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OIL SHALES OF COLORADO

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BY.
R. D. GEORGE

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LETTER OF TRANSMITTAL

STATE GEOLOGICAL SURVEY,
UNIVERSITY OF COLORADO, November 30, 1920.

Governor Oliver H. Shoup, Chairman, and Members of the Advisory Board of the State Geological Survey.

GENTLEMEN: I have the honor to transmit herewith Bulletin 25 of the Colorado Geological Survey.

Very respectfully,

R. D. GEORGE,
State Geologist.

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ILLUSTRATIONS

PREFACE

The bulletin here presented is not offered as the result of a comprehensive and systematic study of the problems of oil shale, but rather as a selection from a large body of facts and results which have accumulated from somewhat disconnected, and at times incomplete investigations in field and laboratory work extending over a period of fourteen years. Several portions of the material here brought together have been used in addresses and in articles in various magazines and other publications.

The writer began his study of the shales in 1906, when he followed the southern outcrops of the shale beds from near Tucker, Utah, to Rifle, Colorado, collected samples of the shales and made a series of tests of the shales as possible future sources of oil, and as material for road building. After these first studies, except for a few tests of material sent to the Colorado Geological Survey laboratory, very little was done until 1915, when, at the request of certain officers of the United States Navy, the study was resumed in a more systematic and purposeful manner. A laboratory was fitted up with the essential apparatus for retorting, and the distillation tests were made in the University laboratories. But the pressure of other Survey work, especially during the war, tended to force the shale problems into the background.

In compiling the second part of the report the writer has selected what he considered most valuable from the results of his own laboratory work and from those of advanced students and instructors working under his direction. It has been found impossible, except in a few instances, to make specific acknowledgments of the contributions. Those whose work has proved most valuable are: C. F. Poe, now Assistant Professor in the Department of Chemistry; Q. R. Dungan, of the Empire Gas and Fuel Co., and M. J. Wilson. The last two prepared theses for the Master's degree under the writer's direction.

Individuals and companies for whom the writer has made investigations have kindly given him permission to use the results of those studies as he desires. The Survey is indebted to the Empire Gas and Fuel Company for the use of its map of the southern part of the shale area. This is reproduced as Plate II.

Descriptions of the Scotch retorts, and the Scotch processes of retorting and refining have appeared in so many different publications that it seems unnecessary to give space to them in this Bulletin. As to the costs of installation and operation, the unsettled industrial conditions and the rapidly fluctuating prices of materials render any statement little better than guesswork. In pre-war times the cost of a retorting plant having a capacity of 1,000 tons per day, and a refining plant to take proper care of the crude obtained, and provided with adequate storage facilities, trackage and other necessary equipment, was placed by various responsible engineering firms at about $2,500,000.00. This provided only for mining, crushing, tramming and retorting the shales; condensing and refining the oils into motor, burning and lubricating oils; separating and refining the wax; separating and refining the ammonia. It did not provide for the saving of any of the many possible by-products which may or may not prove commercially profitable. It did not provide for such marketing facilities as tank cars and other equipment.

In Colorado the securing of a water supply may add considerably to the initial outlay necessary.

R. D. GEORGE.

OIL SHALES OF COLORADO

INTRODUCTION

Before the discovery of the vast oil resources of Pennsylvania, and their development in the early sixties, there were over fifty companies in the United States producing oil by the distillation of coals and shales. But the rapid development of the Pennsylvania field forced the producers of shale oil to the wall. After Pennsylvania came Ohio, West Virginia and California, in the seventies, and a host of others in the eighties. The possibility of oil from shale was forgotten in this country. But in Scotland, France and other countries not provided with petroleum, the distillation of shales went on, methods were improved, the operation costs cut, and the output increased.

Pennsylvania reached her maximum production in the nineties, and is now pumping less than one-third as much as she did in 1891. Ohio began to decline in the nineties and is now producing only one-third of her greatest yields. West Virginia, through the extension of her fields and much new drilling, has made a hard fight, but is falling off. Colorado, Indiana, Illinois and Kansas reached their high oil mark about 1916. New states have become producers, and Texas, California and one or two others have increased their output. But the fact remains that in proportion to the wells drilled the production of the United States is declining.

When the ever-increasing demand is taken into consideration, it is evident that the time is not far distant when a part of the supply will again come from the distillation of shales. When that time comes Colorado will unquestionably take a prominent place.

THE OIL SHALE MAP OF PART OF GARFIELD COUNTY[1]

PRIMARY CONTROL

A base line was taken from the DeBeque, Colo., topographic sheet and extended from the summit of Red Pinnacle in Sec. 16-7-97 to the summit of Mt. Logan in Sec. 35-7-97. From this base line a triangulation net was carried over the whole area by means of a plane table and telescopic alidade. Prominent, well-defined natural objects were used as stations in some cases, but nearly all of the stations were pine or aspen poles braced at the base.

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[1] By R. L. Heaton, Geologist, Empire Gas and Fuel Company.

MAPPING

All mapping was done by plane table and open-sight alidade, using the triangulation stations for control.

Besides occupying the primary stations, the geologists located themselves at intermediate points by resection and intersection, and sketched the geology between these points. Unoccupied points were also located by intersection and used in the same manner.

The two roughly parallel lines shown on this map represent the upper and lower ledges of richer shale. They are designated as (A) and (B) on the map legend, as well as on the photographs. A center line representing the steep white cliff which occurs so prominently over all of this area was shown on the original map, but was omitted from this one because it contains no beds of commercial shale.

Fig. 1 shows the general relationship of the beds containing the richer shale. The ledges represented at (A) vary greatly throughout the area in thickness and oil content. They are composed almost entirely of the massive variety of shale, and the thickness and number of separate beds mapped as the upper group (A) increase from the western to the eastern part of the area. Fig 2 shows the characteristic weathering of these ledges on Mt. Callahan, where there are five beds varying in thickness from 3 to 10 feet, and in oil content from 25 to 45 gallons per ton.

Ledge (B), as seen in Fig. 1, is about 50 feet thick and is easily recognizable throughout the area. About the middle of this cliff there occurs the bed of rich paper shale varying in thickness from 4½ feet in places on Carr and Clear creeks to 9 feet 8 inches on Mt. Callahan at the mouth of Parachute creek. Samples from this bed ranged from 35 to 85 gallons to the ton, using a field retort, and the average for the whole area was 54 gallons to the ton. The letter (B) on Fig. 3 is at the center of this bed at a locality on Carr creek where it is 6 feet thick and runs approximately 60 gallons to the ton. Fig. 4 shows the same stratum near Newton's trail on Clear creek, where the characteristic paper weathering has given way to a more massive appearance by reason of its being partly protected from the weather by the cliff of leaner shales above.

This bed has by far the best commercial possibilities of any in the field. It is being mined at Mt. Logan. There is a similar bed a few feet lower in ledge B, which is from 3½ to 4½ feet thick and runs about the same in oil content.

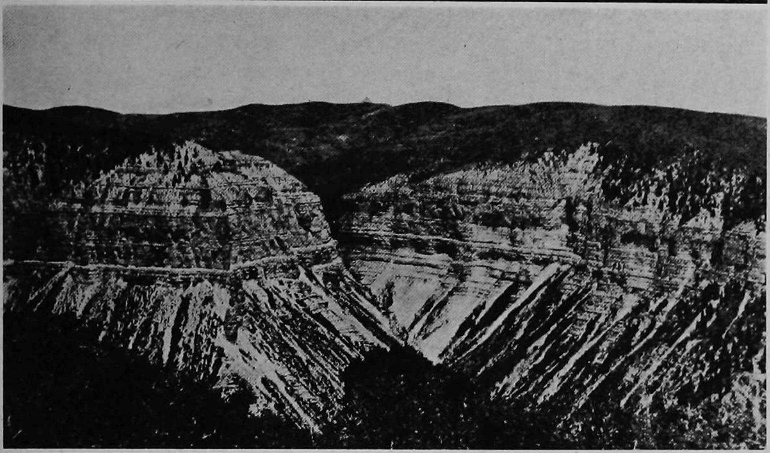

Fig. 1. View showing upper and lower ledges of rich shale (A and B). Sec. 33, T. 5 S., R. 98 W., Clear Creek.

Fig. 2. Upper shale ledge (A) showing typical massive shale. Sec. 16, T. 7 S., R. 96 W., Mount Callahan.

Minor drainage was sketched. Points were located along the main streams and the water courses sketched between them as in the geologic mapping. All roads were shown in like manner. Trails were sketched in a general way only. The showing of ranch houses was not considered important enough to warrant the time necessary in locating them.

LAND SURVEY

It was borne in mind that the value of this map would depend upon the accuracy of the geological mapping, and upon the showing of all areas of shale in the proper section, according to the existent land survey.

This plan was carried out as far as possible by locating on the map, by triangulation methods, all land survey marks that could be found and checking them by reference to copies of the land plats from the U. S. Land Office at Glenwood Springs. Where section and township corners could be checked in this way, results were very satisfactory. In case no government marks were present, the land survey was taken directly from the land plats and checked, wherever possible, with marks left by private surveys which were run from official government marks.

Twp. 5 S., Ranges 95 and 96 W. and Twp. 6 S., Ranges 96 and 97 W. have been recently surveyed, and corners are plentiful. According to this survey the off-set along the First Correction Line South is about three miles, instead of about one-third of a mile, as shown farther west by the old survey. Both are correct, and this necessitates the cutting out of a part of Twp. 5 S., R. 98 W. by Twp. 5 S., R. 96 W. of the recent survey. Many maps erroneously continue this off-set of three miles, on westward to the Utah line.

The drainage, roads and land survey of Twp. 8 S., Ranges 97 and 98 W. were taken from the DeBeque topographic sheet. For Twp. 8 S., R. 96 W. these were taken from the land office plat.

ELEVATIONS

The elevations shown on this map were taken, for the most part, by aneroid. Some of them, in the western part of the field, were taken from Hayden's Atlas.

GEOGRAPHY

LOCATION

The Green River shales of northwestern Colorado occupy an area which will aggregate approximately 2,000 square miles in Garfield, Rio Blanco and Mesa counties. There is also a considerable area in Moffat County, but of this less is known.

TOPOGRAPHY

The topographic features of the area are those of a broad, northward-sloping dissected plateau, from which Grand and Battlement mesas have been detached by erosion. Along the broader valleys the shaly parts of the formations have been carved into steep, pointed hills characteristic of the badlands. In places the plateau area is similarly eroded. The lesser valleys are canyon-like, and even the outer margin of the main valleys presents abrupt faces wherever sandstones form any considerable portion of the strata. On the southern border of the main plateau area, facing the Grand River, are the Book Cliffs which rise to a height of 2,000 to 3,000 feet above the valley floor. On the east side are the Petrolite and Gray hills, and on the west are the Cathedral Bluffs. In many places the Book Cliffs present very steep slopes and vertical walls which cannot be scaled, though at numerous points trails lead to the pasture lands above.

DRAINAGE

The Grand River flows along the southern border of the main shale area, but cuts off from this considerable volumes of shale in Battlement and Grand mesas. The smaller streams tributary to the Grand are Rifle, Parachute, Roan, Kimball and East and West Salt creeks. Cutting through the northern part of the area is the White River, with its tributaries, Flag, Sheep, Piceance, Yellow and Douglas creeks, on the south. The Yampa River, with its broad valley merging into that of the White, cuts off the shale area of Moffat County from that of Rio Blanco to the south. The Snake River crosses the Moffat County area.

The rivers and their larger tributaries are perennial, but many of the streams are intermittent. The level parts of the valleys are occupied by irrigated ranches wherever the water supply is sufficient. The waters of the scantier streams become strongly alkaline as their flow decreases.

CLIMATE

The total annual precipitation of the region is between 12 and 14 inches, of which a considerable portion comes in the spring and early summer months. On the high plateau area the winter temperatures are low, but the dry atmosphere is bracing and invigorating, and conditions are rarely such as to prevent the pursuit of outdoor occupations. There is nothing in the strictly climatic conditions which would interfere with all-year operation of shale oil plants.

VEGETATION

The vegetation is sparse. The trees include mainly sage brush, pine, cedar and aspens. Grass is scarce, except on the more favored parts of the flat plateaux and mesas, where it is fairly abundant and very nutritious. In many parts of the shale area there is sufficient timber to meet all ordinary operation requirements.

All kinds of grains, maize and vegetables grow abundantly. The fruits of this part of the state are noted in all markets. The natural rainfall, especially on the table lands, is such that a total failure of crops is rare, and frequently unexpectedly large yields are obtained.

TOWNS

The principal towns of the area immediately tributary to the shale deposits are: Rifle, Grand Valley, DeBeque and Meeker. Glenwood Springs is on the railway to the east, and Palisade and Grand Junction to the south and west. In the northern part of the area, Rangely is a small trading point. Rio Blanco, Highmore, Sulphur and Piceance are postoffices within the shale area.

INDUSTRIES

The principal industries of this part of Colorado are ranching, cattle raising, fruit growing and coal mining. Coal mining is an important industry, and the coal produced is of excellent quality and could be secured at a reasonable price for power purposes in case it should prove desirable to supplement the fuel products of the oil shale industry by the use of coal.

CULTURAL FEATURES

The southern part of the area is served by the Denver and Rio Grande Railroad, Uinta and Book Cliffs railways. To the north the Denver and Salt Lake is completed as far as Craig, the county seat of Moffat County. Surveys have been completed for the extension of the road to Salt Lake City. The Denver and Rio Grande Railroad follows the Grand River from Glenwood Springs to the western border of the state. The Uinta Railway leaves the Denver and Rio Grande at the town of Mack, near the western border of the state, and takes a north or northwesterly course to Watson and Dragon, in Utah. The Book Cliffs Railroad is a short line from Grand Junction to Carpenter, about ten miles north of the river.

A well-built state highway follows the Grand River from Glenwood Springs to DeBeque. At DeBeque it makes a considerable detour to the south, but returns to the river valley at Almond. Another state highway runs from Rifle northward to the valley of the White River, where it joins the state highway, which follows the White River to and beyond the Utah boundary. Good highways follow a few of the larger stream valleys. Fair roads are found in others, and mere trails afford access to many parts of the shale territory. Steep trails, rarely suited to the use of vehicles, lead from the valleys to the high plateau areas.

GEOLOGY

STRUCTURE AND FORMATIONS

In the Rocky Mountain region the mountain-making movements at the close of Cretaceous and the opening of Tertiary times caused the formation of a great basin-like depression of roughly rectangular outline, occupying the northwest corner of Colorado, the southwest quarter of Wyoming, and the northeast corner of Utah. This depression was roughly 300 miles from north to south, and 200 miles from east to west. The drainage from the surrounding country poured into this great basin, forming a lake having an area nearly double that of Lake Superior.

The Uinta Mountain fold almost completely divided the southern one-third of the lake from the northern two-thirds, but the same conditions of deposition prevailed north and south of the Ridge. At the present time the area south of the Uinta Mountains is known as the Uinta Basin, while that north of the Range is divided into the Green River Basin and the Red Desert Basin. The streams flowing into the lake carried with them vast quantities of sediment from the surrounding country, and the waves spread this material rather uniformly over the floor of the lake, building from it four great Eocene rock series—the Wasatch, the Green River, the Bridger and the Uinta—having a total thickness of several thousand feet.

In the course of time the gradual filling of the basin with sediments and the deepening of the valley of the Green River caused the disappearance of the lake and the emergence of a broad, slightly undulating expanse of soft sedimentary rocks consisting mainly of shales with a relatively small percentage of sandstone and a still smaller proportion of limestone. On this new surface the Green, the White, the Yampa, the Duchesne, and the Grand rivers and their tributaries immediately began the work of carving out the present topography, the main features of which consist of broad mesas or plateaus deeply cut by canyons and bordered by steep slopes and cliffs. The most striking topographic feature of today is a great plateau from 8,000 to 9,000 feet above sea level, facing the Grand River and sloping gently northward. South of the river, Grand and Battlement mesas are but remnants of a more ancient plateau, separated from its northern neighbor by the valley of the Grand River. Most of the strata of these great Tertiary formations still lie in horizontal position, but here and there, as against the southern slope of the Uinta Mountains, they are tilted at angles varying from a few degrees to as high as twenty degrees.

Of these four great rock series, the second from the bottom, the Green River, is the one of special interest at the present time on account of the oil shales which occur mainly in the upper half of the formation.

The oil shales occur in bands and strata ranging in thickness from a few inches to 75 or 80 feet. In many instances they are sharply set off from the underlying and overlying strata, while in others there is a gradual transition from rock practically free

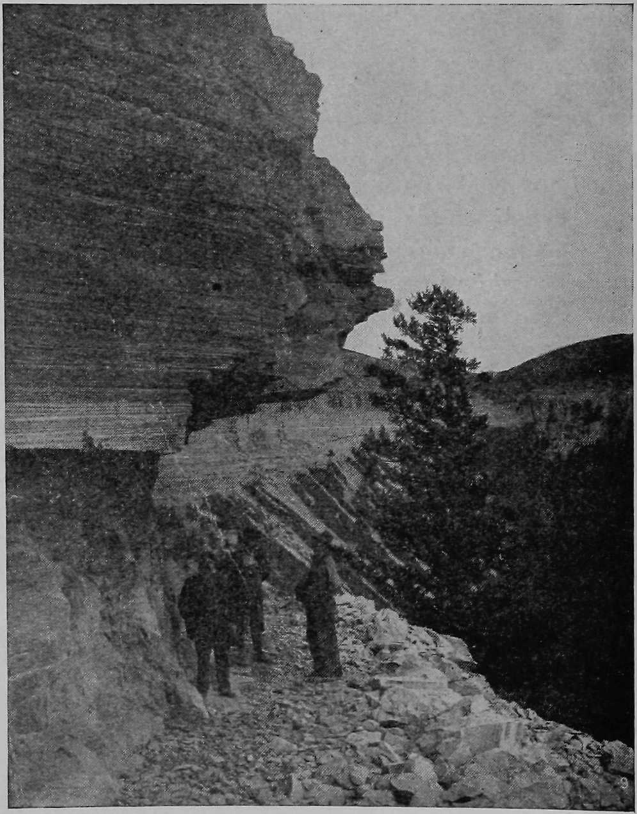

Fig. 3. Close view of oil shale deposits in northwestern Colorado (near Grand Valley) having a thickness of over 500 feet. Oil content varies from 35 to 90 gallons to ton of shale.—Courtesy Denver & Rio Grande Railroad.

from bituminous matter to layers which will yield as high as 60 to 80 gallons of oil per ton, and from this again to the almost barren rock above. Within the thicker oil shale bands the oil content varies widely. Thin layers may be selected which will yield as high as 90 gallons per ton, and strata from 5 to 10 feet in thickness may be found which will average from 60 to 70 gallons per ton. Between these will be leaner strata ranging down to barrenness.

The oil shale differs widely in color, composition, specific gravity, hardness and brittleness.

The lean shales are commonly of lighter color, ranging from whitish gray to buff, and sometimes blue gray.

As a rule, the lighter the color of the shale the greater is the proportion of very fine sand, while the darker the shale the greater the proportion of clay substance and organic remains. The richest shales range in color from a deep brownish mahogany to almost jet black on fresh surfaces.

A very little experience with shales enables one to select the rich and discard the lean by the weight of the material alone. The leaner material has nearly the usual specific gravity of common shales, 2.4 to 2.7, while the richest ranges from 1.6 to 1.85 (water 1.0).

Under the hammer the leaner strata behave as do ordinary shales. The more siliceous rock breaks with a smooth conchoidal fracture and is very brittle, while the richest is exceedingly tough and hard to break, especially in the direction of its structure. Across the bedding it breaks with a smooth conchoidal fracture, frequently exposing a delicate color banding of black and brownish black. The fresh fracture surfaces of the rich shale are silky or satiny in appearance. If such surfaces are scratched with a pointed instrument the mark left has a waxy surface, and the powder formed along the scratch resembles a grayish wax, cohering in masses and not falling apart in fine grains. The stroke of the hammer on the rich shale yields a sound similar to that produced when a hard, heavy wood is struck. The leaner shales give out the usual sounds when struck.

The rich shales withstand weathering much better than do the lean shales and non-bituminous rock, and, as a consequence, the edges of the rich strata project somewhat beyond the general surface presented by the leaner oil shales and the non-bituminous shales.

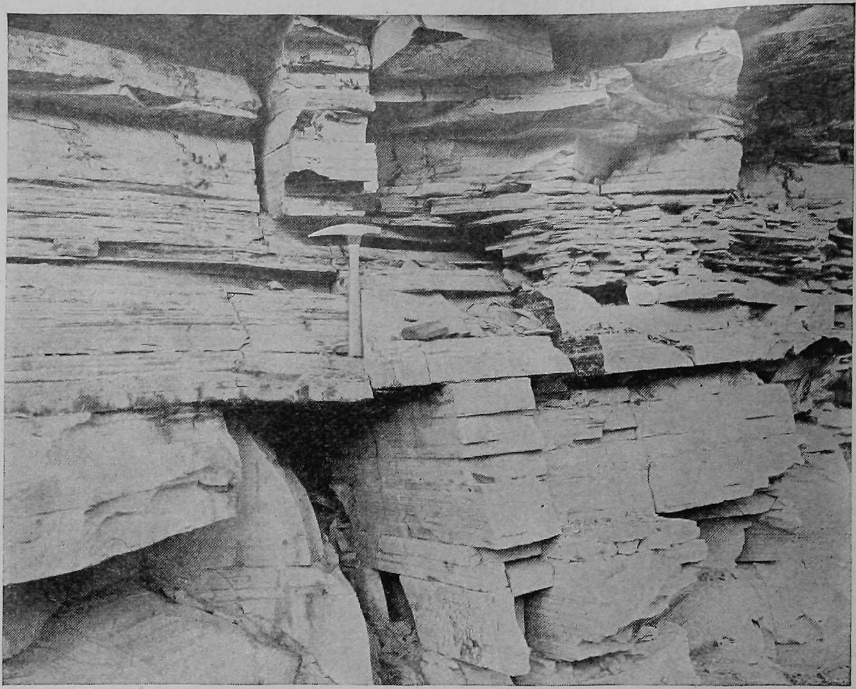

Fig. 4. A close-up view showing a rich deposit of oil shale.—Courtesy Denver & Rio Grande Railroad.

In weathering, the oil shales develop two rather characteristic types: First, the massive form in which the fracturing cuts the exposed edges of the strata into irregular patterns with relatively little reference to bedding planes, although fractures along bedding planes form some of the boundaries of the blocks. The weathered surfaces of some of these massive shales bear small lumpy projections, usually of lighter color and composed of sedimentary matter, with but little bitumen or organic content. The other type of shales, known as the paper shales, weathers into thin flexible laminæ with smooth, clean-cut surfaces. Some bands of the massive shale have a rough, wavy, concentric, or lens-like structure, the center of which frequently weathers into small caverns and openings filled with fine papery material and often highly charged with iron sulphate and other soluble salts. This structure appears to occur more commonly in the strata that contain a considerable amount of iron sulphide.

Thin slivers of either the paper shales or the massive shales burn quite readily, and hot, smoky fires may be developed with but

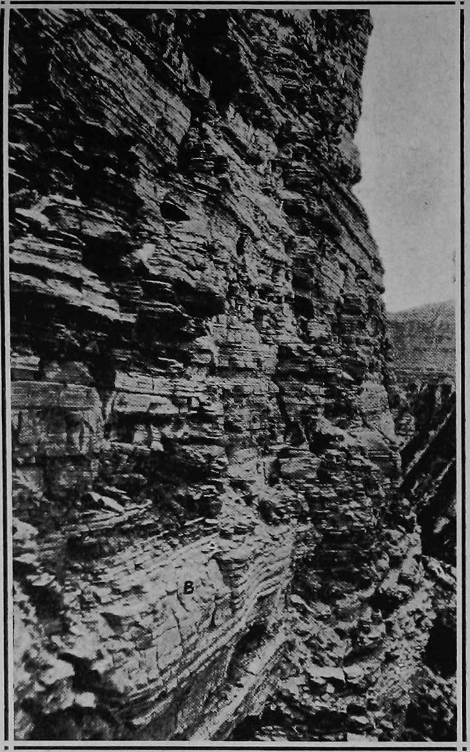

Fig. 5. Lower shale ledge (B) showing typical paper shale.
Sec. 26, T. 5 S., R. 100 W., Carr Creek.

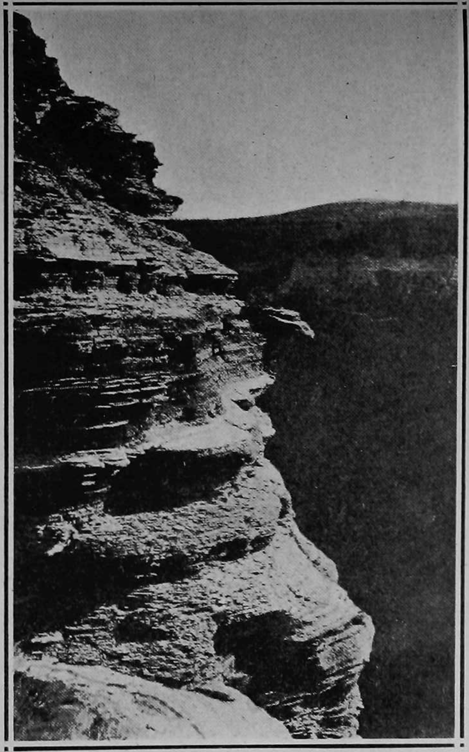

Fig. 6. Detail of lower shale ledge (B) in cliff, with lean shale above.
Sec. 10, T. 6 S., R. 98 W., Clear Creek.

little trouble from any of the richer shales. At a number of points along the cliffs the shales have been kindled, and fires have burned for a considerable time, converting some of the shale into red, brick-like debris, and some of it into a slag resembling that from furnaces. The origin of the fires can only be guessed. The position of the points suggests the possibility that lightning kindled the brushwood and the heat so developed was sufficient to ignite the shales.

PETROLOGY OF THE OIL SHALES

These great deposits of oil shale may be spoken of as an unfinished oil field. The shales, with their great content of organic matter, were laid down in horizontal position and large areas have remained but slightly disturbed since the time of their formation. The whole series of rocks in which the shales occur consists of alternating layers of shale with little organic material, shale with much organic material, and sandstone with little or no organic remains. It is reasonable to suppose that if the oil shale area were subjected to great earth movements, compressing and folding the strata and arching them into anticlines and bending them into synclines, the heat and pressure developed as a result of such movements would cause the distillation of oil and gas from the organic remains. The oil and gas so formed would find their way into the porous sandstones and would be prevented from escaping by the overlying impervious shales. As these substances are lighter than water they would travel through sandstone strata charged with ground water to the crests of the anticlines, and the gas, oil and water would arrange themselves in the order of specific gravity in the anticline, and we should have a duplication of the commonest form of oil structure and the commonest condition of the occurrence of oil and gas.

A rough analysis of the shale made as proximate analyses of coal are made, shows that it contains a varying percentage of organic material which can be converted into vapors and gases, and which is known as volatile matter. This volatilized matter is largely hydrocarbon gases condensible into oil. There is also a percentage of fixed carbon, which is not convertible into gases and vapors by the methods used, and a residue of inorganic material, mainly extremely fine sand and clay corresponding roughly to the ash of the coal. In the gases formed there is also a small amount of nitrogen which, in the workable shales, ranges from 0.5 per cent to 0.75 per cent. Other tests appear to show that the hydrocarbons developed from the shales naturally belong mainly to the paraffin series, commonly known as the saturated hydrocarbons. But in the retorting process a large percentage of unsaturated hydrocarbons, chiefly olefins, is formed.

As nature left this oil undistilled and the oil field unfinished, man must supplement her work before he can obtain the oil. This may be done by heating the shales in retorts, converting the organic substances into vapors, and condensing a part of the vapors into liquid oil, while the uncondensable part contains methane, the principal constituent of natural gas, together with the products of combustion. The moisture of the shale is converted into steam which is condensed with the oil.

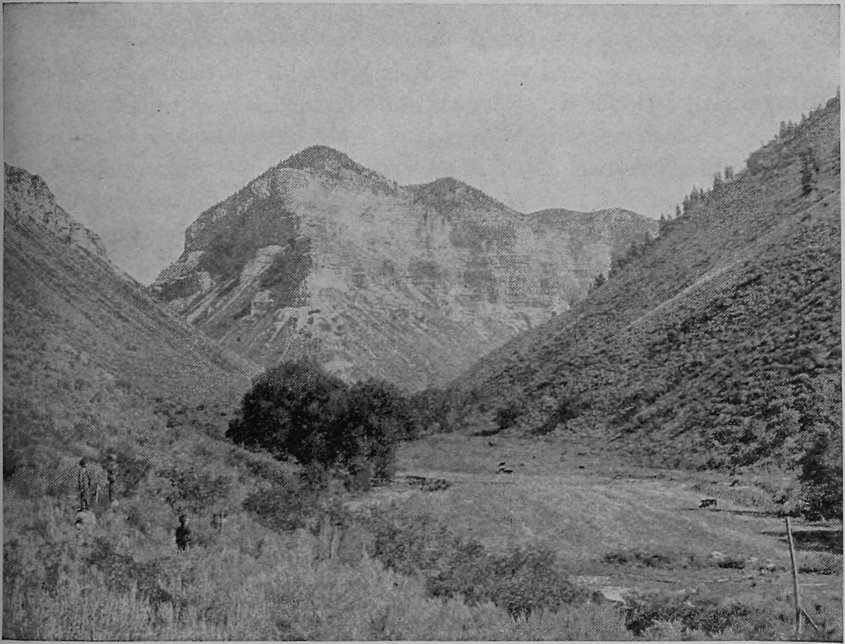

Fig. 7. Vast bodies of oil shale awaiting development on Parachute Creek, near Grand Valley, Colo.—Courtesy Denver & Rio Grande Railroad.

As to the quantity of oil contained in the shales of Colorado, Utah and Wyoming: It is as yet impossible to speak with any certainty, but there can be little doubt that there is in workable beds of shales in Colorado alone ten times as much oil as has been produced in the United States since the discovery of petroleum in Pennsylvania in 1859. The areas underlain by oil shale in Utah and Wyoming are much larger than those in Colorado, but the oil shale strata are thinner, and the oil content is lower, but it is not unreasonable to assume that each of these states contains as much oil in workable beds of shale as does Colorado's smaller area.

Many other states also contain workable beds of oil shales, though they are for the most part much leaner than those here described. The Nevada shales, and some of those of California, are equally rich, but the areas are smaller.

ORIGIN OF THE SHALES

An examination of the shales does not reveal the presence of oil as such. They are not merely porous shales into which oily or other bituminous matter has seeped or penetrated. Within the body of the shales there are no pores or pockets filled with oil. If the oil of the Green River formation had seeped into the strata, it would naturally have found its way to the most porous layers which are the sandstones. But it is a notable fact tnat the sandstones of the Green River formation contain little or no oil or oil-yielding substance, while the richest shales are those in which the mineral matter such as sand and clay is the most finely divided and would naturally be the least porous. The conditions of deposition must have been in some respects similar to those which have given us our great accumulations of coal. The only approach to a developed bituminous material found in the shales is the occurrence of very minute specks and occasional lens-shaped masses from one-sixty-fourth to one-thirty-second of an inch in diameter of a lustrous solid hydrocarbon closely resembling gilsonite. Solution tests of the shales substantiate the view that but little of the oil obtainable from the shale exists in the rock as true organized hydrocarbon. An examination of thin sections from some of the shales shows countless fragments of plant material, such as pollen grains, spores, leaf and stem fragments, and others. When a chip of the shale is leached of its sand and clay substance it forms a soft, spongy mass, consisting of a matrix of extreme fineness in which plant fragments are buried.

From a consideration of these facts it would appear probable that the oil obtainable from the shale comes from two sources:

(1) The small specks of true hydrocarbon scattered through the shale, and which have doubtless been derived from the organic remains buried in the rock.

(2) From the distillation of plant (and possibly, but not probably, animal) remains composing the spongy mass remaining after the inorganic matter is dissolved out.

Some of the plant remains were found by Dr. C. A. Davis, of the United States Geological Survey, to be in upright position as though the plants had grown on the floor of the lake and had been buried in their growing position by the accumulation of plant fragments and extremely fine inorganic sediment.

For the purpose of detailed study a series of 50 to 60 thin sections were cut from shales in various relations to the bedding planes and ground down to a thickness of one-thousandth of an inch for examination under high power microscopes. The sections represented shales of all grades, from those containing a trace of bituminous or plant matter to others giving an oil yield of 90 to 100 gallons per ton.

In sections from the leaner of these shales the organic materials are extremely scarce, and only the merest fragments of highly altered plant fibre and occasional spores or pollen grains proved the presence of vegetation in the lakes in which the sediments were deposited. At first thought one would expect the microscope to reveal in the richest shales the largest amount of recognizable organic material, but contrary to this expectation, comparatively few of the sections of the richer shales gave even fair opportunities for the study of the organic remains responsible for the bituminous matter they contained. In these the organic matter appeared to be reduced to much smaller particles and to be much more completely bituminized than in some of the shale having a much smaller oil content. The lustrous black shales rarely afforded sections in which recognizable plant remains could be found. In the writer's experience, by far the best material for microscopic examination is the dark mahogany brown, faintly lustrous shale. In some of the sections from this type of rock shreds of plants, fragments of fruits or seeds, pollen grains and spores were often so numerous as to occupy more than half the field of the microscope. Cell structures are, in some cases, beautifully preserved, and the paleobotanist should be able to recognize and classify broadly a considerable number of plants represented in the shales.

The peculiar waxy appearance of the material formed by scratching or scraping the richer shale is believed by some observers to be due to the presence of a solid hydrocarbon, possibly a paraffin wax of very high melting point. But heating and solution tests do not appear to support this view. It seems inprobable that in even the richest shale there could be a sufficient amount of true hydrocarbon present to give the waxy luster to the powder. It is noticeable also that some of the richer shale yields a brown, noncoherent powder without waxy luster. It is more probable that the waxy luster is due to the presence of kaolin or a related hydrous aluminum silicate. McCoy[2] succeeded in getting a little oil by subjecting certain shales to extremely high pressure, but it is not clear that the oil was not generated in the process of compression. It is known that at certain points along the Petrolite Hills, and at other places in the shale areas, there are indications of a liquid hydrocarbon in the shales. But these indications are found where the folding and compression have been pronounced, and where, as a natural consequence, greater heat would have been developed.

Considering the shale deposits as a whole, the following points appear to be established:

(1) The shales contain no hydrocarbons liquid at atmospheric temperatures.

(2) Solid hydrocarbons in minute particles and in widely differing quantities occur in the shales.

(3) There appears to be no direct relationship between the oil yield and the presence of these hydrocarbons. Shales apparently free from the hydrocarbon particles may give a high yield of oil, while others in which such particles are numerous may give a low yield.

(4) The shales contain much partially bituminized vegetable matter (and possibly some animal matter), and the quantity of this and the hydrocarbons together, determines the possible oil yield.

(5) The degree of bituminization of the vegetable matter differs from place to place, and from stratum to stratum. This change in the vegetable matter is comparable to the evolution of coal from plant matter.

(6) The oil-yielding materials of shales are related to lignite, or coal, on the one hand, and to the true hydrocarbons on the other. The closeness of the relationship to each depends upon the nature and degree of the alteration of the vegetable matter of the shale. While the facts just stated throw much light on the origin of the shales, there remain many problems which must be worked out before the whole story of the oil shales can be written.

STRUCTURAL FEATURES OF THE SHALES

In the field the geologist readily recognizes two broad classes of shale strata. By far the most important of these are the massive strata, which, throughout the greater part of the area, form 75 per cent to 90 per cent of the total volume of workable material. The other type, on account of the weathering of the outcropping edges of the strata into very thin laminæ, is known as paper shale.

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[2] Notes on Principles of Oil Accumulation. Jour. Geol., vol. 27, pp. 252-262, 1919.

In some places these two types appear to be quite distinct, and for long distances one may look in vain for the evidence of paper shale in the strata which are predominantly of the massive type. But here and there, especially in the areas in which iron sulphide and the weathering product, iron sulphate, occur, even strata predominantly massive may take on the paper structure. There are, in many places, strata which are prevailingly of the paper type, but in some places the papery shales give place to the massive forms.

A third, but very subordinate type, may be called wavy or curly shales. These occur in strata of both massive and papery types. In a few places these curly shale strata form good horizon markers.

The distribution of joints in the shale strata is extremely irregular and inconstant. In some places one may follow the outcropping edges for hundreds of yards without finding a prominent joint, while in other places prominent joint planes are somewhat closely spaced, but their vertical courses are rarely long enough to cut the thicker strata from top to bottom. This will also prove of considerable importance in the mining of the shales.

Along the outcrop of the shales one may observe widely varying conditions of stratification. In some places a large body of shale may appear to form but a single stratum which will reach a thickness of 10 to 12 feet. In other places the outcropping edges may be distinctly divided into strata a few inches to 2 or 3 feet in thickness. In places the laminæ of the paper shale may become less and less distinct, until all evidence of their existence is lost and the whole bed presents the appearance of a single compact stratum.

The importance of jointing and stratification as aids to mining is naturally very great, and while it may be impossible to select areas in which both are well defined and prominent, it is rarely impossible to take advantage of one or other of these structural features.

WEATHERING

The bitumen content has rendered the oil shale strata more resistant to the processes of weathering than are the enclosing barren clay shales and the softer sandstones. Small fragments of oil shale are notably scarce in the debris and alluvium at the foot of the precipitous cliffs in which the oil shales outcrop. Large blocks bounded by joint planes are common in the coarser talus at the foot of the cliffs and on the steeper slopes. An examination of the exposed edges of the massive shale strata impresses one with the idea that, except for the breaking away of the larger joint blocks, the erosion of the shales must be quite largely a process of solution and disintegration into the extremely fine sand and clay particles which form the inorganic matrix in which the organic material is contained. By exactly what agencies the bituminous matter is removed in this process of disintegration it is impossible to say. But it is certain that the process of the removal of this material is slow and goes but slightly ahead of the separation and disappearance of the inorganic material. The removal of the bituminous material from the richer shales leaves a grayish white layer over the surface, which, on unfractured blocks, ranges from a mere film to rarely more than one inch in thickness. Where the shale is finely laminated by closely spaced joints the weathering has penetrated to a greater depth. But it is a notable fact that the oil content of the massive shale has rarely been diminished by weathering to a depth of more than 2 or 3 inches.

In the case of the paper shales, weathering has opened up the fine layers and separated them in fan-like forms, into which the weathering agencies have penetrated for a considerable distance. But even in these, with rare exceptions of deep jointings, has the oil content been markedly reduced beyond 3 or 4 feet from the outer surface.

COMPARISON OF SCOTCH AND COLORADO SHALES

COMPOSITION

Analyses of the Scotch and the Colorado shales show that they vary from place to place and from stratum to stratum, both in their organic and inorganic contents. The Scotch deposits vary in both these particulars much more than do the Colorado shales. Lean shales alternate with the richer both vertically and along the bedding plane. The Colorado shales are much more uniform in oil content along the strata, and there is probably much less rapid variation in a vertical direction. The inorganic materials of the shales appear to have little bearing upon the problems of the oil shale industry. The more siliceous or sandy beds of Colorado shale may break more readily in mining than do the more clayey strata, but in the retorting the character of the inorganic matter plays no part so far as the writer has been able to observe. It was thought that the sandy shales would yield their oil more readily than would the clayey, but tests do not substantiate this idea.

POSITION OF THE SHALE BEDS

The Scotch shales are much less accessible than are large volumes of those of Colorado. Most of the shales of Scotland are mined through shafts or slopes. The Colorado shales outcrop in nearly horizontal strata along the Book Cliffs front and along the walls of valleys cut into the great plateau area. But it must be kept in mind that back of these outcrops lies the great mass of shales miles from any possible frontage on the valleys. The mining difficulties confronting the shale operator owning claims without such frontage are comparable to those of the Scotch operator, with the additional problem of a very scanty water supply.

STRUCTURAL FEATURES

The Scotch shale beds are folded and faulted, and thicken and thin rapidly along the strata, while the Colorado shales lie mainly in horizontal strata, rarely folded or faulted, and of remarkably uniform thickness. These conditions are most favorable for mining operations, and should greatly reduce the cost of getting the shale to the retorts. The amount of timbering required should be much less, and the cost of timber per prop should be much less.

THE OIL CONTENT

The average yield of the Scotch shales is reported at about 18 to 20 imperial gallons, or 22 to 24 U. S. gallons, per ton. There are billions of tons of shale in the Colorado deposits which will yield double this amount of oil, and vast quantities that will yield from 50 to 60 gallons per ton. In addition to these are immense tonnages which will equal the Scotch in oil content.

The ammonia yield of the Scotch shales is probably double that which may be expected from the Colorado shales, but there can be no reasonable doubt that the ammonia will prove a valuable product, and will add considerably to the profits.

THE CRUDE OIL

There is but little real difference between the qualities of Scotch and Colorado crude shale oils. Both present serious refining difficulties as compared with well oils, but products of the highest commercial quality may be secured. Prejudices will have to be overcome, and possibly a campaign of education will be necessary. But *need* and *scarcity* are the most potent factors in overcoming prejudice, and are very apt teachers.

ESTIMATE OF TONNAGE IN SHALE STRATA

The specific gravity of shales yielding from 40 to 60 gallons per ton is about 1.6, and approximately 20 cubic feet will weigh one ton. An acre contains 43,560 square feet, and an acre-foot of shale contains 43,560 cubic feet, or 2,178 tons. Assuming a yield of one barrel of oil per ton of shale, each acre-foot would contain 2,178 barrels of oil.

For many miles along the sinuous face of the southern exposures of the Green River shales there is an aggregate thickness of upwards of 35 feet of workable shale in beds exceeding 3½ feet in thickness and containing an average of over one barrel of crude oil per ton. An acre of such territory would contain over 76,000 barrels of oil, and a square mile would contain 48,640,000 barrels. The total shale area of Colorado exceeds 2,000 square miles. Assuming that it averages 20 feet of workable shale containing 1 barrel of oil per ton, the total crude oil content would be approximately 56,000,000,000 barrels. This is ten times the estimated remaining supply of petroleum in the known oil fields of the country.

MINING SHALE

The problems of shale mining are much the same as those of coal mining, and will be solved in the same way. The Colorado shales lie mainly in almost horizontal beds between strong shale roof and floor rocks, or occasionally sandstones. The thickness of the beds and the oil content are remarkably constant.

Timbering will be necessary in places, but not nearly so generally necessary as it is in coal mining in Colorado. Permanent entries, air ways and haulage ways can be maintained largely by walls of lean shale and waste.

The conditions appear to be favorable for long wall and panel methods of mining.

Much has been said regarding the possibilities of gas in the workings. Nothing is positively known. It would be very reasonable to expect the same gases as are found in coal workings, both from open pockets in the shales and from slow filtration from the body of the rock. The writer made a few tests by plugging the mouths of drill holes in the solid shales to see whether they would fill with gases, but the results were negative. This is not conclusive, and only experience will answer the question.

Cutting machines, such as are used in coal mining, can be adapted to the needs of shale mining. Greater strength and different cutting devices will be necessary, but the principles and general design of the machines will serve the purpose.

Since the shales are tough and non-friable, they will be much harder to break than is coal. But as no precautions are necessary to avoid the formation of slack and to secure lump, much heavier charges of powder may be used.

Along the southern front of the great shale area the beds are at an elevation of 1,200 to 2,000 feet above the valley floors. If plants are located in the valleys the mined shale can be lowered by gravity trams or milled down to loading tunnels near the level of the valley floors.

Much has been said about steam shovel mining. Not one acre in a thousand of the shale deposits offers any possibility of handling by such a method. Here and there a few acres of good shale are exposed at the surface. But the amount of shale which will be mined by steam shovel will be negligible as compared with the vast tonnage which must be reached by entry and shaft.

As to the cost of mining, the only guides we have are American or Colorado coal mining and Scottish shale mining. It would seem probable that the costs of opening and equipping a shale property would be much the same as those of opening a coal mine for a like tonnage production. The costs per ton thereafter should also be comparable, and will probably range from 80 cents to $1.20. They certainly should not exceed the higher figure.

SHALE OIL PROBLEMS

The obtaining of oil from shales is a chemical process, and chemical reactions depend upon physical conditions. The physical conditions are imposed by the apparatus we use and the way in which we handle that apparatus. If our apparatus or our handling of it imposes physical conditions out of harmony with the results desired, it is useless to look for success.

The secret lies in knowing what reactions must take place to produce the desired results, and in shaping the apparatus and operating it to secure those reactions.

The writer has tested the Scotch oil shales and oil shales from England, South America, Canada, California, Nebraska, Colorado, Utah, Wyoming, and other states. These shales differ in details, but the fundamental problems of treatment are the same—the problems of hydrocarbon chemistry. These problems have been solved in Scotland, France, and Australia. The history of the Scotch shale oil industry is a story of scientific research by chemists and engineers of the highest rank. It is a story of evolution—the survival of the fittest in apparatus and in manipulation. The road is strewn with discarded apparatus—the best in its day. It is marked by abandoned theories and processes, each having played its part in reaching the goal.

Instead of profiting by the half century of experiment and experience of Europe, most of our experimenters have ignored all the progress made at the expense of ceaseless toil and millions of money, and have chosen to be "pioneers," secretly cherishing the thought that chance, inspiration, revelation, or their own superior mentality will enable them to present to the world a process beside which the poor old Scotch process will recall the days of the stage coach.

They are presenting theories and "inventing" apparatus and processes abandoned twenty or thirty years ago. If they want to improve on the Scotch process why not start from the best and advance? The exact form of the retort is not vital, but the physical conditions it imposes are vital. The form of the retort may be the best ever, but the operation of that retort may produce the worst results ever.

It is highly probable that modifications of the Scotch retorts and processes may be needed to meet peculiarities of our Colorado and Utah shales, but the broad needs are the same.

Our shales far exceed the Scotch shales in oil content, and, consequently, the tendency to coke and clog will be greater. This is not an insuperable difficulty. Our shales contain a smaller percentage of nitrogen available for ammonia. This fact may make certain modification of treatment desirable. But it is not fundamental. Our shales appear to contain more pyridines than do the Scotch. This will modify the refining process, and certain changes in the retorting may help us to avoid the difficulties they present.

Much has been said about extracting the oil in 20 minutes, 30 minutes and other short times. Possibly it can be done, but the getting of the oil (or oil and fixed gas) is not the final goal. The ultimate purpose is the refined products. Will the 20-minute oil yield them?

The scientific problems to be faced by those who seek to establish a shale oil industry fall into two groups:

(1) The problems of retorting.

(2) The problems of refining.

In this country, up to the present, attention and effort have been directed almost entirely to the solution of the problems of the first group. This is due to the natural, but erroneous assumption, that crude shale oil is like crude well petroleum, and may be successfully refined by the same processes.

This assumption would be reasonably safe if oil shale were merely shale saturated with petroleum. True oil shales like those of Colorado and Scotland contain no ready-made oil, but are rich in the materials from which oil may be formed if the shales are properly treated.

The problems of retorting are:

(1) To convert as much as possible of the oil-making material of the shale into oil or other useful products.

(2) To secure a crude oil containing the largest possible percentage of the most valuable constituents, such as gasoline, kerosene, and lubricating oils, and the smallest possible percentage of worthless and harmful materials which must be removed as waste in refining.

(3) To secure a crude oil which is easily fractionated into gasoline, kerosene, lubricating oils and others, and which yields fractions or cuts which are easily refined.

(4) To secure as large a yield of ammonia and other valuable by-products as possible without sacrificing more desirable results.

(5) To reach the highest commercial efficiency without sacrificing the raw materials of the company or of the country.

The cost of equipment and operation of the refinery will depend very largely upon the efficiency of the retorting plant in meeting and solving these problems. It will be found that a little greater care in retorting will be rewarded by a great reduction in refining costs.

RETORTING PROBLEM 1

The carbonaceous material of Colorado oil shales appears to occur in three forms: (1) true hydrocarbons in the solid form occupying extremely small pits and intergranular spaces in the shale; (2) plant and possibly animal matter which has undergone changes akin to those by which vegetable matter is converted into coal, and (3) fixed carbon. The relative proportions of these three materials are far from constant. In some thin layers of rich shales the true hydrocarbons, probably paraffins, greatly exceed the other two materials. Such shales, when examined under a powerful microscope, show great numbers of grains of true hydrocarbons, and very little, if any, vegetable tissue. The fixed carbon content of such shale is generally low. On the other hand, there are shales in which the vegetable tissue greatly exceeds the hydrocarbons in quantity.

In retorting, the true hydrocarbons appear to vaporize at a comparatively low temperature, while a higher heat is necessary to develop hydrocarbon gases from the vegetable matter, and the last hydrocarbon gases are given off at a very high temperature. Whether these last come from the true hydrocarbons or from the altered vegetable tissue it seems impossible to determine. The fixed carbon cannot be converted into hydrocarbon gases unless, in some way, the necessary hydrogen is supplied, and it is doubtful whether the hydrogen and carbon can be made to unite under the conditions prevailing in the retort.

From the above facts it will be clear that the shales are not simple homogeneous materials yielding their gases by volatilization at a definite temperature or within a narrow range of heat.

Two questions arise: Is the gas which comes off at the highest temperature worth getting, or is it secured at too great cost of fuel and time? Is there any means of securing these last hydrocarbons at lower temperatures? To the first question the answer must depend upon the proportion of these high temperature hydrocarbons to the total yield, and their character and value. The proportion will be different in different shales. Their value is probably questionable, as the high temperature necessary is likely to make a large percentage of uncondensable gases valuable only for fuel, and other compounds which will add greatly to the difficulties of refining. The second question may, with reasonable certainty, be answered in the affirmative. The use of superheated steam in the retorts seems to reduce considerably the temperature required to drive them off. And with low temperature retorting there is a notable reduction in the proportion of uncondensable gases, and the formation of undesirable compounds. To secure these last gases for their fuel value would be penny-wise and pound-foolish, since equally good fuel can be obtained much less expensively.

The fixed carbon has a high fuel value, but unless air is admitted it cannot be directly utilized as fuel, and will remain in the spent shale.

When steam is used in retorting, the conditions in the retort resemble those which obtain in the manufacture of water gas, but the best temperature for the formation of the oil vapors is too low to convert the carbon into carbon monoxide. If, after the oil vapors are driven off, the shale is passed on to another section or chamber of the retort or to a secondary retort and there raised to a much higher temperature to get the maximum yield of ammonia, steam passing through this highly heated mass will not only aid in getting the ammonia, but will cause the formation of water gas by the union of the oxygen of the steam with the fixed carbon of the shale, and the freeing of the hydrogen. In this way the fuel value of the carbon may be saved, and the fuel value of the hydrogen added. A temperature of 1300° F. is necessary.

When superheated steam is used the desirable gases are given off at lower temperatures. Its value in the solution of other problems will be discussed later.

RETORTING PROBLEM 2

Some well petroleums yield a large percentage of gasoline, kerosene and other light oils; others yield little or none of these light fractions. Some yield high grade lubricating oils; others are worthless for this purpose. Some well petroleums would yield so little of any refined products that they are valuable only for fuel. Others contain valuable constituents, but yield them only at prohibitive cost, and so cannot be profitably refined.

Crude shale oils differ just as widely in their possibilities, but the differences are due to the methods of manufacture, not to uncontrollable natural causes. Oils are composed chiefly of hydrocarbons, but there are many different forms of hydrocarbons. In the formation of common petroleums in the earth, the character of the original materials, the heat and pressure to which they are subjected, the presence or absence of water, the character of the rocks and mineral substances with which they come into contact, are all factors in determining what hydrocarbons will be formed, and, consequently, the character and value of the oil which will result. It is equally true that the conditions of retorting will determine the character and value of the shale oil produced.

What are the controllable conditions in retorting shales for the manufacture of oil?

(1) The rate of applying heat.
(2) The maximum temperature reached.
(3) The presence or absence of water in the retort.
(4) The access of air.
(5) The time the shales are held at different temperatures.
(6) The pressure in the retort.
(7) The removal of the vapors from the retort.
(8) Added materials as catalysts or as actual chemical reagents, etc.
(9) The size of the material.
(10) The heat distribution—even, uneven, etc,

As to the rate of applying heat.—Time must be given for the heat to penetrate the shale, and for the gases to form and escape. Experience shows clearly that certain gases are formed at low temperatures, others at intermediate temperatures, and others at high. Time is a factor in all chemical reactions. If the gases formed at low temperatures are the best for oil making, the heat must be applied slowly to give time for it to penetrate the shale and for the gases to form. In lime burning this is a most important factor. Too rapid application of heat causes sintering of the stone and the closing of the pores by which the gases escape. A similar condition results in the shales by the excessive formation of fixed carbon through the cracking of the hydrocarbons of the shale and the cracking of the gases, both of which are wasteful and result in excessive production of fixed or non-condensable gases, and an oil low in gasoline. Objectionable compounds are formed which must be removed in refining, and some of them present the greatest difficulties encountered in refining and greatly add to its cost.

The gases evolved at low temperatures are the best, the proportion of condensable gases is largest, the percentage of paraffin gases is highest, and that of impurities is lowest. The oil obtained is more easily refined, and the proportion of light oils is greatest.

The maximum temperature.—Retorting at too high temperature results in the formation of much fixed gas, a low content of gasoline and kerosene in the crude oil, inferior lubricating oil and a crude hard to refine. The Scotch call it "burnt oil." It is possible to raise the temperature so high as to decompose the hydrocarbons into the elements hydrogen and carbon, in which case no oil would be obtainable. But the shales contain varying proportions of material which can be converted into hydrocarbon gases only at temperatures so high as to be very injurious to those gases formed at low temperatures. Similar high heat is required to get the best yield of ammonia. In some shales the amount of carbonaceous material convertible only at high temperature is negligible, but in others it may reach 15 to 20 per cent. The problem suggests several possible solutions.

(a) Sacrificing the high temperature material and the high yield of ammonia. This would save fuel and time and increase the throughput, and might compensate for the loss of oil and ammonia.

(b) Doing the principal part of the retorting at a relatively low temperature and passing the shale on to another section of the retort, or to a finishing retort, where it may be subjected to the high temperature necessary for securing the last of the hydrocarbons and the higher yield of ammonia. This is largely practiced in Scotland, possibly because the ammonia is relatively a more important product than it would be in America.

(c) Intermittent retorting—securing the main yield of condensable gases at low temperature and finishing the charge at high temperature. It is known that the high temperature gases contain a lower percentage of condensable gases than do the low temperature gases, and are of less value.

It should be stated that with the use of superheated steam in the retort the last hydrocarbons and the larger yield of ammonia are secured at lower temperature than by dry distillation. In fact, it is impossible to secure the full possible yield of ammonia without the superheated steam.

Experiment and experience and markets will decide which of these solutions of the problem will be best.

Presence or absence of water in retort.—The beneficial effects of superheated steam in the retort appear to be:

(a) Evolution of gases at lower temperature, saving time and fuel.

(b) Twice or three times the yield of ammonia.

(c) A lower percentage of unsaturated hydrocarbons, possibly as a result of the protection afforded by the steam, and a lower yield of uncondensable gases, and of compounds which render refining more difficult.

(d) A higher percentage of lighter oils in the crude.

(e) The prevention of coking and clogging.

(f) The more rapid removal of the oil vapors from the retort preventing decomposition.

(g) It serves as a heat distributor and equalizes the temperature throughout the retort.

Under certain conditions the use of superheated steam is unsatisfactory. In tests made on samples of Kimmeridge shales from England the superheated steam caused the formation of an obstinate emulsion which made fractionation and refining difficult and slow. This was also noticed in testing a California shale. Once or twice obstinate emulsions were formed in treating Colorado shales, but the manipulation was at fault.

As to the quantity of superheated steam required: The best results were obtained by the use of water at the rate of about 650 to 750 pounds per ton of shale.

Access of air.—It is argued that the admission of air will increase the yield of ammonia. This is more than doubtful, since hydrogen and nitrogen unite at temperatures above 500° C. and at very high pressure. It is highly probable that both the nitrogen and the oxygen of the air may form injurious combinations. Air certainly causes mechanical difficulties. Some Scotch operators admit air at the bottom of the retort where the temperature is run up to get the last of the ammonia. The oxygen of the air unites with the fixed carbon forming monoxide, a fuel gas.

Time required at different heats.—The low temperature distillation requires much more time than does the finishing high temperature.

Pressure in the retort.—Experience in the cracking of oils suggests the desirability of retorting under pressure to prevent the cracking or decomposition of the paraffin hydrocarbons. But experiments in retorting under pressure have proved unsatisfactory. On the other hand, the longer the gases are allowed to remain in the hot atmosphere of the retort the greater the cracking and decomposition. A slight suction reducing the pressure about one pound below that of the atmosphere causes a quick removal of the gases formed and probably prevents much decomposition. The Scottish retorts are run under slightly reduced pressure.

The removal of vapors from retort.—This should be as rapid as possible. Many attempts have been made to remove and keep separate the vapors formed at different temperatures and effect a selective condensation. But, while this is possible in small measure, it is doubtful whether it will be possible or profitable commercially. It is highly desirable to have numerous outlets for the gases so that they may escape from the retort without passing a long distance through the hot shale.

Added materials to facilitate or improve results.—Various materials have been mixed with the shales in the retort with a view to bringing about reactions and improving the products. Such materials belong to two classes:

(a) Catalysts—substances which induce or condition reactions but which do not take part chemically.

(b) Substances which enter into the chemical reactions.

Definite beneficial results have been obtained in laboratory work along this line, but considerations of cost of materials, bulk and handling appear likely to offset the advantages gained. To be commercially valuable and practical the materials must be cheap, readily available, effective in small amounts, and must produce positive results which tell in the quality of the finished products or in the operation of the retorting and refining processes.

Perhaps one of the most desirable results to be aimed at would be the elimination of the very persistent coloring matter which characterizes shale gasoline and kerosene.

The size of the material.—The heat penetrates the shale very slowly. It is, therefore, desirable to crush it rather fine, so as to give large surfaces in proportion to the volume of the fragments. It should not be so fine as to pack and clog.

Heat distribution.—On account of the slowness with which heat penetrates the shale, it is of the greatest importance that the body of shale should not be too thick. In certain forms of retorts stirring devices may be used to facilitate heat penetration of the mass, and so prevent overheating or spot heating and the objectionable results which follow it. Heat distribution is greatly aided by superheated steam.

Even the best crude shale oil contains a large percentage of unsaturated hydrocarbons called olefins. Until recently the refining methods used in the United States removed the olefins from kerosene and gasoline. This led to the erroneous notion that olefin hydrocarbons could not be used in motor fuel, and a number of writers have stated that shale oil could not be used for the production of gasoline. If by ''gasoline'' is meant a motor fuel consisting mainly or wholly of paraffin or saturated hydrocarbons, their contention is correct, for the loss would be prohibitive. But if the term ''gasoline'' is made to include all fuel suitable for use in internal combustion engines similar to those used in automobiles, the contention is far from correct. The motor spirit put on the market by the Scottish shale oil companies contains from 60 to 70 per cent of olefins, and 30 to 40 per cent of paraffins, and it gives highly satisfactory results.

It is an established fact that the diolefins, and not the olefins, are the chief trouble makers in gasoline produced by cracking. They are responsible for the foul odor, for the change in color, and for the formation of the heavy resinous oil which commonly forms in such gasolines. If the diolefins are completely removed the olefin-rich gasoline will not deteriorate with age to a greater extent than will ordinary paraffin gasoline. The chemical properties of the olefins and diolefins are so very similar that the problem of separating them is a very difficult one. The sulphuric acid treatment can be so modified as to remove practically all the diolefins without serious loss of olefins. A gasoline containing over 60% of olefins has stood in the Survey laboratory (in direct sunlight a large part of the time) for over two years without noticeable change of color.

Other methods of removing the diolefins have been worked out in the laboratory, but whether they will prove to be of commercial application has to be determined. Certainly, a method will be found, if, indeed, the Scotch refiners are not using such a process.

In European countries large quantities of benzol are used as motor fuel. This is obtained by the distillation of coal and consists of members of the benzene series of hydrocarbons, and contains no paraffins. And yet it is a satisfactory motor fuel.

Some of the cracked gasolines on the market in this country contain as high as 25 per cent of unsaturated hydrocarbons (olefins), and the users do not even know it.

The problem is not to secure from shales a "gasoline" or motor spirit composed of paraffin hydrocarbons, but to secure a motor spirit or motor fuel which will do the work regardless of its composition. Experiments have shown that the explosive energy of an olefin motor spirit is delivered more slowly, but through a longer period of time, than is that of a paraffin gasoline. They have also shown that the total energy delivered weight for weight is from 10 to 15 per cent greater for the olefin motor spirit than for the paraffin gasoline, and that the mileage obtained is correspondingly higher. Olefin gasoline vaporizes at a lower temperature than does ordinary gasoline of the same gravity, and is, therefore, a better winter motor fuel.

The gist of these facts is that shale oil "gasoline" will deliver 10 to 15 per cent more energy, and will deliver it in a steadier or less jerky fashion, causing less vibration and. therefore, less strain upon the motor and the machinery driven by it.

It is contended that the olefins deposit more carbon in the cylinders. This is a problem of carburation and has been solved.

RETORTING PROBLEM 3

Some shale oils are hard to fractionate, hard to refine and yield but little of value. Some are so spoiled in the making as to be unrefinable and worthless except for fuel. Colorado shale oil should yield 16%-22% natural gasoline, and 40%-45% kerosene.

The crude oil should be easily fractionated and the fractions should be easily refined.

The best results in this direction are obtained by low temperature retorting with superheated steam. High temperatures and dry distillation tend to unnecessary reduction of the light oil content, the formation of crudes from which but little gasoline can be obtained without cracking, and the production of obstinate sulphur compounds and nitrogen-containing compounds which render refining difficult and costly and reduce the grade of the products obtained. It also reduces the yield of condensable gases and increases that of the fixed gases.

The advocates of certain retorts make the boast that they yield enough fuel gases to do the retorting. This boast means simply that they are doing poor retorting—making fixed gases instead of condensable gases, and by the same token are getting a low yield and inferior oil. It is infinitely better to secure as high a yield as possible of condensable gases; in other words, to accomplish the purpose for which retorting is done; namely, to make oil, and that of the highest grade possible. If fuel is needed it can be made to much better advantage and of better quality by installing a producer plant using shales as raw material. A single producer plant would supply several retorts.

It is impossible to avoid the formation of fixed gases, but if the retorting is properly done the volume need not exceed 3,000 to 4,000 cubic feet per ton of shale, depending upon its normal yield of oil. The proportion of hydrocarbons in this should not exceed 35 per cent. If a successful effort is made to convert fixed carbon into combustible gases the volume will be larger.

The essential features of the successful retort will include as many as possible of the following: Large daily capacity, ease of operation, perfect control as to temperature and the movement of the charge, the rapid withdrawal of the vapors, provision for the use of superheated steam, a possible gradation of temperature, inexpensive construction and upkeep and long life. It should also be so constructed as to make possible the conversion of the non-oil making carbonaceous matter of the shale into fuel for operating purposes.

AMMONIA YIELD

A large number of analyses of Colorado shales show an average nitrogen content of 0.6 per cent, which would correspond to 56 pounds of ammonium sulphate per ton. Only a part of the nitrogen is available for the formation of ammonium sulphate, probably not more than one-half. In retorting, a part of the available ammonia comes off at low temperature, and a part at high temperature. Superheated steam increases the yield of ammonia from two to three times, but a large part of it comes off at the high temperature. Unless it is found desirable and profitable to use the high temperature for the purposes of oil extraction, it may be doubtful whether the increased yield of ammonia would justify the high heating which would surely injuriously affect the hydrocarbon gases if they were heated to the same degree. Under certain conditions of market and demand for ammonium sulphate, it might be profitable to pass the shales directly from the main retort into a separate finishing chamber in which the full yield of ammonia could be secured. The gases formed could be turned directly into the fuel supply.

OTHER BY-PRODUCTS

Much nonsense has been written about the many valuable by-products of oil shales. It is true that many commercial products can be made from the shales, but most of those commonly listed can be made much more cheaply from other forms of raw materials. This is true of dye stuffs, medicinal salts and many other chemical substances which are now made from coal tar and the accompanying liquors.

A substance resembling ichthyol has been made, but it is very doubtful whether true ichthyol has been produced.

Synthetic rubber has been much talked of, but it is safe to say that nothing approaching a commercial process or a commercial quantity has been discovered.

A substance resembling gilsonite and possibly suitable for a rubber filler may be separated from the tarry residues.

Paraffin wax of high grade and readily marketable may be produced in commercial amount and profitably.

Analyses of several samples of spent shale showed an average potash content of 18 pounds per ton of spent shale. This is water-soluble and could be leached out at little cost.

The spent shale has been proclaimed a fertilizer, but it contains nothing of value in this way except the potash, and it would be absurd to list it with fertilizers.

It has also been talked of as raw cement material, and as brick material. It is of less than average value for either of these purposes.

One advertiser of shale oil stock has found that it is the best of material for glass and porcelain making. This is nonsense.

It is not even good road material and its disposition will present a problem.

The tars, still carbon, or coke, and the heavy residual oils will be utilized about the plants or converted into marketable products.

Lubricating oils of the highest grade are made from the Scottish shale oils, and laboratory quantities of lubricating oils made from Colorado shale oils have given extraordinary results when tested in actual use. They retain their viscosity or body at much higher temperatures than do oils of similar density and flash point made from well petroleum.

GOLD, SILVER, PLATINUM, ETC.

Much misinformation is being given out regarding the precious metal content of the spent shales. A large number of assays by thoroughly reliable and competent chemists and assayers have failed to give a single return which could by any reasonable means be called commercial. Traces of gold were found in possibly one-half the tests made by the Colorado Geological Survey.

PROBLEMS OF REFINING

The problems of refining may be summed up:

To refine, without undue waste, an oil containing from 50 to 60 per cent of unsaturated hydrocarbons, a considerable percentage of pyridines and other compounds.

To secure good color and permanence of color.

To secure a reasonably sweet odor.

To secure an oil which will not corrode the cylinders of the engines in which it is used.

To secure the largest possible percentage of valuable marketable products from the crude.

It has been shown that the unsaturated hydrocarbons (olefins) yield more energy, and yield it in a steadier and more sustained thrust than do the paraffin hydrocarbons; that an olefin gasoline of the same gravity will vaporize more readily, and is, therefore, a better winter motor fuel than a paraffin gasoline; that the shale lubricating oils, rich in olefins, hold their body (or viscosity) better under high frictional heat than do the paraffin-rich lubricating oils of the same density, and are, therefore, from this standpoint more desirable lubricants. It is evident, then, that the first problem of the refiner is to work out a refining process which will save all the olefins and give the user the benefit of their superior qualities, whether in the engine or on the bearings. The Scotch shale oil producers are doing this, and it is entirely reasonable to say that the American refiner can and will do it.

Today the makers of cracked gasoline are putting on the market a good-looking and well-behaved gasoline containing over 20 per cent of olefins. If they can do this the shale oil refiner can surely meet his problem. But it must be remembered that shale oils contain constituents which are not found in well petroleum, and these must be taken into account. In the laboratory this has been done, and the gasoline is water white and "sweet smelling" after two years. Commercial refining is a somewhat different problem, but there is no reason for discouragement. It can be done commercially.

A well-made crude shale oil should yield 16% to 22% of motor spirit (or gasoline), and about 40% of the kerosene fraction. These will contain roughly 60% to 70% of olefins. If the kerosene is not desired, a fraction containing it and a considerable percentage of heavier distillate may be taken off and cracked. The resulting gasoline will bring the total yield of gasoline up to 40% or over.

The odor of shale gasoline is not like that of common gasoline, but it is not more unpleasant. Some people believe it induces headache more readily, but it need not be used as a perfume or as a substitute for smelling salts.

The color problem appears to many experimenters very serious. It is hard to remove the dark brownish-purple color which characterizes the light fractions of shale oil, but it has been and can be done. Is this coloring matter injurious? Must it be removed? In the early days of the Pennsylvania oil industry the kerosenes put on the market were of many shades of color from water-white to greenish, violet, bluish and reddish. Did the color affect the quality? Refiners say it did not.

It may be that a campaign of education would remove the color problem.

It has been said that shale gasoline has a corroding effect on the motor. This is specifically denied by oil technologists familiar with the facts. Tests were made in the Geological Survey laboratory with laboratory-made shale gasoline from Colorado shales and no injurious results could be found on brass, bronze, steel or iron parts after tests lasting for weeks.

Another problem of the refiner is to secure the largest possible yield of marketable products from the shale oil. Only a few years ago kerosene was practically the only marketed product from well petroleum. Today such products are legion, and the profits are millionaire-making. It is entirely probable that many similar by-products will be made from shale oil. At present, gasoline, kerosene, lubricating oils, waxes, tars and cokes and ammonium sulphate may reasonably be counted upon as commercial products. Hundreds of others are chemically possible since the best of shale oil is more complex than is well petroleum, but many of the chemical possibilities do not appear to be commercial possibilities since the substances can be produced more cheaply from other raw materials.

The fundamental problems of the shale oil industry are solved. Details will have to be worked out, and apparatus and manipulation will have to be modified to meet the peculiarities of our various American shales. In the opinion of the writer, the retorting presents fewer difficulties than does the refining. But many of the refining problems will vanish if the retorting is done scientifically and in obedience to chemical laws. It must not be supposed that the same methods of refining can be successfully applied to oils which are chemically very different. If this is kept in mind and the chemistry of the unsaturated hydrocarbons is mastered, the refining problems will very soon yield to chemical skill.

LABORATORY STUDY OF COLORADO SHALES

HISTORICAL SKETCH

At the present time the distillation of shale for oil and the various by-products is an important industry in Scotland, France, Australia and New Zealand. In Germany a brown coal (lignite) is distilled in much the same way for tar and ammonia.

In 1681 an English patent was issued for the making of tar and pitch from coal; in 1694 the making of "pitch, tar and oyle out of a kind of stone" was protected by a patent. The first commercial plant for such work appears to have been established in Sunderland, England, and produced "petroleum" and ammonia from coal as early as 1815.

In France the distillation of shales began about 1830, and has maintained the rank of an industry ever since.

Though the distillation of oil from shales and shaly coals has been carried on in many countries, in no other has the industry reached the magnitude or the success to which it has been carried in Scotland, and today the Scotch process is standard the world over. Previous to the development of the Pennsylvania oil fields there were between fifty and sixty plants making oil from cannel coal, cannel shales and bituminous shales in the United States. The manufacturers used the Scotch process and paid the Scotch patentees a royalty. These plants were located in Massachusetts, New York, Pennsylvania, Ohio, Kentucky, Virginia, Connecticut, and West Virginia. In Canada several shale oil plants were in operation at the same time. When the abundance and cheapness of petroleum products made the shale oil industry unprofitable, many of the American shale oil plants were converted into well oil refineries.

At the present time there are seven plants in Scotland handling a total of about 4,000,000 tons of shale per year.

The lamp oil of a few years ago was called coal oil, because of the fact that at first it was derived almost exclusively from low-grade coals. But later the distillation of shales yielded a similar oil. The name coal oil was retained and was handed down to the lamp oil obtained from petroleum.

RETORTING

The experimental work in retorting was carried on with the following aims in view:

(1) The most favorable temperature to secure the largest yield of the best oil, keeping always in mind the problems of the refiner.

(2) The possibility of securing valuable by-products without sacrificing the cardinal aim of the shale oil industry—to secure *oil*.

(3) Economy of operation as to fuel, speed of throughput and construction.

The apparatus consisted of:

(1) Common pot retorts, such as are used for mercury distillation, fitted for the use of superheated steam if desired, and provided with thermometers in wells, for accurate records of temperature.

(2) Horizontal pipe retorts fitted as above.

(3) Inclined and horizontal pipe retorts arranged to rotate.

These were all small and capable of holding charges up to about ten pounds of crushed shale.

(4) Condensers, ammonia and oil scrubbers, gas collectors, gas meters, absorbers, etc.

(5) A vertical cylindrical retort having a capacity of 75 pounds of crushed shale.

DESCRIPTION OF PLANT

The principal units of this larger plant are: (A) the steam unit; (B) the retort unit; (C) the condensing unit, and (D) the scrubbing unit. (See the figures.) A description of each unit, in order, follows:

The Steam Unit.—This consists of a boiler (1) equipped with a water-gauge, (2), a pressure gauge, (3), a pressure relief valve, (3a), and two heaters (4) and (5). The boiler (1), which is an ordinary water tank tested to 250 pounds pressure, and having a capacity of about ten gallons is placed on a small iron stand (6) and is supplied with water through pipe (7), equipped with drain-

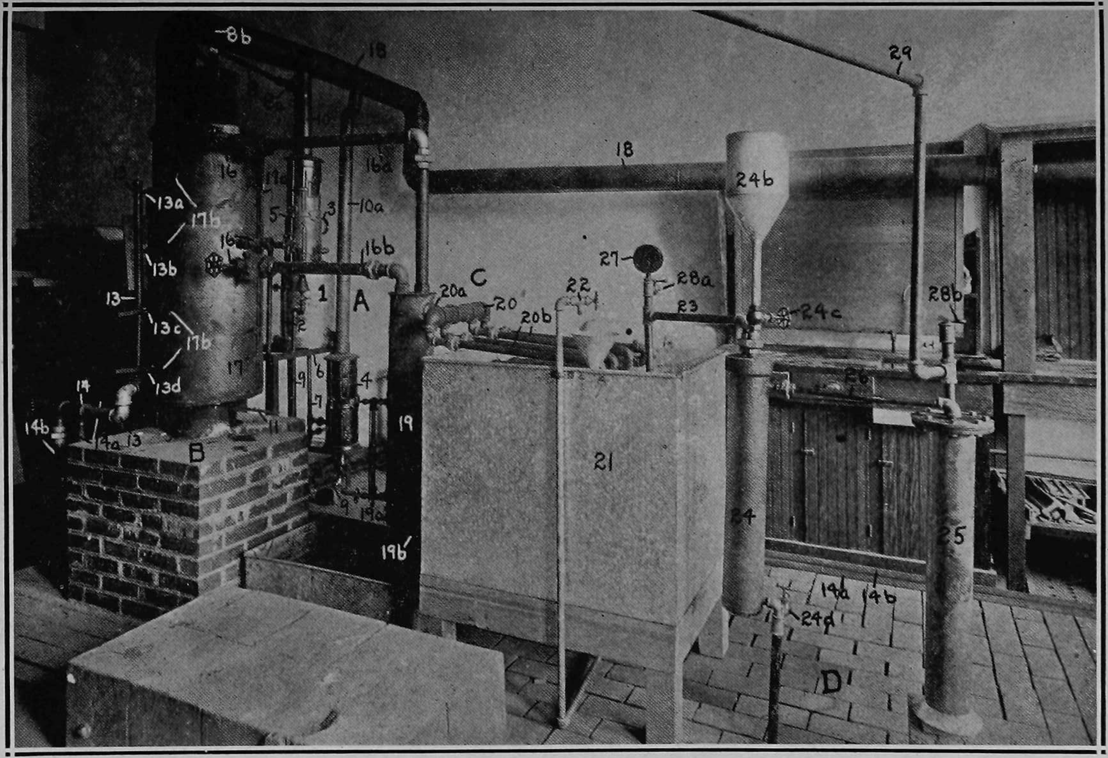

Fig. 8. Side view of eduction plant.

cock (7a). Heater (4) is used to raise the steam, while heater (5) is used as a super-heater, the steam passing out through pipe (8) into the super-heater and through valve (8a) into the retort. The heaters are ordinary "Lion" gas water-heaters (4) being size 1½, while (5) is size 1. Both are supplied by pipes (9) with gas. The copper coil of heater (5) is covered with graphite paint to prevent burning out, since the coil contains only steam and air. The products of combustion in the heaters are led to the main stock pipes (18) by pipes (10) and (10a).

The Retort Unit.—This unit consists of the retort proper, with the necessary heating apparatus, outlets for the gases formed, and control valves, most of which is enclosed in the shell (17). The relative positions are shown in figure 9 by the dotted lines. The retort proper (12) was made by rolling a piece of ¼″ iron into a frustum of a cone, six inches in diameter at the top, seven inches in diameter at the bottom, and forty-five inches high. The retort was then butt-welded and flanges welded on at the top and bottom. The bottom was closed by screwing on a plate which had four circular apertures in it. On the inside of this plate is a similar plate, which, however, is not screwed to the retort, but is rigidly fastened to a lever (11) by means of which one can change the relative positions of the two plates, and thus the retort can be emptied by bringing the holes together, or can be sealed by turning the lever (11) so that the holes are not coincident. The top is closed by means of a plate fastened on by set screws, and through which the steam line (8) is run. This steam line runs to within six inches of the bottom of the retort, and the steam is diffused by means of a rosette. Fitting (8b) is a gate valve, through which the thermo-couple of a pyrometer can be inserted if it is desired. The inside of the retort is perfectly plain.

The retort is heated by means of four circular burners (13a, b, c, and d) made of ¼″ pipe, drilled, and bent in the form of a semi-circle. One of these burners is placed on each side of the retort, at four different places, one about three inches from the bottom, and the other three at intervals of twelve inches above this. The burners lead off from a common gas supply pipe (13), which is in turn supplied with gas by pipe (14a), and with compressed air by pipe (14b). In fitting (14) is a cone with a small aperture, through which the air is forced under pressure, and the suction created by this jet tends to draw in the gas and form a perfect mixture for combustion. Each burner is protected from the burner below by thin iron shields being placed around them.

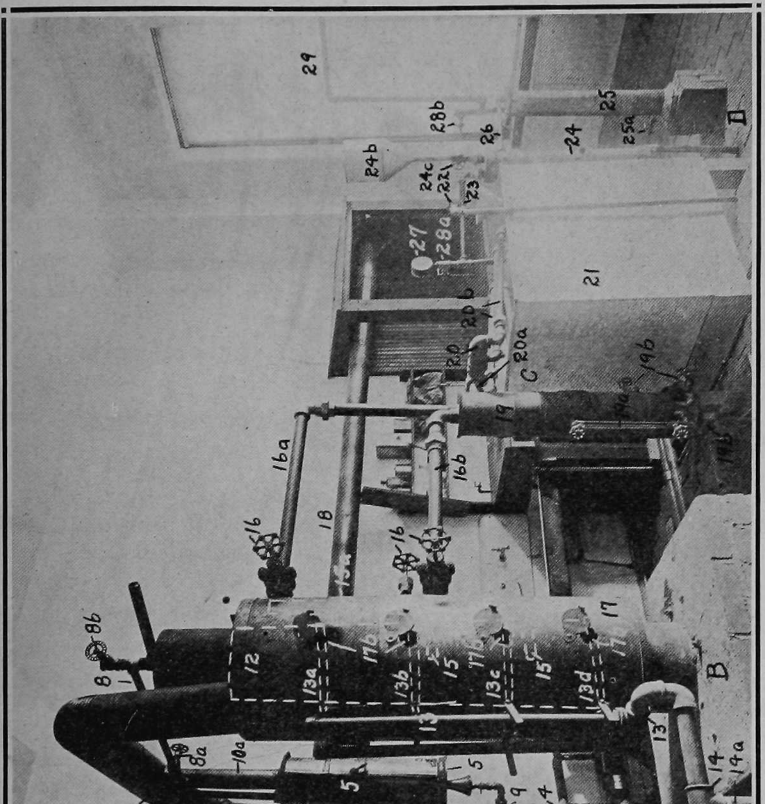

Fig. 9. View of education plant from retort end.

The vapors formed during distillation are led off by means of four ¾″ pipes, welded to the sides of the retort, two on each side and staggered, the first on one side being three inches from the top, and the second twenty-one inches from the top. On the other side, the highest one is twelve inches from the top, and the other thirty inches from the top. These pipes are designated on the photographs as (15). They are curved to meet in a common main (15a), which is a 2″ pipe standing parallel to the retort. This main, then, discharges into either of the 2″ take-offs (16a or 16b), according to the way in which the valves (16) are adjusted.

The retort proper, with the burners, shields, vapor outlets, and vapor mains, are all enclosed in a shell (17) made up of two thin sheets of iron with a sheet of asbestos between them, and provided with a door (17a) and peep-holes (17b), through which the burners can be lighted and their behavior observed. The products of combustion from the burners are led off through the pipe (18) and into the stack by means of a slight suction in the stack. The whole unit is set upon a brick foundation and is provided with an ash pit to care for the spent shale.

The Condensing Unit.—The condensing unit consists of two condensers, viz.: an air condenser (19) and a water condenser (21). The air condenser (19) is a sheet iron cylinder, six inches in diameter and thirty-four inches high. It is provided with a sight-glass (19a) and with drain cocks (19a) set at different levels. The pipes (16a and 16b) lead nearly to the bottom of the condenser, and the vapors are then allowed to rise in the cylinder, while any part which will condense collects at the bottom of the cylinder, and is drawn off. The condensation is caused by the temperature of the surrounding air, no cooling water being used in this case. The vapors not condensing here are drawn off through the pipe (20a) into the header (20), where they are divided through four ¾″ coils (20b), which are immersed in running water, supplied by faucet (22) and taken off by siphon (22a). The four coils lead into a common receiver (21a), which is a small iron tank eight inches in diameter and twenty-four inches long, immersed in the tank (21). This receiver has a drain cock (21b) by means of which the liquid collecting in (21a) can be drawn off.

The vapors and gases still uncondensed are led off by pipe (23) to the next unit.

The Scrubbing Unit.—This unit consists of the oil scrubber (24) and the ammonia scrubber (25). The oil scrubber (24) is a cast iron cylinder four inches in diameter and twenty-eight inches

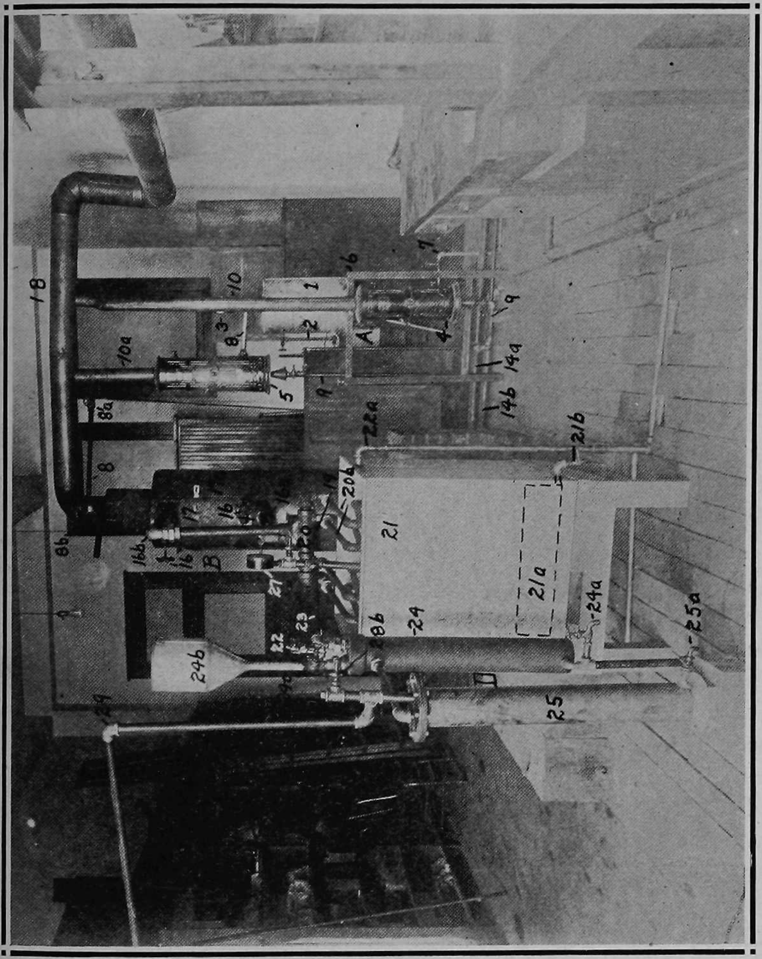

Fig. 10. View of eduction plant from scrubber end.

high, having a drain cock (24a) at the bottom and equipped with an oil reservoir and feed (24b). The cylinder is filled with pebbles, and the pipe (23) leads nearly to the bottom of the cylinder. The gases then bubble up through the pebbles, meeting the oil from (24b) as it trickles down over the pebbles. This oil absorbs any gasoline which may be left in the fixed gases. The gases are led into the next scrubber by the pipe (26), which, as before, leads nearly to the bottom of the scrubber (25). This scrubber is four inches in diameter and thirty inches high. It is made of sheet lead, and, while designed for an ammonia scrubber, it may be used as an auxiliary oil scrubber. For use as an ammonia scrubber it is partly filled with pebbles and a dilute solution of sulphuric acid used as the scrubbing medium. This scrubber is equipped with a drain cock (25a) and with a tube through which the tank can be filled. On this tube is placed the vacuum gauge (27). The stop cocks (28a and b) are used to test the suction and also to sample the gases at those places.

The gases from the last scrubber are led off through pipe (29) by means of a small suction pump, and are wasted up the stack. This small suction pump is connected directly to the series of pipes, and by means of it a slight vacuum is maintained in the system constantly.

Matthew J. Wilson made a large number of retortings under the direction of the writer, using a small pot mercury retort connected with a steam superheating device on the one side, and a water-cooled condensing coil and a series of receivers on the other. The retort was fitted with a thermometer placed in a well, and the temperatures were watched, controlled and recorded. The uncondensed gases were passed through an ammonia scrubber, and a gasoline scrubber to trap the last of the ammonia and any light condensable hydrocarbons which might pass the main condenser. In the first series no steam was used.

The crude oils recovered were fractionated, and the gasoline and kerosene were analyzed for unsaturated hydrocarbons. The results of the work are presented in a thesis which may be consulted at the University Library at Boulder.

The conclusions he reached may be stated as follows:

The most favorable range of temperature for retorting Colorado shales is between 280° C. and 340° C. (536° F. and 644° F.), and the best temperature appears to be about 320° C., or about 608° F.

Crude oils obtained within this range of temperature yielded an average of 33.7% of straight run gasoline (cut at 460° F.), and 20.7% of kerosene (cut at 600° F). The gasoline fractions averaged 54% of saturated hydrocarbons and 46% of unsaturated.

The kerosene fractions averaged 44.2% saturated hydrocarbons, and 55.8% of unsaturated.

The ammonia yield was small. To secure the best yield of ammonia it would be necessary to raise the temperature very considerably.

The yield of gasoline in the scrubber averaged 6.4 gallons per ton. The absorbing oil was a light kerosene cut.

The gravity B of the crudes ranged from 26.2 to 37.1 B, and averaged 30.2 B.

The results of many tests with the big retort, both with and without superheated steam, point to a temperature ranging between 320° C. and 420° C. (608° F. and 788° F.) as the most favorable when quantity and quality of oil are both considered.

Another series of retortings was made by Wilson, using superheated steam. The results obtained differ so widely among themselves and from many others obtained in the Survey laboratory that one is forced to the conclusion that a part of the retortings are not representative. For this reason the conclusions which follow are based upon those retortings which appear consistent with the great body of results previously obtained by steam distillation.

(1) The yield of crude is higher.

(2) The yield of ammonia is nearly double.

(3) The saturated content of the gasoline fraction is higher, averaging 63.3%.

(4) The kerosene yield is larger, but the gasoline yield is smaller.

(5) The saturated content of the kerosene is higher.

(6) The gasoline recovery in the scrubber averaged 9.3 gallons per ton.

(7) In general the crude is better than that resulting from dry retorting.

A series of tests was made in the large retort, using a general running temperature of 410° C. (770° F.), and after practically all the oil was taken off at this heat, the temperature was raised to over 600° C. (1,112° F.), and superheated steam was added in large volume. The purpose was to get all the ammonia and to convert the fixed carbon into fuel gases. The results were highly satisfactory. The ammonia yield was increased from 19.5 pounds per ton to 25.2 pounds per ton. The fixed carbon was almost entirely converted into gases. The spent shale was a pale ash-gray, showed no tendency to cohere in masses, and ran from the retort without the slightest clogging. At temperatures below 1,400° F. in the presence of superheated steam, some of the Colorado shales begin to mass by fusion. This causes more trouble than does coking.

The fuel gas obtained was a fair grade of water gas having a fuel value of 330-340 B. t. u. per cubic foot. The volume and fuel value will depend upon the fixed carbon present in the shale and the volume of steam used.

FORMS OF RETORTS IN COMMERCIAL USE

The vertical shaft retort, consisting of a cast iron or steel upper section and a firebrick lower section, is almost universally used in Scotland. Recently, two or three types of retorts resembling certain forms used in carbonizing coal, have been tested and put into use. The patent records would lead one to the conclusion that every conceivable modification of furnace or retort design had had its advocates, and that there can henceforth be nothing new (in this line) under the sun, but the mill still grinds, and the "best" appears every day.

LOCATION OF RETORTING PLANTS

The considerations which will determine the location of retorting plants are: Water supply, available sites, dumping ground, refinery possibilities, pipeline facilities, transportation and others.

In a shale oil plant water is used for the following purposes: The generation of steam for power, the making of superheated steam for retorting purposes, condensing vapors both in retorting and refining, and for steam distillation. It is estimated that the total use of water in these ways in the Scotch plants exceeds a ton of water for each ton of shale put through. But the actual consumption of water probably falls far short of this figure, since much of it is recovered and used over and over again. Even if the consumption is but one-third the use, the problem becomes a formidable one in a semi-arid region.

The shale fronts lie far back from the rivers. Many of the creeks flowing to the rivers head far back in the shale area. But most of them are intermittent and are dry in the midsummer, and frozen up and nearly dry in the winter. Even if their waters were not appropriated for irrigation purposes they would be a very unsafe dependence for a large industry. The only reliable source of waters is the rivers.

The question naturally arises as to whether it would be better to bring the shale by gravity to the water or to take the water to the shale against gravity. The logical place for refineries is along the rivers and along the railways. Shale oil will not move easily through pipe lines. The narrow canyons which cut into the shale beds do not furnish the best of dumping grounds.

To build a refinery at and for every retorting plant would be a very wasteful procedure, since a single large refinery could take care of the crude produced by several large retorting plants, thus reducing overhead and operating expenses. The building and maintenance of railways in the narrow canyons would be attended by many difficulties.

Plant sites affording water, dumping room, storage room and all the requirements of a large industry are not plentiful in the canyons.

FIXED GASES FROM RETORTING

In discussing the question of the fixed gases formed in the process of retorting, both the quality and the quantity must be considered. In general, it may be said that the total fuel value of the fixed gas from a ton of shale of given oil content would afford the best ground upon which to judge of the efficiency of the retorting in this regard. But, as will be shown later, this method may be very misleading.

The volume and quality of fixed gases formed will depend upon several factors, such as: The quality of the shale, the use or non-use of superheated steam, the admission or exclusion of air, the temperature of retorting and other conditions.

Other things equal, the richer the shale the larger will be the yield of fixed gases, since they come mainly from the organic matter of the shales. The addition of superheated steam may result in the dissociation of a certain amount of water into hydrogen and oxygen. Some of the oxygen may be used up in oxidizing carbon, and there will be an excess of hydrogen. It is even possible that a certain small measure of hydrogen may combine in the process of hydrogenation of unsaturated hydrocarbons, but it is much more probable that hydrogen will be freed in the process of cracking heavier hydrocarbons into lighter forms, as in the cracking of petroleum.

The admission of air to the retort will mean the addition of oxygen and nitrogen to the fixed gases. Some of the oxygen may be used in forming carbon dioxide and carbon monoxide.

The temperature at which the gases are driven from the shales is one of the most important factors in controlling the formation of fixed gas. It is possible to convert the entire hydrocarbon-forming content of the shale into the fixed gas methane and solid carbon. This is an extreme case. It is also possible by retorting at too high temperature to crack much of the hydrocarbon gases formed into the fixed gas methane and a condensable gas.

In Scotland it is customary for operators to convert a large part of the fixed carbon of the shale into carbon monoxide in much the same way as water gas is formed in commercial gas making. After the main part of the retorting is done the impoverished shale is heated to a high temperature to secure the larger yield of ammonium sulphate. Steam is driven or drawn through the heated shale and the oxygen of the steam unites with the carbon, forming carbon monoxide and carbon dioxide. A part of the hydrogen of the steam unites with the nitrogen of the shale, forming ammonia, and the remainder passes on to the condensers. In this way the volume of fixed gases is greatly increased, and much fuel is formed for retorting purposes.

In general (unless the fixed carbon is converted into gaseous fuel as just mentioned), it may be said that the lower the total fuel value of the fixed gases in proportion to the oil content of the shale, the more efficient is the retorting process, and the higher the fuel value of the fixed gases in proportion to the oil content, the less efficient is the retorting.

It is sometimes argued that fuel is needed for further retorting and that it is profitable to make it in the process of retorting. This is entirely wrong, since a retorting practice which results in the formation of a gas of high fuel value is bad from other standpoints and yields a poor grade of oil.

If it is desirable to secure fuel from the shale, it would be much better to install a producer gas plant, using shale for the generation of producer gas, and piping it as needed to the retorts.

In judging the significance of the fixed gas formed in any retorting process it is necessary to know the oil content of the shale and the volume and fuel value of the fixed gases formed. For example: Two operators may be retorting the same shale. One may get a large yield of fixed gas of low fuel value. The other may get a small yield of fixed gas having a high fuel value. Referring to the following table of fixed gas analyses: There is a considerable range in the fuel value per cubic foot of the gases, but unless the oil content of the shale and the volume of the fixed gas formed are known, it is impossible to say which retorting was the more efficient from the standpoint of loss by the formation of fixed gases.

To illustrate: Sample 5 of the following table was from a shale which yielded 50 gallons of oil per ton, and the total yield of fixed gas was 4,586 c. ft. per ton. The total fuel value of the fixed gases per ton was 4,586x322.30=1,478,067.8 B. t. u., or at the rate of 29,561 B. t. u. per gallon of oil content. Sample 6 was from a shale which contained 19 gallons of oil per ton, and the total yield of fixed gas was 3,658 c. ft. per ton. The total fuel value of the fixed gas per ton was 515,641.4 B. t. u., at the rate of 27,139 B. t. u. per gallon of oil content. From the standpoint of fixed gas formation the retorting of sample 9 was more successful than was that of sample 6. However, it must be stated that neither sample shows highly efficient retorting. It is quite possible to reduce the fuel value of fixed gas formed to the rate of 15,000 B. t. u. per gallon of oil content of the shale. The methane content should be lower in both these samples.

ANALYSES OF FIXED GASES FROM RETORTINGS

	Gal. oil per ton (2000)	Gas Volume per ton	Carbon Dioxide	Illum. Gases	Oxygen	Carbon Monoxide	Methane	Hydrogen	Nitrogen	B. t. u. per Cubic Foot
1. Colorado shale	63.0	......	14.2	3.8	0.8	0.9	22.7	41.2	16.4	462.9
2. Colorado shale	63.0	......	32.3	1.0	3.4	0.6	6.7	34.9	21.1	213.8
3. Colorado shale	63.0	......	40.0	0.4	1.8	0.1	1.6	46.3	9.8	180.9
4. Colorado shale	......	......	11.7	4.8	2.7	1.7	28.2	32.2	18.7	466.6
5. Colorado shale	50.0	4,586	11.8	1.9	5.5	25.0	17.2	4.6	34.1	322.3
6. Colorado shale	19.0	3,658	41.2	0.0	4.4	17.5	10.2	0.0	26.7	168.3
[3]7. New Brunswick	40.1	......	28.67	1.33	0.0	5.06	11.02	52.92	0.0	319.8
[4]8. Scottish shale (Henderson retort)	......	......	0.0	2.19	0.0	0.0	34.2	64.44	0.0	630.34
[5]9. Scottish shale	......	......	22.08	1.38	1.18	9.77	3.70	55.56	6.33	293.0
10. Broxburn, Scotland. Survey laboratory, Boulder	28.5	2,960	13.0	3.5	2.5	2.1	21.3	39.4	18.2	441.3
11. Broxburn, Scotland. Survey laboratory, Boulder	28.5	......	12.4	3.1	2.4	2.5	28.8	24.5	21.3	561.2

The analyses here given were chosen for the purpose of showing the wide range of composition of the fixed gases formed in retorting shales.

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[3] Ells, R. W., Bituminous or Oil-Shales of New Brunswick and Nova Scotia, etc. Canada Dept. Mines, No. 55, 1909, p. 16.
[4] Redwood, I. I., Mineral Oils and their By-Products, 1897, p. 250.
[5] Jour. Soc. Chem. Ind., 1897, p. 983.

The question is often asked as to whether, in the process of efficient retorting, the yield of fixed gas will be sufficient to carry on the retorting. Unquestionably it will not. It is only a matter of arithmetic to show the insufficiency of the heat from this source. But the conversion of the fixed carbon into carbon monoxide will also play a part. However, it is reasonably certain that a considerable amount of other fuel will be necessary. This may be obtained, as already suggested, from shale, or it may be got from coal. The Scotch use coal to supplement the fuel formed in the process.

THE NITROGEN OF THE SHALE

AMMONIA YIELD

A series of analyses made in the Survey and University laboratories showed an average nitrogen content in the Colorado shales of 0.60%. A similar series of analyses made by the U. S. Geological Survey[6] gave an average nitrogen content of 0.64%. But the nitrogen content is not at all regular. A few samples of rich shale had a nitrogen content exceeding 2.00%, while other shales containing from 30 to 40 gallons of oil per ton contained from 0.35% to 0.50%. If all the nitrogen of the shale were recoverable in the form of ammonium sulphate, the yield from shales running 0.6% nitrogen would be 56.7 pounds per ton. That from shale containing 0.64% nitrogen would be 59.4 pounds per ton.

The dry retorting of the shales as shown by scores of tests fails to give an ammonium sulphate yield comparable to the theoretical amount. The average nitrogen content of the shales used in making the tests recorded in the tables on p. 69 was 0.60%, or equivalent to an ammonium sulphate yield of 56.7 pounds per ton. The actual average recovery from the gases by dry distillation was 4.62 pounds, or 8.15% of the theoretical possibility. By steam distillation (with gypsum), the average recovery from the gases was 17.93 pounds per ton, or 33.7% of the theoretically possible.

Analyses of the oils show that a fairly large part of the nitrogen passes over into the oil mainly in the form of pyridine compounds. Four determinations at Boulder gave an average content of pyridine compounds of 3.74%. However, the number of such determinations was too small to give a safe basis for a generalization. A series of fifteen analyses made by D. T. Day for the U. S. Geological Survey[7] shows an average pyridine content of 3.93%.

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[6] U. S. Geol. Survey Bull. 641, p. 159, 1916.
[7] U. S. Geol. Survey Bull. 691, p. 48, 1918.

The question arises: Is the larger yield of ammonium sulphate obtained by steam retorting due to the breaking up of pyridine compounds? This might be answered by comparing the pyridine content of oils obtained by steam and by dry distillation from the same shale.

Dungan tested samples of shale gasoline for pyridine and got negative results.

In the case of most of the Scottish shales the ammonia yield is larger from those poor in oil than from the higher grades. There appears to be no such relationship between ammonia content and oil content in the Colorado shales.

Analyses and other tests show that the Colorado oil shales contain carbonates of calcium (lime), iron and magnesium. Of these, by far the most important is the calcium or lime carbonate. Occasionally one may find a stratum which may be called a calcareous oil shale, but in the greater body of the shale the carbonate content is too small to justify the name. It is a noticeable fact, however, that the ammonia water collected in the oil reservoir is almost always somewhat carbonated, and from some shales as much as 25 per cent of the ammonia may be recovered as carbonate. It is even possible to cause the ammonium carbonate crystals to form on the walls of the condensing chamber above the oil.

Ammonia also passes over in the form of the hydrate to such an extent as to give the water a strong odor. In the laboratory the easiest and most convenient method of collecting the ammonia is by passing the gases containing it through a sulphuric acid solution in a tall bottle containing beads, coarse sand or small pebbles. The beads break up and distribute the gases and complete contact with the acid is secured.

In the larger apparatus in the Survey laboratory a pebble-filled lead chamber three feet high forms an effective ammonia scrubber.

PHENOLS

All true shale oils, as well as oils from lignitic shales, contain phenolic compounds, and may be regarded as a possible source of carbolic acid. But there is serious doubt as to the quantity being sufficient to warrant an expensive installation to save them, as they occur so much more plentifully as a by-product in coking. Six samples of shale kerosene and six samples of the skimmed oil were analyzed for phenols, but no analyses of the crude oil were made. The results are given in the following table:

		Phenols in Kerosene	Skimmed Oil
8	Parachute Cr.	1.10%	0.54
14	Parachute Cr.	1.60	0.76
16	Parachute Cr.	1.66	0.77
17	Parachute Cr.	1.70	1.05
18	Parachute Cr.	2.00	1.35
19	Parachute Cr.	1.92	0.77
	Average	1.66%	0.87%

POTASH IN THE SHALES

Practically all shales contain a small amount of potash derived largely, and in many cases wholly, from the igneous rocks from which the shale material originally came. It is possible that a small amount of the potash of some shales comes from imprisoned marine waters. In some instances land plants may have contributed to the potash of certain shale strata, but as the potash so derived is largely or wholly water-soluble, it would be carried away by ground water.

The fossil plants of the oil shale beds are largely algae, and are not likely to have contributed appreciably to the present potash content of the shales. Analyses of the raw shales show that the potash is what chemists call insoluble potash; that is, potash not soluble in water.

The high temperature to which the shales are subjected in the retorting process renders a part, possibly all, of the potash water-soluble. Analyses of the spent shale from Parachute and Conn creeks showed an average water-soluble potash (K_2O) content of about 0.9%, or about 18 pounds per ton of spent shale. The range of water-soluble potash shown by the analyses was from 0.25% to 2.02%. There is no apparent relationship between the oil content and the potash content.

As to the commercial possibilities of the potash: It is very easily extracted and it would seem possible that the spent shale could be deposited in dumping basins filled with water, and allowed to remain there a short time for the dissolving of the potash, and then dredged out or removed by belt conveyors to the final dump. When the water became highly charged or saturated with potash it could be drawn off into salterns or could be allowed to evaporate and deposit its potash in the basins. The process would be simple and inexpensive and would probably prove profitable in large plants. For example, a plant handling 1,000 tons of shale per day would deposit from 600 to 700 tons of spent shale which would yield, roughly, 10,800 to 11,600 pounds of potash. This is a possible by-product worthy of consideration.

The large ammonia yield of the shales, the possible potash yield and the proximity of the great phosphate beds of Idaho, Utah and Wyoming suggest the possibility of an artificial fertilizer industry. The Salt Lake and Colorado smelters working on sulphide ores should be able to furnish an abundance of sulphuric acid to meet the needs of such an industry.

REFINING TESTS

The following is a brief summary of the results of a very large number of refining tests made in the Survey and University laboratories by and under the direction of the writer.

The purposes of the tests may be stated as follows:

(1) To find out in what respects the regular refining processes used for well petroleum would apply to shale oils, and in what respects they would have to be modified.

(2) To determine what refined products could be obtained from shale oil, and to compare them with the corresponding products from well petroleum.

(3) To reach well founded conclusions as to the commercial possibilities of the shale oil industry.

The apparatus used included:

(1) The regular glassware equipment commonly used in oil laboratories, consisting of flasks, condensers, heating devices, etc.

(2) A series of small tubular and box stills of iron and steel, condensers and treating apparatus.

(3) Cracking stills similar in form to the Burton commercial pressure still, and several others, including vertical stills with and without dephlegmators and arranged to condense under still pressure or under atmospheric pressure.

FRACTIONATION

Crude oil ranging in quantities from 100 cc. up to 5 or 6 gallons was fractionated in various forms of apparatus. Except for special experiments the usual cuts were made—crude gasoline at 392°-410° F., crude kerosene at 572° F. Beyond this, test cuts were made at various temperatures for lubricating oils, cracking distillates, asphalts, etc.

The remarkably close relationship between the methods of retorting and the results of fractionation was evident at every point.

In general, it may be said that the best results were got from crudes obtained by retorting at temperatures not exceeding 725° F. Such oils yield, rather uniformly, 15 to 20 per cent of crude gasoline and 30 to 35 per cent of crude kerosene. A very large part of the kerosene fraction comes over at temperatures between 392° F. and 465° F.

The Survey laboratory records show the results of about 750 distillation tests and probably over 1,500 tests and partial tests, in refining. The tables beyond give the results of tests which may be regarded as typical. They were made at different times, in different apparatus and by different operators.

	1	2	3	4	5
Gravity of crude, B.	26.8	27.0	27.0	27.2	26.4
Gasoline, per cent	15.4	15.0	15.35	15.3	16.8
Gravity of gasoline	51.0	51.0	50.5	50.5	50.8
Kerosene, per cent	32.3	32.5	31.0	33.3	30.7
Kerosene, gravity	36.0	36.0	35.5	35.0	36.2
Gas oil, per cent	23.1	22.5	26.0	20.0	18.4
Gas oil, gravity	33.5	34.0	33.0	34.0	33.8
Light lubricating, per cent	17.0	17.0	15.5	18.5	17.8
Gravity, light lubricating	30.0	30.5	31.0	30.5	31.2
Heavy lubricating, per cent	10.0	11.5	9.5	11.7	12.4
Gravity heavy lubricating	24.5	25.0	26.0	25.5	24.7
Coke and loss	2.2	1.5	2.7	1.4	3.9

	6	7	8	9	10
Gravity of crude, B.	30.7	31.2	26.9	32.2	29.4
Gasoline, per cent	18.9	20.4	14.8	21.1	18.2
Gravity of gasoline	52.1	53.0	51.1	53.6	51.6
Kerosene, per cent	30.0	27.8	35.0	25.6	28.2
Kerosene, gravity	37.4	37.6	38.0	37.7	38.0
Gas oil, per cent	18.9	14.4	13.0	20.4	19.7
Gas oil, gravity	34.1	30.3	30.7	30.6	32.8
Light lubricating, per cent	14.6	18.4	17.4	15.9	20.5
Gravity, light lubricating	30.4	31.0	31.2	30.2	30.5
Heavy lubricating, per cent	14.2	15.4	12.8	10.1	9.9
Gravity heavy lubricating	24.9	26.2	26.0	24.8	24.4
Coke and loss	3.4	3.6	7.0	6.9	3.5

UNSATURATED AND AROMATIC HYDROCARBONS

These tests were conducted according to standard methods, and the following summary presents in generalized form the results obtained:

(1a) Crude obtained by dry retorting at temperatures above 800° F. commonly contains from 60% to 75% of unsaturated hydrocarbons, and measurable percentages of such aromatic hydrocarbons and derivatives as benzol, toluol, phenols, and pyridine compounds. The crude is considerably cracked and the yield of gasoline is consequently higher than that from less cracked shale oil. Extremely high temperatures cause excessive cracking and the formation of an unduly large amount of fixed gas. The yield of crude is reduced thereby.

(1b) The gasoline fraction from such crude oil carries approximately the same percentage of unsaturated hydrocarbons. The average of fifteen samples was 68% of olefins. The aromatic substances do not appear in the gasoline in proportionate quantity. In fact, tests sometimes fail to show their presence.

The kerosene fraction commonly carries a higher content of unsaturated hydrocarbons than does the gasoline, and aromatic compounds may appear.

(2a) The crude from dry distillations at temperatures between 725° F. and 800° F. contains 50% to 60% of unsaturated hydrocarbons, but much less aromatic material, though strong traces are found. Some cracking has occurred, but the gasoline yield is smaller than that from the higher temperature retortings.

(2b) The gasoline and kerosene fractions are more nearly equal, and the residue after they are taken off is comparatively easily cracked and will add considerably to the total yield of gasoline. The percentage of unsaturated hydrocarbons ranges from 50 to 60.

(3a) Crude shale oil from retorting at temperatures below 725 gives the highest yield of gasoline and the lowest of kerosene. The unsaturated hydrocarbons often fall below 45 per cent, and are sometimes as low as 35 per cent. The distillate ranging in gravity from 26 to 32 B′ is good cracking material and yields readily at a pressure of 75 pounds per square inch, and a temperature of 755° F. to 825° F.

(4a) The crude from steam retorting at intermediate temperatures is the best. The percentage of light oils is commonly higher, the percentage of unsaturated and aromatic compounds is lowest, and the oil is more easily refined. But even in this the percentage of unsaturated hydrocarbons ranges around 35% to 40%.

The large percentage of unsaturated hydrocarbons (olefins and diolefins) in shale oil makes it necessary to adopt methods of refining differing widely from those used in refining well petroleum. The olefins yield a little more power than do the paraffins, and they deliver it in a steadier and more sustained stroke. They must be saved and used. To remove them would be a criminal waste, both of good motor fuel and of good sulphuric acid. The average content of olefins in shale gasoline and kerosene will be 50 per cent or over. To remove them would be to cut the gasoline and kerosene yields in half, and make the acid treatment so expensive as to be prohibitive.

Good, clean, sweet-smelling gasoline containing 60 per cent olefins is made on a fairly large scale in Scotland. It has been made in the laboratory of the Survey, but by methods which may not prove commercial. However, the obstacle to success was not the saving of the olefins. That is relatively easy. The difficulty lay in removing the coloring matter.

Unrefined shale gasoline and kerosene are dark brownish purple, and the coloring matter is very persistent. It appears to be an oxidation product, and its development is aided by bright sunlight. Samples of gasoline may be cleared to a very pale straw yellow, but if allowed to stand in an open vessel exposed to light and air they will darken to brownish or purplish red in a few hours. A thin purplish-brown film forms on the walls of the vessel and the deposition may continue for several days. This is readily soluble in alcohol. The color change takes place very slowly in well-filled bottles closely stoppered, and the film is scarcely perceptible.

The decolorization of oils is commonly accomplished by one or other of the following methods:

(1) The changing of the coloring substance into one which is soluble in, and may be removed by, one of the washing agents used in refining—sulphuric acid, sodium hydroxide (or sodium carbonate), or water.

(2) The absorption of the coloring matter by filtering material such as fuller's earth, boneblack, charcoal, clay, etc.

It should be noted that the coloring matter in the shale oils is closely associated with the non-paraffin matter of the oil, but whether it is an unsaturated hydrocarbon derivative or an aromatic derivative the writer is unable to state. When the entire non-paraffin matter of the oil is removed by acid and other treatment methods, a water white gasoline or kerosene is easily secured. But if the unsaturated hydrocarbons are preserved the coloration persists very stubbornly. Experiments appear to show that it is not due merely to the oxidation or other sub-atmospheric change of olefins, since several samples of gasoline rich in olefins have been kept for periods of several months to two years with no more coloration than commonly occurs in straight run paraffin gasoline. There are in the Survey laboratory samples of gasoline and kerosene over three years old. The gasoline may still pass for "prime white," while the kerosene is pale lemon yellow.

In the attempt to render the coloring matter removable by washing with acid, alkali or water, the following reagents were used: Hydrochloric acid, nitric acid, aqua regia, acetic acid, tannic acid, acid sodium carbonate, potassium dichromate, potassium permanganate, chlorine water, chlorine, zinc dust and acetic acid, potassium nitrate, stannous chloride, hydrogen peroxide, ferrous and copper sulphates and common salt, ferric chloride, sodium phosphate, lead acetate, ammonium molybdate, potassium ferrocyanide, potassium ferricyanide, potassium cyanide, ammonium hydroxide, dimethyl glyoxime, mercuric chloride, barium chloride, and others. The tests show that equally good results may be obtained with 2% of concentrated sulphuric acid and 4% of caustic soda solution having a gravity 14 B′.

Complete contact of the oils with the reagents was secured by agitating with compressed air, but it is doubtful whether the agitation was, in all cases, continued long enough. A desirable modification of the method would have been to agitate by mechanical means without introducing compressed air.

In carrying out the filtration tests the filter materials were used both separately and in various combinations. When two or more materials were used they were sometimes mixed, and sometimes used in separate layers. Both deep and shallow filters were used, and experiments were made by putting the filtering material in a flask with the oil and agitating for a considerable time. The filter materials used include: American and foreign fuller's earth, animal charcoal, wood charcoal, common salt, a variety of clays, spent shale, slacked lime, partially slacked lime, quick lime, diatomaceous earth, Portland cement, gypsum plaster, pumice and various others.

So far as the writer's own tests are concerned the best results were obtained with a stratified filter, composed of layers of clay from the Pierre shale, layers of wood charcoal and layers of common salt. By one such test an almost water-white gasoline was obtained which remained water-white at least eight months, when an admiring promoter of a shale oil company carried it away to show what he himself had obtained from his own shale. However, it is not certain that the filtration was entirely responsible for the results obtained, as the gasoline had gone through some of the chemical tests already mentioned.

A long fuller's earth and animal charcoal column gave through a small amount of very good looking gasoline, but the effectiveness of the filter declined rapidly. Quick lime appeared to improve the color temporarily, and other experimenters, including a former member of the staff of the U. S. Bureau of Mines, claims permanent results.

Redistillation tests were carried out in various ways, using filtering or absorbing materials in the flasks, and passing the vapors through such substances. The resulting gasolines were subjected to acid and alkali treatment. The following materials were used: American and English fuller's earth, animal charcoal, diatomaceous earth, caustic soda, caustic potash, solid phosphoric acid, anhydrous aluminum chloride, common salt, partially slacked lime, slacked lime, iron turnings, black copper oxide, powdered copper, litharge, barium chloride, and others.

These substances were used both alone and combined. The results obtained were no better than the results of repeated distillation without them.

It was thought by Dungan that the color might be due to furfuran or furfuraldehyde, but the usual tests for these substances gave negative results. He also tested for turpenes but got negative results.

In order to determine whether the color resulted from colloidal matter, the gasoline was subjected to an electrostatic stress of about 50,000 volts for one and one-half hours. The results appeared to show the absence of colloidal coloring matter.

Many tests and analytical determinations have been made, the results of which have but little bearing upon the problems of refining. For this reason they are not described.

Several grades of lubricating oils have been made, and comparative tests as to their value have been carried out with impartial care.

The lubricating fractions were treated with 4% of concentrated sulphuric acid, followed by 6% of caustic soda at a temperature of 94° F. The oil was then treated with fuller's earth and filtered hot.

The lighter lubricating oils were cherry red, but the heavier were almost black. Descriptions of the oils follow:

Light oil:

Gravity	31.0 B.
Flash	220° F.
Fire	240° F.
Viscosity	Saybolt
At 60° F.	53
At 100° F.	44.5
At 200° F.	34.6

Heavy oil:

Gravity	19.7 B.
Flash	448° F.
Fire	490° F.
Viscosity	Saybolt
At 60° F.	Too thick to test.
At 100° F.	3340.
At 200° F.	732.

Record of a test of the light oil on a Tinius Olson machine:

Journal-steel. Bearing-brass.
Area of bearing, 7 square inches.
Total load on bearing, 300 pounds.
Pressure per square inch, 42.9 pounds.
Revolutions per minute of journal, 800.

Time	Temperature of Bearing	Load	Friction	
			Total	Coefficient
4:53	78	300	35	0.117
4:56	98	300	7	0.023
4:59	110	300	4	0.013
5:02	114	300	4	0.013
5:05	120	300	4	0.013
5:08	123	300	4	0.013
5:11	124	300	4	0.013
5:14	124	300	4	0.013
5:17	124	300	4	0.013
5:20	124	300	4	0.013

Maximum Temperature, 124° F.
Minimum Coefficient of Friction, 0.013.

The following statements regarding the lubricating oils are quoted from a thesis by Q. R. Dungan.

The lubricating oils obtained from the shale were excellent. The light lubricating oil resembled so-called ''gas engine oil'' in gravity, and flash and burning points, but the viscosity was much lower. In the friction test, the oil gave a much lower coefficient of friction, and kept the bearing at a much lower temperature than is usually the case with heavier oils. The coefficient of friction of lard oil is 0.035, while this oil gave a minimum of 0.013, under the same conditions. The maximum temperature of the bearing in this test was only 124° F., while the ordinary lubricating oils usually allow a maximum temperature of about 160° F. This is a remarkable performance, especially in view of the fact that the load on the bearing was much greater than would be used with an oil so light as the one tested. The heavy lubricating oil would probably be called a ''heavy cylinder oil.'' On account of its high paraffin content it was nearly solid at 60° F., slightly below room temperature. Its viscosity, and flash and burning points would show it to be well adapted to cylinder lubrication. A small quantity was vigorously agitated with an equal volume of water at 100° F., and the resistance to emulsion also showed its excellent qualities. In the friction test, the coefficient of friction reached a minimum of 0.01, while the maximum temperature of the bearing was only 146° F. In comparing the shale lubricants with petroleum lubricants, the superiority of those derived from the shale is apparent.

The calorific value per pound of the gasoline by Junkers calorimeter was 18,660 B. t. u.

PARAFFIN WAX

Only a few tests were made to determine the quantity of paraffin wax per ton of shale of given oil yield. But these tests showed the close relationship between retorting methods and the wax yield. In general, it may be said that a yield of wax ranging from 2% to 6% of the crude oil can be secured by the usual commercial processes. The wax was not fully refined in the Survey laboratory, but a pale yellow product was obtained.

The Scotch wax is of very high grade, and commands a price considerably higher than that from well petroleum. The highest melting point wax on the market is that from Scotch shales.

ASPHALT

The percentage of asphalt in crude shale oil appears to vary with the methods of retorting. An oil produced in an internal combustion retort contained 5.6 per cent of asphalt, whereas, crude produced from the same shale in a closed retort using superheated steam contained 1.4 per cent of asphalt. Another retorting of the same shale without steam gave an oil containing 2.5 per cent of asphalt. It may be said that the average asphalt content of shale oil is between 2.5 and 3.5 per cent.

The answers to the problems proposed on p. 60 are as follows:

The ordinary methods of refining mineral oils must be greatly modified to meet the needs of shale oil refining. The most important modifications have been indicated.

The refined products obtainable from shale crude are: Gasoline, kerosene, light and heavy lubricating oils, paraffin wax, residual tars and cokes. The gasoline and kerosene will be hard to clarify. But in matters of use they will equal, if not surpass, those from well petroleum. The lubricating oils are superior to those from well oil. The wax is of superior quality. No satisfactory tests of the uses and value of the residual tars and coke have been made.

As to the commercial possibilities of the shale oil industry there can be no reasonable doubt. But it must be kept in mind that the happy-go-lucky methods of petroleum winning will spell ruin if followed in the shale oil industry. It is an industry demanding technical skill of a high order. Large expenditures of capital are necessary to success. It is anything but a gambler's enterprise, or a get-rich-quick proposition. It is essentially a manufacturing industry, and is destined to be the largest in Colorado.

THE CRACKING OF SHALE OILS

Cracking may be defined as the breaking up of heavier hydrocarbons into lighter hydrocarbons. (The real chemical process is by no means so simple as this definition would suggest.) The products of cracking are mainly lighter hydrocarbons and carbon or coke. The character and relative quantity of the products of cracking will depend upon the hydrocarbons subjected to cracking, and upon the temperature and pressure to which they are subjected. Thus, there may result only lighter hydrocarbons which are liquid at atmospheric temperatures, or these and gases, or liquid and gaseous hydrocarbons and residual carbon. The liquid hydrocarbons may belong to the saturated or the unsaturated groups. At low pressures unsaturated hydrocarbons are very likely to form. Saturated hydrocarbons are best secured by cracking at high pressure. A charge of Parachute Creek shale retorted at a pressure of 80 to 85 pounds per square inch yielded a crude oil containing 46.2 per cent of unsaturates, whereas the same shale retorted at atmospheric pressure gave a crude containing over 60 per cent of unsaturates.

Permanent gases and coke are accompaniments of all cracking, but the smaller the quantity of these the more efficient is the process.

The extraction of oil from shale is essentially a process of destructive distillation, and there can be no doubt that a large percentage of the unsaturated hydrocarbons in shale oil is due to cracking. The large percentage of hydrogen in the fixed gases is in keeping with this view. In some measure the percentage of "straight run" gasoline obtained from the crude depends upon the formation of light hydrocarbons by cracking in the retort.

In the Survey laboratory the writer directed two series of experiments in which the operators used the same shales but different methods of retorting. The results are interesting and instructive. Taking seven tests in each case the results stand as follows:

Series D

	Gallons per ton	Ammonium Sulphate, Pounds per ton	Gasoline Average	Kerosene Average
1	48	16.49	...	...
2	45	16.48	...	...
3	55	18.16	...	...
4	55	18.82	16.5%	40.0%
5	67	20.12	of the crude oil.	
6	55	17.17	...	...
7	57	18.29	...	...
Average	54.6	17.93		

Average yield of the two light fractions, 56.5%.

Series W

	Gallons per ton	Ammonium Sulphate pounds	Gasoline percentage	Kerosene percentage
1	28	3.27	48.0	19.0
2	46	3.87	32.0	19.5
3	34	5.94	31.0	21.5
4	36	6.22	31.0	20.0
5	48	3.82	40.0	23.0
6	31	4.21	38.0	22.0
7	20	5.00	35.0	22.0
Average	34.7	4.62	36.4	21.0

Average yield of the two light fractions, 57.4%.

In the first series raw gypsum was put into the retort to furnish superheated steam by the slow yielding of its water. In the second, dry distillation was used. The results speak for themselves.

With superheated steam the yield of crude was increased 20 gallons per ton, or nearly 60 per cent; the ammonium sulphate was increased 13.31 pounds per ton, or almost 300 per cent. The gasoline yield from the crude by dry distillation was more than double, while the kerosene was approximately half. These differences were due to excessive cracking in the dry retorting. The average percentage of light oils from the two crudes was about the same—56.5% by steam distillation and 57.4% by dry distillation. But when total results are compared a notable difference is apparent all along the line. Calling each charge one ton, we have the following comparison:

Retorting

	Steam	Dry	Difference
Crude, gallons	382.2	242.9	139.3 in favor of steam
Gasoline, gallons	66.9	88.1	21.2 in favor of dry
Kerosene, gallons	152.9	51.03	101.87 in favor of steam
Ammonium Sulphate, lbs.	125.51	32.34	93.17 in favor of steam

These examples are not regarded as showing average possibilities. They have been chosen from many similar comparative tests because they illustrate the points of difference between the results of the two methods of retorting. The average differences between the yields of crude oil by the two methods are much smaller. The relative differences in the gasoline and kerosene yields are representative ,and the notable difference in ammonia yield may be taken as fairly representative of what may be expected in practice. The steam-produced crude cracks more easily than the other, and the total available gasoline yield is higher.

CRACKING TESTS

In general, it may be said that shale oil cracks readily, if it is produced by proper methods of retorting. In our experience, well-made shale crudes yield from 14 to 20 per cent of the "natural" gasoline fraction, and the average may be placed at 16 per cent. Such a crude will yield a distillate which cracks readily at a pressure of 70 to 80 pounds per square inch, and a temperature of 720° F. to 770°F. The most satisfactory procedure seems to be as follows:

The gasoline and kerosene are taken off as usual. The residual oil is then run down in a coking still until only a soft asphaltic tar is left in the still.

If the yield of light oils has been about 50 per cent, by volume, of the crude, the distillate so formed should be about 20 to 24 per cent, by volume, of the crude. This oil cracks readily at a comparatively low temperature, yielding from 40 to 50 per cent of its volume of a gasoline equal in quality to the "straight run." In this way the gasoline yield is raised by about 12 to 15 per cent, or to approximately 28 to 30 per cent by volume of the crude.

Experiments in cracking the kerosene fraction were not quite so satisfactory. A series of experiments under a pressure ranging from 70 to 100 pounds, and a temperature ranging from 420° C. to 460° C. (788° F. to 860° F.), gave a yield averaging 17.7 per cent of gasoline of 51 Be′ gravity. Assuming that the kerosene fraction was 40 per cent of the crude, the cracking of the kerosene would add to the general gasoline yield about 3.5 per cent of the volume of the crude oil.

By these methods the total yield of gasoline would be as follows:

Straight run	16.0%
From kerosene	3.5%
From residue, say	13.5%
Total	33.0%

Better results were obtained by taking off the gasoline fraction and running the remainder down to a tarry residue. The distillate so formed would be about 60 per cent, by volume, of the crude. This cracks readily and yields, by ordinary cracking methods, about 40 to 50 per cent of its volume of gasoline. If it is cracked in two or three stages, allowing the still to cool, and removing the settled carbon, the yield may be brought up to 60 or 65 per cent of the volume of distillate used. By this process the yield of gasoline from shale oil would run about:

Straight run	16.0%
From distillate	27.0%
	43.0%

If the distillate is cracked intermittently the yield can be raised to:

Straight run	16.0%
From distillate	36.0%
	52.0%

The following summary records the results of a series of cracking tests made on shale kerosene:

Oil used cc.	500	500	500	500
Gravity B'	35.5	35.0	33.5	35.0
Gasoline yield cc.	69	73	100	105
Percentage yield	13.8	14.6	20.0	21.0
Gravity B'	52.0	51.0	51.0	51.0
Pressure used (pounds per sq. in.)	70.0	70.0	100.0	100.0
Maximum temp. C.	420	420	460	460
Loss per cent	5.0	8.0	8.0	7.0

The pressure ranged from 70 to 100 pounds per square inch, and the temperature from 785° F. to 860° F.

It is a recognized fact that kerosene is the hardest fraction there is to crack, whether well oil or shale oil is used. The results here shown are remarkably good. It will be noted that the yield of gasoline at the higher pressure and high temperature averaged over

40% higher than that at the lower pressure. Based on the percentage by volume of distillate the results are: For the lower temperature and pressure 14.2%; for the higher 20.5%.

A test of a distillate somewhat heavier than kerosene at a temperature of 815° F. and a pressure of 105 pounds per square inch gave a gasoline yield of 41% of the volume of the distillate.

It should be remembered that a gasoline composed largely of unsaturated hydrocarbons and having a gravity of 51 to 53 B′ vaporizes as easily as a gasoline composed of saturated hydrocarbons having a gravity of 58 B′ or higher.

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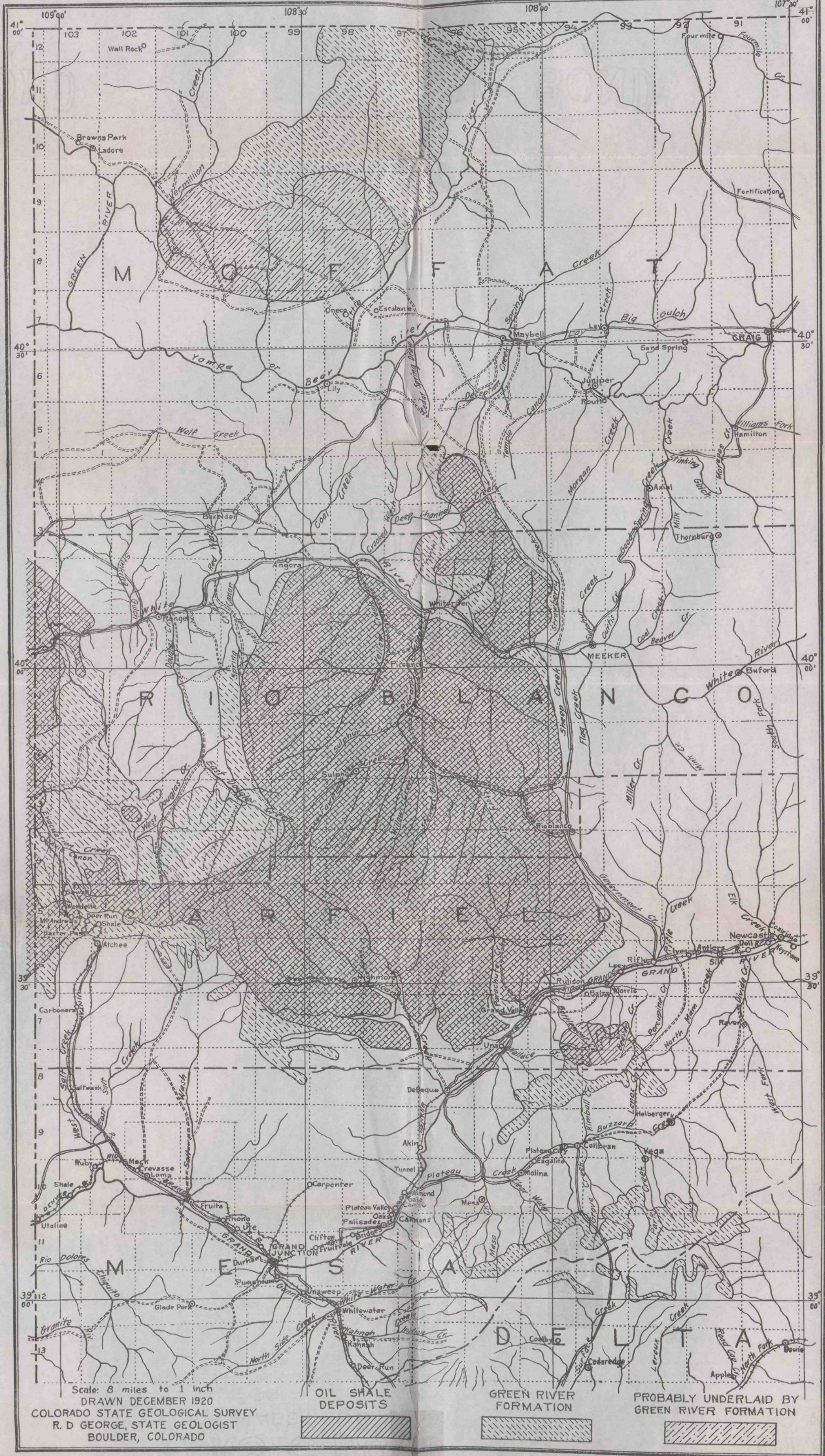
OIL SHALE DEPOSITS OF NORTHWESTERN COLORADO



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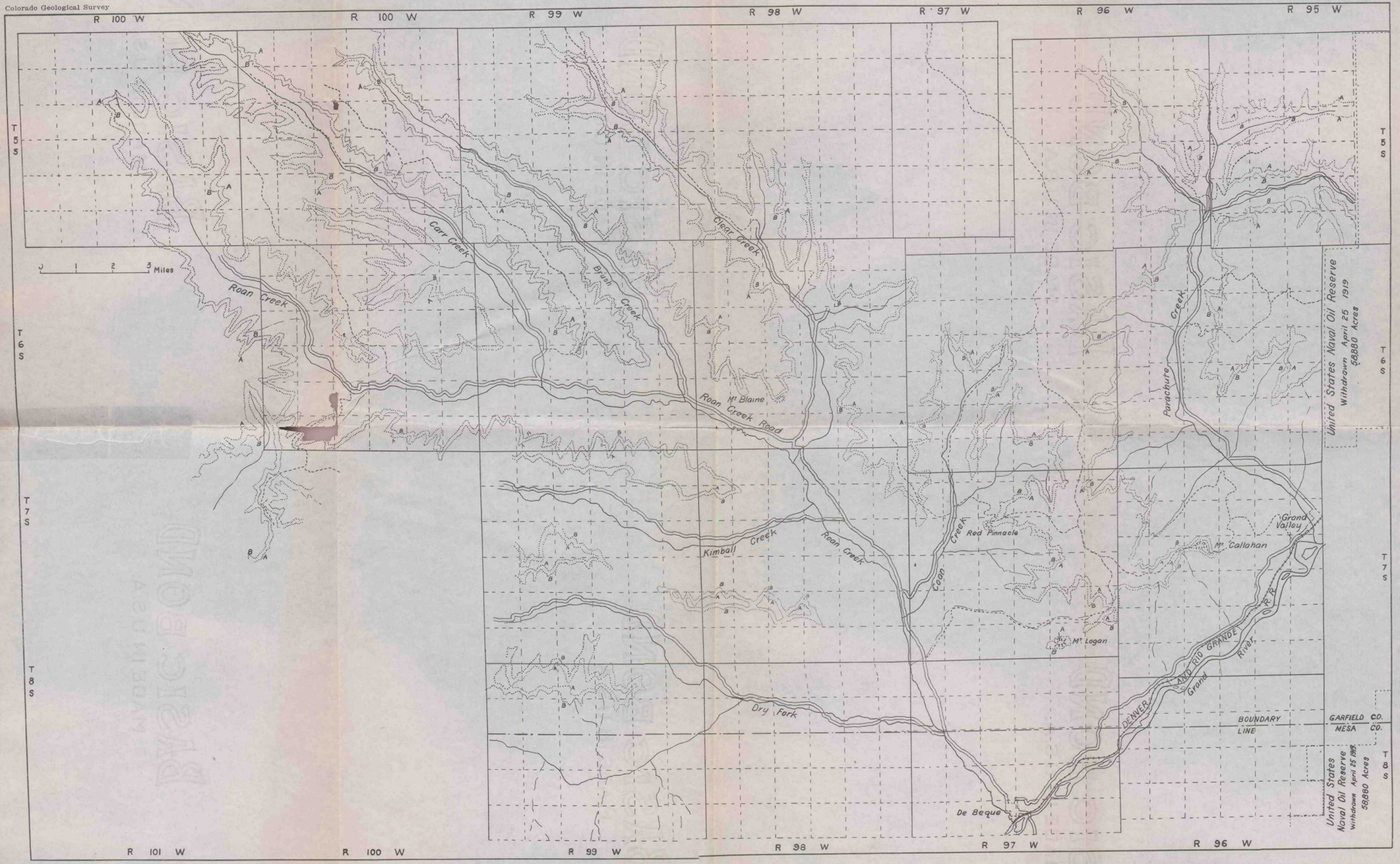
Colorado Geological Survey

Bulletin 25, Plate II



Oil shale map of part of Garfield County.