sixth if gas sales were to increase further and there is a good possibility of this. Clearly the amount of sulphur which might be produced from the oil sands is not in itself too significant. What it really amounts to is the amount of sulphur which might be produced from one or two good-sized sulphur-recovery plants.

The timing of the sulphur produced from the Athabasca oil sands could be the significant factor. The sulphur industry at large is looking forward to future growth in sulphur consumption to soak up the excess supply and stabilize the price structure of the industry. Any sizeable amounts of new sulphur coming into production could have a dislocating effect on the industry.

What could happen is that sulphur production from the Athabasca oil sands could forestall the time when the demand for sulphur would catch up to supply. Rather than being a boon to a sulphur starved world as it might have been at one time, it could serve to contribute to oversupply and depress the industry.

TABLE 5. CANADIAN EXPORTS OF ELEMENTAL SULPHUR—BY COUNTRY OF DESTINATION
1961-1962

Country	1961	1962
United Kingdom	—	9,999
Belgium-Luxemburg	—	5,079
India	—	5,474
Malaya	246	886
Pakistan	—	1,481
Indonesia	—	1,823
Korea	—	3,877
Philippines	—	982
Taiwan	14,762	13,674
Australia	1,502	21,437
United States	178,011	292,451
TOTAL (long tons)	194,521	357,163

Source: Dominion Bureau of Statistics—Trade of Canada

PROGRAM

of

SECOND ATHABASCA OIL SANDS CONFERENCE

held in the

Northern Alberta Jubilee Auditorium
Edmonton

on

October 30 and 31, 1963

Wednesday, October 30th, 1963

MORNING

8:00 12:00 noon, Wednesday, October 30, 1963, Registration at the Northern Alberta Jubilee Auditorium, Edmonton

9:00 Opening Address by **A. R. Patrick**, Minister of Industry and Development, Introduced by **E. E. Pelzer**, President, Edmonton Geological Society (Social Room).

SESSION I (Social Room)

Chairman: R. E. Folinsbee, Head, Geology Department, U. of A.

9:30 Evaluation of Oil Sand Reserves—**J. R. Pow, G. H. Fairbanks, W. J. Zamora,** Oil and Gas Conservation Board, Calgary

10:00 Geological Controls on Athabasca Oil Sand Reserves—**G. A. Stewart,** Imperial Oil Ltd., Calgary

10:30 Paleogeomorphology of the Buried Devonian Landscape—**R. Martin** and **F. G. S. Jamin,** Rudolf Martin & Associates Ltd., Calgary

11:00 Petrology of Coarse Sands at the Base of McMurray Formation—**M. A. Carrigy,** Research Council of Alberta, Edmonton

11:30 Arctic Oil Sands Compared With Athabasca Oil Sands—**J. C. Sproule** and **G. V. Lloyd,** J. C. Sproule and Associates Ltd., Calgary

12:00 Lunch

AFTERNOON

SESSION II (Social Room)

Chairman: D. L. Flock, Associate Professor of Petroleum Engineering, U. of A.

1:30 Flow of Formation Fluids and Origin of the Athabasca Oil Sands—**B. Hitchon,** Research Council of Alberta, Edmonton

2:00 Source and Manner of Emplacement of the Bitumen—**W. B. Gallup,** Geological Consultant, Calgary

2:30 Continuity of Bedding Within the McMurray Formation—**R. W. Ansley** and **W. G. Bierlmeier,** Cities Service Athabasca Inc., Edmonton

3:00 Coffee

3:30 Shear Strength of Oil Sand—**R. M. Hardy** and **R. A. Hemstock,** University of Alberta, Edmonton, Imperial Oil Ltd., Calgary

4:00 Use of Bucket-Wheel Excavators—**H. W. Schellhorn,** Fried. Krupp, Essen, West Germany

4:30 Discussion Period.

EVENING SESSION

(Macdonald Hotel)

8:30 Banquet Address and Presentation to **Dr. K. A. Clark** by **S. M. Blair,** President, Canadian Bechtel Limited

Thursday, October 31st, 1963

MORNING

SESSION III (Social Room)

Chairman: H. S. Armstrong, Vice-President, University of Alberta

9:30 Oil Sand Separation Methods—**K. A. Clark**, Consultant to the Research Council of Alberta, Edmonton

10:00 The Sand Reduction Method of Extraction—**J. A. Bichard, C. W. Bowman, R. M. Butler,** and **J. L. Tiedje**, Imperial Oil Enterprises Ltd., Sarnia

10:30 Use of Sound Waves in Oil Sand Separation—**T. J. Bulat**, The Bendix Corporation, Davenport, Iowa

11:00 An Anhydrous Method of Extraction—**J. H. Cottrell**, Cities Service Athabasca Inc., Edmonton

11:30 Steam Drive an In-situ Thermal Recovery Process—**T. M. Doscher, R. W. Labelle, L. H. Sawatsky,** and **R. W. Zwicky**, Shell Canada Limited, Edmonton

12:00 Lunch

AFTERNOON

SESSION IV (Social Room)

Chairman: R. A. Given, Technical Superintendent, Cities Service Athabasca Inc.

1:30 Production of Low-Ash Asphalt and Coke—**D. S. Pasternack**, Research Council of Alberta, Edmonton

2:00 Composition of Bitumen Fractions Determined by Structural-Group Analysis—**M. L. Boyd** and **D. S. Montgomery**, Dept. of Mines and Technical Surveys, Ottawa

2:30 Significance of Porphyrin Pigments in Oil Sands—**G. W. Hodgson, E. Peake, B. Baker**, Research Council of Alberta, Edmonton

3:00 Coffee

3:30 Outlook for the Alberta Sulphur Industry—**W. G. Brese**, Department of Industry and Development, Edmonton

4:00 Project Oilsand (a narrated strip film on use of nuclear energy—**M. L. Natland**, Richfield Oil Corporation, Los Angeles

4:30 Discussion Period