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# T H E S I S

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# SOME INVESTIGATIONS OF THE OIL SHALES OF COLORADO

BY

Q. R. DUNGAN

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**Presented to the Graduate Faculty in partial fulfillment**
**of the requirements of the degree of**
**Master of Science.**

UNIVERSITY OF COLORADO

1919

# PREFACE

The purpose of the work here undertaken, was, originally to study the characteristics of the oil obtained from the oil-shale of Western Colorado, and to compare its properties with those of ordinary petroleum. In order to do this it would be necessary to distill the shale and separate the resulting crude oil into the various products usually obtained, and to examine each one of these products separately.

However, it was not expected in the work here described, that it would be possible to investigate the whole problem of the commercial possibilities of the oil shales, as this is a problem requiring much more material, apparatus, and time, than was available. Consequently, the problem was narrowed down to an investigation of the products obtained by distilling the shale with a single given process, and in the treatment of the products, special attention was given to the gasoline fraction.

The writer has much pleasure in expressing his sincere thanks to Professor R. D. George, Director of the Colorado Geological Survey, for his many helpful suggestions and kind assistance in the problems met with, and without whose interest, the progress of the work would have suffered. The writer also gladly acknowledges the assistance rendered by Doctor J. B. Ekeley, of the Chemistry Department of the University of Colorado, by members of his staff, and by Professor J. A. Hunter, in charge of the State Oil Laboratory, in giving assistance and lending apparatus, when needed; and by other members of the faculty of the University, in giving expert advice on various subjects.

## REFERENCES

During the progress of the work, much of the modern literature on petroleum and allied subjects was consulted, and, wherever possible, specific reference will be made. However, a partial list of the sources of information is given below.

### BOOKS

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United States Geological Survey Bulletins

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## INTRODUCTION

A few years ago, the people of America were astounded by reports that the supply of available petroleum oils, especially gasoline, was fast being consumed, and that the end of the oil-well industry was in sight. Many of these reports were undoubtedly exaggerated, but Senate Document #310, sets forth the facts clearly and logically, and the conclusions arrived at, probably give the best insight into the actual state of the industry.

It is stated that the known oil-fields contain less than 7,629,000,000 barrels of oil, and that since the discovery of oil in 1859 to Jan. 1, 1918, there have been 4,200,000,000 barrels of oil produced in the United States. The production has risen from 2000 barrels in 1859 to 286,000,000 barrels in 1917. By a simple calculation, it can be seen that the oil-fields will be depleted in 26 years, at the present rate of production without allowing for any increase, which is bound to come wth the increased use of motor-vehicles and machines. Moreover,[1] during 1916 and 1917, there were taken from storage 43,000,000 barrels of oil, leaving only a six months supply above ground, for refining. To keep pace with the increased demand, the refining industry must be enlarged. Consequently,[2] during 1918, there were built118 new refineries bringing the number to 477, and increasing the refining capacity

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I. Morrell and Egloff, Met.&Chem. Eng., 18,11,601, July 1918

2. " " " vide.

from 40,000,000 to 490,000,000 barrels per year.

As the United States is, by far, the largest producer of petroleum, and since, according to many geologic experts, the chances for discoveries of new oil-fields are very slight, thisiis indeed a serious situation.

In looking over the possibilities for increased supplies of petroleum, the United States Geological Survey decided to investigate the oil-shale possibilities. Among other localities, the oil-shale area of western Colorado, and eastern Utah was worthy of note, and in 1913,[1] a party commenced work in this field. Previous to this time, however, a few large companies had investigated the possibilities and some had acquired holdings in this area. These private investigations were little known, but, nevertheless after Mr. Winchester and his party for the Unites States Geological Survey had made their report, a wild rush was made to western Colorado and a hunt for oil-shale was begun.

Needless to say, there were a great many companies organized to exploit the shale, but a large number of them were only stock selling schemes, and are not now doing business. A few still survive, but, as yet, actual field operations have not been started. A small experimental plant near Grand Junction, operating under the new Galloupe process, has been completed, and also one near De Beque, operated by the Mt. Logan Shale Company. Aside from these,

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[1] Oil Shale in North-western Colorado and Adjacent Area"
D.E.Winchester, U.S.G.S. Bulletin 641 F.

the writer knows of no other plants in this area. A great many laboratories in Colorado and Utah have been experimenting with the shales, as well as other laboratories in the East, and the United States Bureau of Mines is at present, contemplating the building of an experimental plant in the field, probably near Grand Junction.

A great many new patents have been issued or applied for recently, dealing with the treatment of shale, and, as is usually the case, many of them will probably be found worthless. For a little fuller discussion of the types of processes the reader is referred to Arthur J. Hoskins'article "The Winning of Oil from Rocks" in the "Mining and Scientific Press", 118,21,701, for May 24,1919.

Therefore, in view of these conditions, viz, the depletion of the supply of natural petroleum, and the interest and wild speculation in oil-shale, the writer was tempted to undertake the investigations here outlined, hoping that the results obtained might stimulate further investigation and influence to some degree such further development.

## SOURCES AND CHARACTERISTICS OF THE SHALE

The shale used in these experiments was taken from a large quantity which was sampled for the United Oil Company of Florence, Colorado, by Peter Lindauer of Grand Valley, and was obtained for this work through the kind assistance of Professor George. The places from which it was obtained are located along Parachute Creek and the "Book Cliffs" formation along the Grand River, north and west of Grand Valley, Garfield County, Colorado. This locality lies principally in Townships 6 and 7 S., R., 96 W., 6 P.M.[1], and is at the southern or south-eastern boundary of the Uintah Basin formation, where the shale out-crop is very prominent. Here the shale formation extends high above the valley floor, forming the top of the so-called "Book Cliffs".

The portion of shale used for the following tests was very uniform in composition, giving a nearly constant yield of between 60 and 65 gallons to the ton, when distilled in the presence of superheated steam. The appearance of the shale was massive, dark gray to black with a brownish tinge in color, and upon closer examination it was seen that it was made up of many small layers, which varied in color from a light gray, through brown to a dull black. Weathered edges were light gray or nearly white, and comparatively soft, but this weathering did not extend into the shale a greater distance than six inches, and seldom

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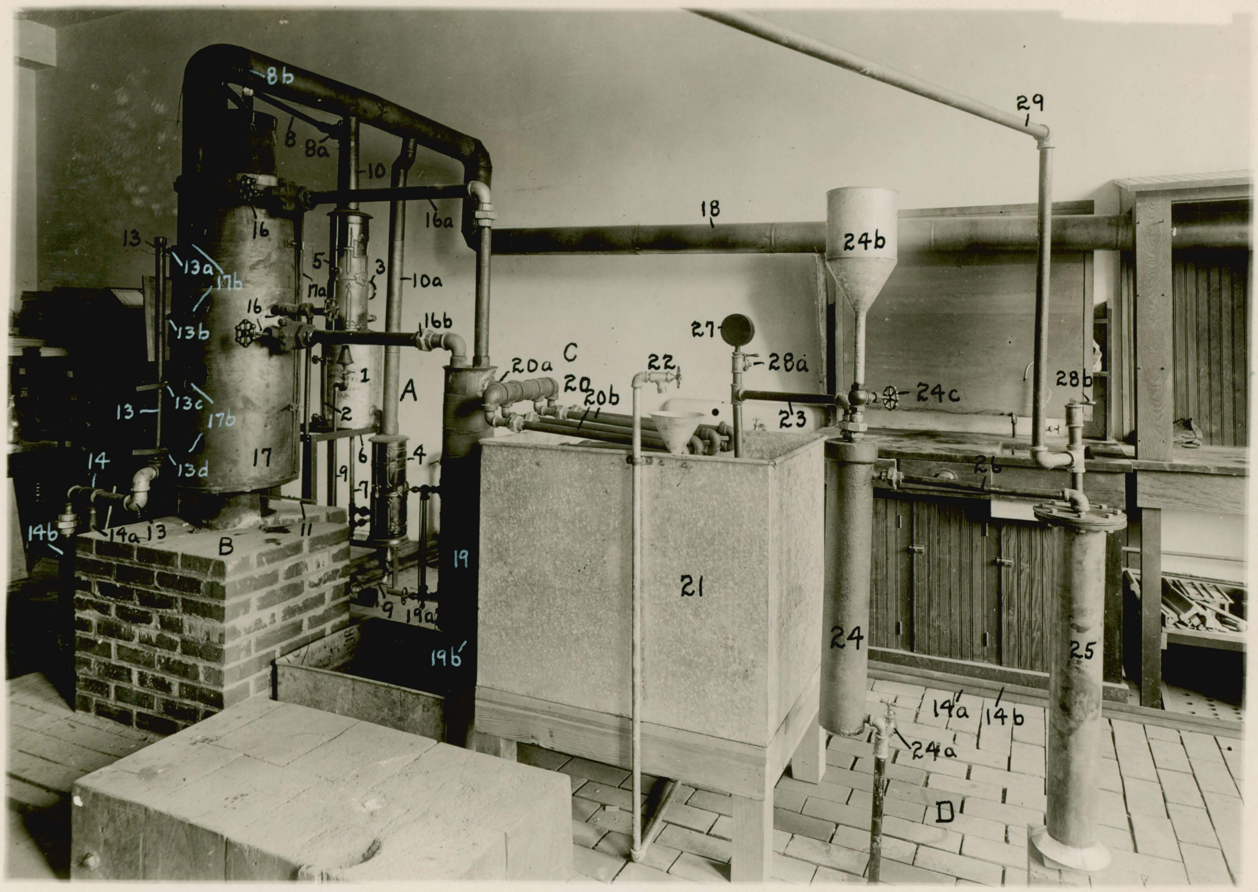
1. See plate XVIII, Bulletin 641 F, U.S.G.S. "Oil Shale in Northwestern Colorado and Adjacent Areas" D.E. Winchester

greater than three or four inches. In hardness, the unweathered shale is about 4, being easily scratched by a knife, and when so scratched, it shows a dull chocolate-brown, waxy, streak. Its specific gravity averages about 1.8. The shale is very tough and somewhat elastic, resembling a very hard rubber. When broken, it breaks across the laminations, and in many trials, it was found that not once did it break along the bedding planes. Its laminated structure, however, is brought out very clearly upon strong heating, after which it is very easy to divide the shale along these laminations.

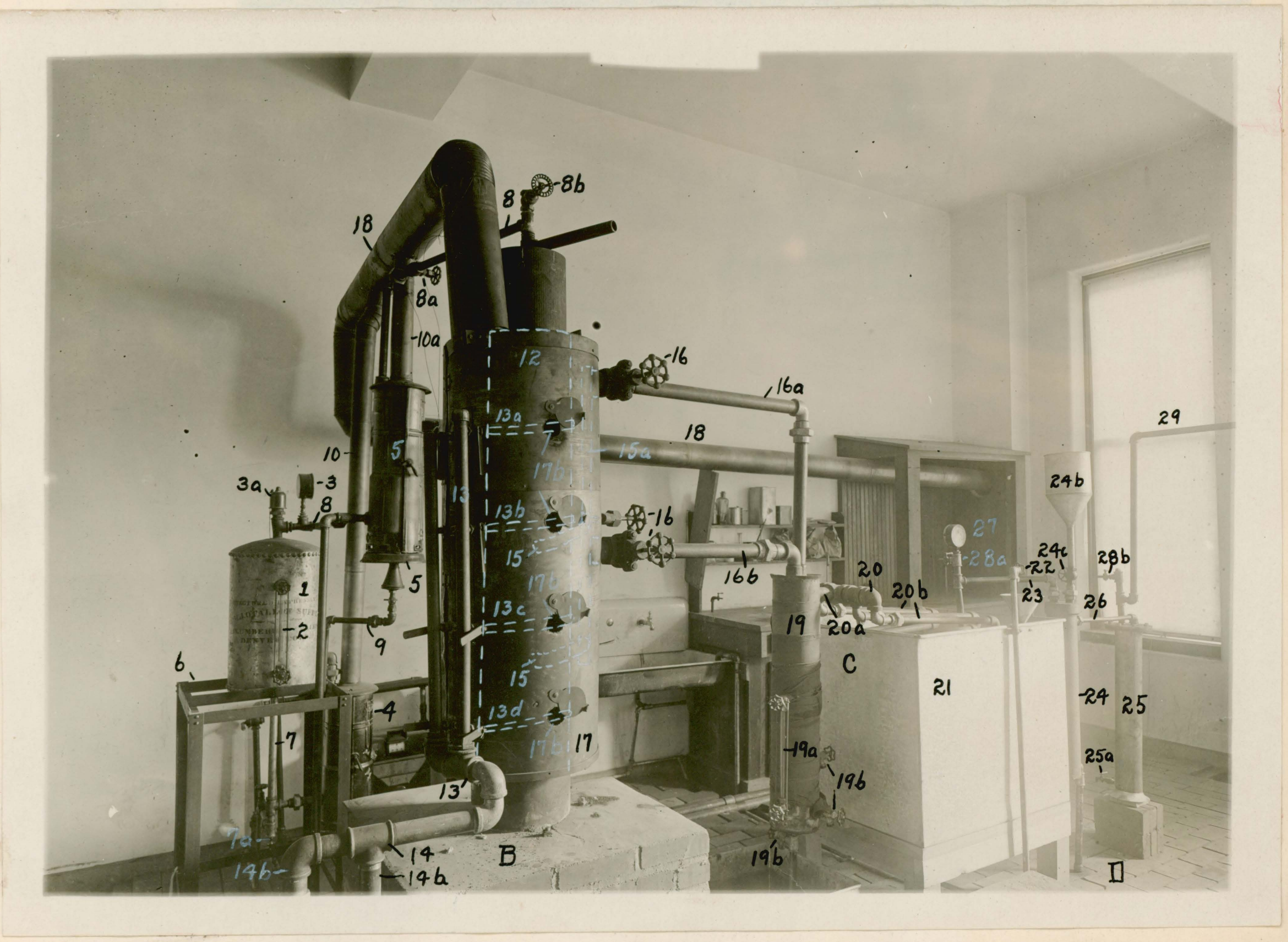
After heating, the shale has a dull black appearance, and will soil the fingers, or paper very easily. This is caused by free carbon, which can be burnt away by strong heating in the presence of air, leaving a clayey substance with the usual composition.

FIGURE I



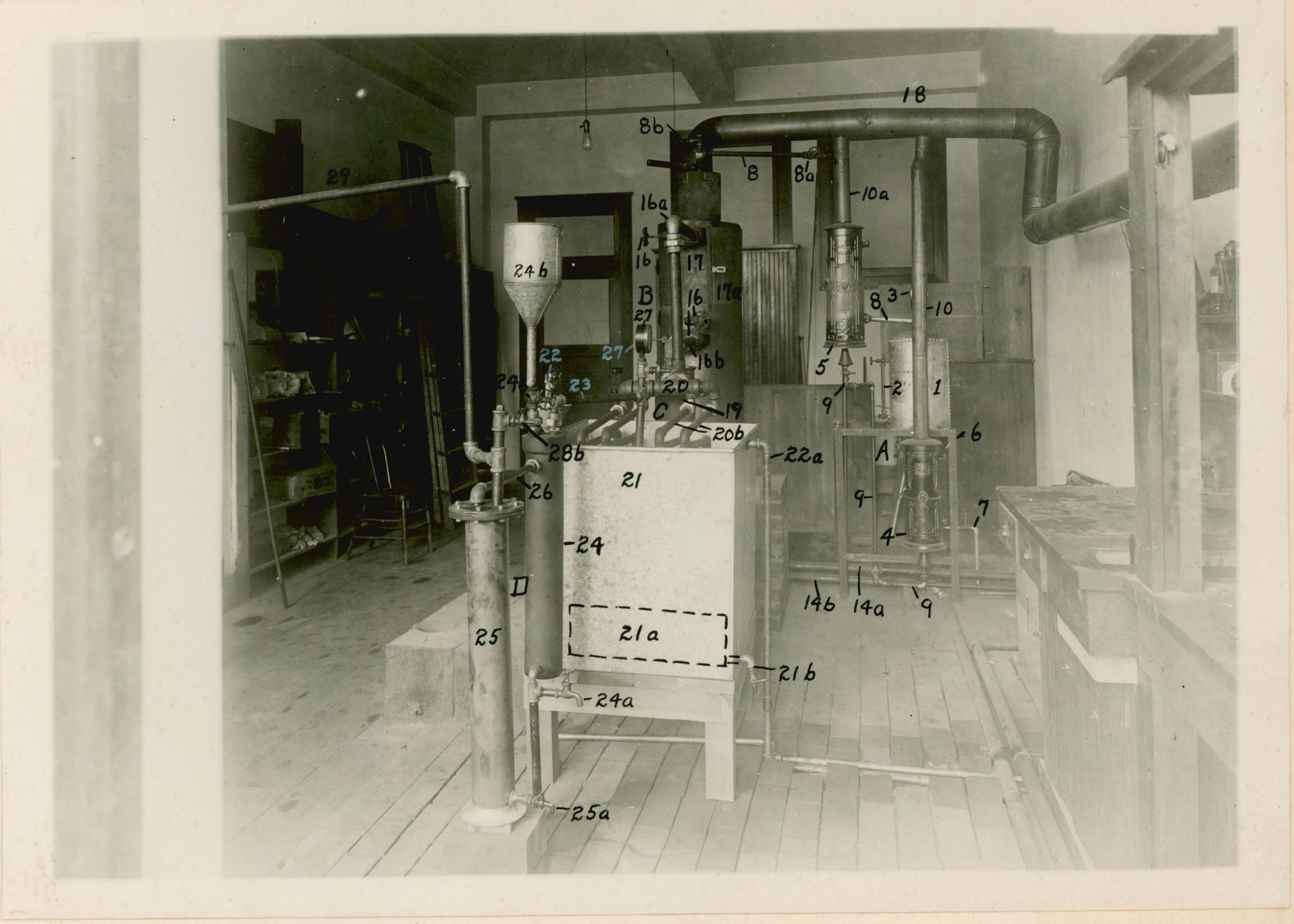
SIDE VIEW OF EDUCTION PLANT

# FIGURE II



VIEW OF EDUCTION PLANT FROM RETORT END.

## FIGURE III



VIEW OF EDUCTION PLANT FROM SCRUBBER END

## RETORTING THE SHALE

DESCRIPTION OF PLANT--The plant used for the eduction of the oil from the shale is that shown in Figures 1,2, and 3. It is located in the Laboratory of the Colorado Geological Survey at the University of Colorado, Boulder, Colorado, and was erected in the summer of 1918 by Professor R. D. George with the assistance of the writer and according to plans drawn up by Professor George.

The principal units 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 lbs. 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-cock, 7a. Heater 4 is used to raise the steam, while heater 5 is used as a superheater, the steam passing out through pipe 8 into the superheated and through valve 8a into the retort. The heaters are ordinary "Lion" gas water-heaters, no.4 being size 1½, while no. 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 stack pipe, 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 2 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 45 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, 13 a,b,c,& 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.

The vapors formed during distillation are led off by means of four 3/4 " pipes, welded to the sides of the retort, two on each side and staggered, the first on one side being 3 inches from the top and the second 21 inches from the top. On the other side; the highest one is 12 inches from the top, and the other 30 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 34 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 it is divided through four 3/4" 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, 8 inches in diameter and 24" 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, 4 inches in diameter and 28 inches 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 4 inches in diameter and 30 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.

Description of Process--To prepare the shale for distillation, it is crushed to approximately ½" size, and all of it is used, including the dust. The plate over the upper end of the retort 12, is removed and about five pounds of fine sand is put in the bottom of the retort to act as a seal against air coming in at the bottom. The shale is then put in on top of the sand, the size of the retort permitting the use of about fifty pounds of crushed shale. The plate with the steam pipe, 8, is then replaced and fastened on with four cap screws, the joint being sealed with a thick pasts of litharge and glycerine, which soon hardens leaving a gas tight joint. The steam line, 8, is then connected to the boiler through the superheater, and the boiler 1, filled about two-thirds full of water, from water line 7a, as shown by sight-gauge, 2. The pressure relief valve, 3a, is set for about 20 pounds pressure, and valve 8a, is closed. All oil is removed from receivers 19 and 21a, and the tank 24b is filled with an oil corresponding to "Straw Oil". The faucet 22 is turned on so as to run water through the condenser tank 21. The burners, 13 a,b,c and d, are now lighted, and the air pressure in line 14b, so adjusted that the flame is just non-luminous, and the shale is thus heated slowly at first. Shortly after the heating is begun, the vacuum pump connected to line 29, is started and a vacuum of 2 inches of mercury is maintained for some time. When the vapors tend to leak out the bottom of the retort, the vacuum is increased until no leakage is apparent, which usually requires a vacuum of about 5 inches of mercury. This vacuum is maintained in order to draw the vapors out of the retort and to overcome the pressure of the oil in the scrubbers, 24 and 25. As soon as vapors begin to pass over, as shown by their escape through cock 28a, when it is opened, valve 24c is opened and the oil in 24b allowed to trickle over the pebbles in the scrubber 24, thus absorbing any light gasoline left in the vapors. When the oil has completely drained out of 24b, it is removed from the scrubber 24, and replaced in the reservcie 24b, and the process repeated.

After the heating has proceeded about four hours and the temperature is about 300°C. steam is generated in boiler 1, by means of heater 4, and when the pressure has reached 20 pounds, the superheater 5, is turned on and valve 8a opened, admitting superheated steam into the retort. This steam diffuses up through the shale, and tends to increase the quantity and quality of the oil obtained, besides affording a vehicle for the removal of the vapors formed in the retort.

The heat is increased, when the steam is turned in, by adding more air to the mixture. The retort is heated just below a dull read or about 500° C. for six hours longer, after which time the heat is turned off. The vacuum pump runs for about twenty minutes additional, after which it is turned off and the run discontinued.

After cooling, the retort is emptied through the bottom plates, by pulling the lever, 11, which brings the holes of the two bottom plates together. The spent shale is prevented from caking in the retort by keeping a sufficient supply of superheated steam going through the shale. Altogether, the water used in the form of superheated steam is about 150% of the shale in weight. It is probable that the caking in the retort could be avoided if the shale were kept in motion. As the retort used was not equipped for such a test, no data on this could be obtained. The condensed oil and water is then drawn out of receivers 19 and 21a and is allowed to separate into two layers, the water going to the bottom and the oil remaining on top. The water is then drawn off and is known as "ammonia-water", while the crude oil is then ready for treatment. The "ammonia-water" will be discussed later.

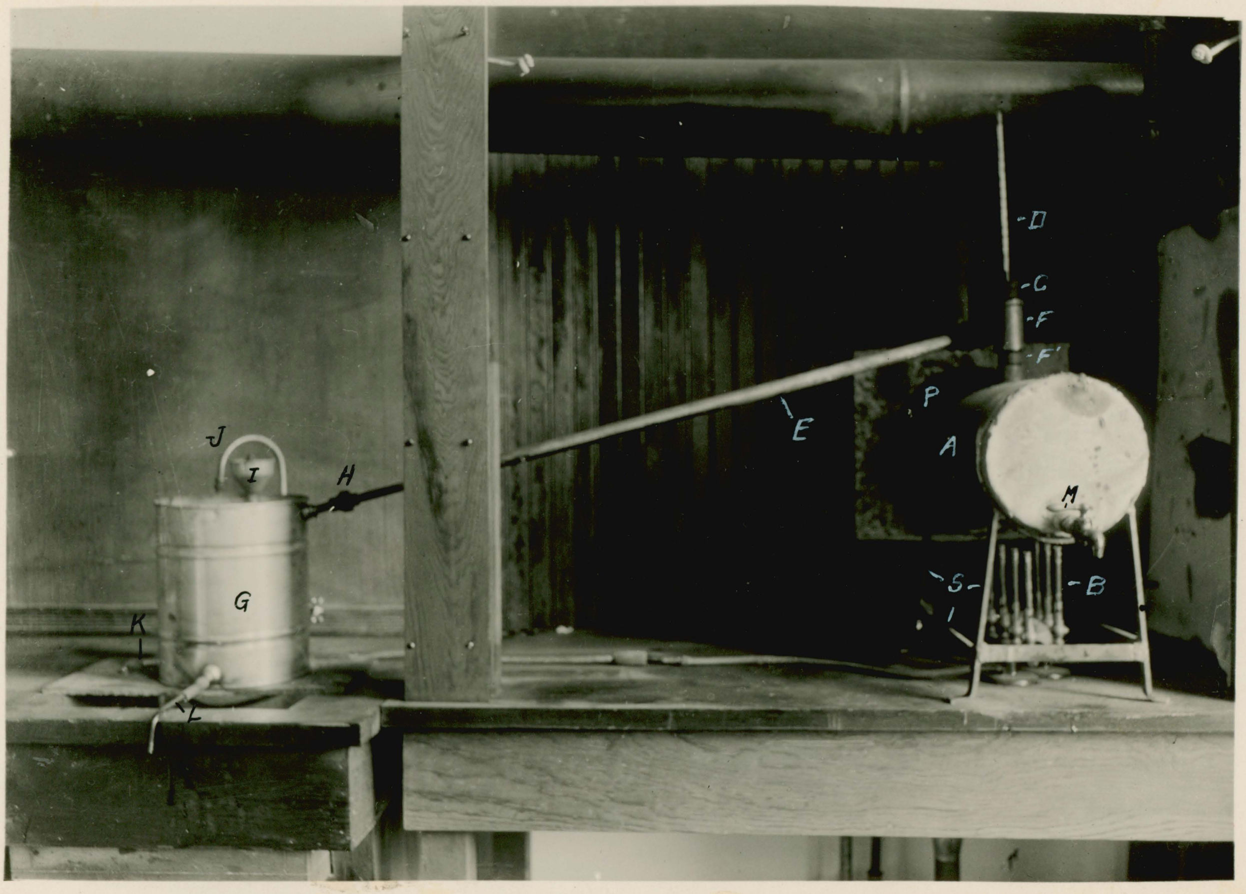
Results of Tests on Distillation of Shale

| Test No. | 1 | 2 | 3 | 4 | 5 | 6 | Average |
|---|---|---|---|---|---|---|---|
| Weight of shale used, pounds | 55 | 52 | 50 | 50 | 45 | 50 | 50.3 |
| Length of run, hours | 11.25 | 10.5 | 10.0 | 10.5 | 10.5 | 10.0 | 10.46 |
| Water used as superheated steam, pounds | 90 | 80 | 85 | 90 | 85 | 80 | 85. |
| Oil recovered, cc. | 6500 | 6200 | 6000 | 6050 | 5800 | 6000 | 6091.7 |
| Gallons per ton | 62.8 | 63.3 | 63.7 | 64.3 | 64.1 | 63.7 | 63.65 |
| Gravity, °Beaume, | 26.8 | 26.5 | 27.0 | 26.9 | 27.0 | 27.2 | 26.9 |

## CHARACTERISTICS OF THE SHALE-OIL

The crude shale oil as taken from the receivers, is very dark in color, being nearly black, with a brownish red cast, and is without noticeable fluorescence. The odor is unpleasant, although not extremely so, and suggests sulphur and alkaloidal bases. The setting point of the crude oil is very little below ordinary room temperature, being about 15° C. (59°F.). After the crude oil has stood in a vessel, more particularly glass, a reddish deposit is formed. This deposit contains some sulphur and a little nitrogen. It is insoluble in water, gasoline, acids, and alkalies, but is very easily dissolved by alcohol.

FIGURE IV



FRACTIONATING STILL AND CONDENSER

## FRACTIONATION OF THE CRUDE OIL

The next step in the treatment of the oil is the fractionation or separation into its various components, such as gasoline, kerosene, lubricating oils, etc. This is accomplished by means of the apparatus shown in figure 4. A is the still, 8 inches in diameter and 20 inches long, made of 1/16" wrought iron rolled to form a cylinder and lap-welded; a flange, F, to which a plate (not visible in the photograph) is fastened by means of cap screws. The joint is made tight by a liberal application of "Smooth-On" Iron Cement No.2. This still A, is placed on a strap-iron stand, S, and is heated by means of bunsen burners, B. At the top of the still, and about the center, a 7/8" hole was drilled and a 1-inch sleeve, F, welded over the hole. To this sleeve was fitted a short nipple, on the other end of which was placed a 1-inch tee. The upper end of the tee was closed by means of a large cork, C, containing a thermometer D, which extended down to about the bottom of the tee. To the side of the tee was fitted a 1-inch street-ell leading into the pipe E, which was connected to the coil of the condenser G, by means of the union H. The pipe E is ½ inch and is fitted to the 1-inch street-ell by means of a ½ x 1" bushing, and about six inches above the union, H, pipe E is reduced to ¼-inch in size. The condenser G, is an ordinary block tin condenser as furnished by the chemistry supply houses, and measures 8 inches in diameter and 10 inches high . The reservoir I is for the introduction of cooling water into the condenser from the faucet, J;and K in the waste-pipe for the cooling water.

Description of Process--To fill the still, the cork C, is removed and a measured amount of the crude oil put in, after which the cork C is replaced and the joint made safe with a thick paste of linseed oil-meal and water, which hardens and prevents the escape of the vapors. The burners B, are then lighted and turned very low at first. A receiver is put under to receive the distillate. The temperature is allowed to rise gradually, and the first fraction distilling over is the gasoline. When the temperature reaches about 96°C., the boiling point of water at this altitude (5,350) there is some "bumping" in the still and a small quantity of water which was still mixed with the oil comes over and settles to the bottom. This water is measured and its amount subtracted from the amount of liquid put into the still, giving the quantity of pure crude oil used.

Everything distilling over below 200° C.(392°F.) is considered gasoline, and when the thermometer, D, registers that temperature the receiver is changed. The fraction distilling between 200° C and 300° S. (392°F and572°F) is taken as kerosene. The next fraction, between 300° C., and about 400°C (572°F·and 752°F.) is considered too light for lubricating oil, and is called "gas-oil". It is used for enriching water-gas, or may be cracked to form more gasoline. After these fractions are taken off, the condenser, G, is disconnected at H, and the next fractions cooled by the air surrounding pipe, E. The lubricating fractions are separated by gravity. When the gravity of the distillate reaches about 28° Be. (.8861) the burners are turned off and the contents of the still allowed to cool somewhat. When cool enough to handled they are drawn off through drain cock, M, and put into an ordinary 2-quart mercury retort. This retort is then covered and sealed and heated in a metallurgical furnace to dryness. The distillate of this last process is the heavy lubricating oil, and the residue in the retort is petroleum-coke. The reasons for carrying out this last fractionation in this manner are:-the greater ease with which the retort can be heated, the greater resistance of the mercury retort over the still A, to heat, and the greater accessibility of the inside of the retort for removing the coke formed. This coke is glossy black in color, very brittle and must be chipped out of the retort. It is nearly pure carbon and is in great demand in certain metallurgical and other processes where a pure reducing agent is needed. It may also be used for fuel, as it burns like ordinary coke, but giving more heat, and leaving practically no ash.

RESULTS OF FRACTIONATIONS

| Test No. | 1 | 2 | 3 | 4 | 5 | 6 | Average |
|---|---|---|---|---|---|---|---|
| Oil Used, C.C. | 6500 | 6200 | 6000 | 6050 | 5800 | 6000 | 6091.7 |
| Gravity | 26.8 | 26.5 | 27.0 | 26.9 | 27.0 | 27.2 | 26.9 |
| Gasoline, cc. | 1000 | 900 | 900 | 910 | 890 | 920 | 920 |
| Percentage | 15.4 | 14.5 | 15.0 | 15.04 | 15.35 | 15.3 | 15.1 |
| Gravity | 51.0 | 51.5 | 51.0 | 51.0 | 50.5 | 50.5 | 50.9 |
| Kerosene, cc. | 2100 | 2000 | 1950 | 1970 | 1800 | 2000 | 1970 |
| Percentage | 32.3 | 32.25 | 32.5 | 32.54 | 31.0 | 33.3 | 32,65 |
| Gravity | 36.0 | 36.0 | 36.0 | 36.5 | 35.5 | 35.0 | 35.8 |
| Gas Oil cc. | 1500 | 1450 | 1350 | 1350 | 1500 | 1200 | 1391 |
| Percentage | 23.1 | 23.4 | 22.5 | 22.31 | 26.0 | 20.0 | 22.9 |
| Gravity | 33.5 | 33.0 | 34.0 | 33.5 | 33.0 | 34.0 | 33.5 |
| Light Lubricating Oil, cc. | 1100 | 1000 | 1020 | 1030 | 900 | 1100 | 1025 |
| Percentage | 17.0 | 16.1 | 17.0 | 17.02 | 15.5 | 18.5 | 16.8 |
| Gravity | 30.0 | 30.0 | 30.5 | 30.5 | 31.0 | 30.5 | 30.4 |
| Heavy Lubricating Oil, cc. | 650 | 700 | 690 | 700 | 350 | 700 | 665 |
| Percentage | 10.0 | 11.3 | 11.5 | 11.56 | 9.5 | 11.7 | 10.9 |
| Gravity | 24.5 | 25.0 | 25.0 | 25.0 | 26.0 | 25.5 | 24.8 |
| Coke, %. | 2.2 | 2.5 | 1.5 | 1.5 | 2.7 | 1.4 | 1.97 |

CHARACTERISTICS OF THE OILS OBTAINED

The various fractions of the oil obtained by the fractionation were put together, all the gasoline fractions being combined, all the kerosene fractions combined, and so on. The properties of each of the crude fractions were then ascertained.

Gasoline.-The gasoline fraction was a very limpid liquid, of 51° Be.(.7735) gravity, and of a yellowish color. Upon standing the color deepened gradually until it was black, and at the same time a purple deposit was left on the glass. This deposit has been spoken of before[1], and is soluble in alcohol.

Kerosene.-The kerosene resembled the gasoline very closely, except in that it has a beautiful purple fluorescence when first distilled, and was not quite so limpid. Its color

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[1] Page 17

was a deeper yellow than that of the gasoline, and, upon standing, this changed to a color much darker than that of the gasoline. Its gravity was 35.5° Be. (.846)

Gas Oil.-This was still heavier and darker in color than the kerosene, and on standing turned black. Its gravity was 33.0° Be. (.859)

Light Lubricating Oil.-This oil came over very dark in color, although the drops appeared to be light red. The same deposit on the glass was noticeable, and the oil became black on standing. The gravity of this combined fraction was 30.5°Be. (.8723).

Heavy Lubricating Oil.-This oil was very black when first obtained in the receiver. On standing the same deposit was left on the glass. The gravity was 25°Be.(.9032)

## TESTS ON THE GASOLINE FRACTION

In the oil industry, there are two ways usually used to remove the lipochromes, or colored substances from[1] the oil. These are,(1) the destruction of the colored substance by some chemical agent and the formation of a compound which is easily soluble in sulphuric acid, water, or sodium hydroxide, and, (2) the absorption of the colored substance by some absorbing agent such as fuller's earth, charcoal or clay.

The oxidation of an oxidizable lipochrome by potassium permanganate or potassium dichromate would be an example of the first process, while the decolorization of oils by fuller's earth would be an example of the second process.

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[1] Chemistry of the Oil Industries", J.E. Southcombe.

These processes would be used either with or without the aid of heat, in most cases. Consequently the tests have been divided into four sections, viz., agitation, or treatment with a reagent in the cold; filtration, a treatment with an absorbent substance both hot and cold; distillation, or treatment with both chemical and absorbing reagents with the aid of heat; and miscellaneous tests which fall into none of the three foregoing classes, or which are combinations. A description of these tests, in order, will be given.

Tests by Agitation.--In preparing the gasoline for the tests by agitation it was re-distilled in glass, and the fraction coming over up to 200° C. (392°F.) was used. Any residue above that temperature was put into the kerosene fraction. All of the following tests were made in glass, the mixture being agitated by compressed air.

Test No.1

200 cc.of gasoline were put into a 500 cc.graduate, and 100cc of concentrated (1.84) sulphuric acid added. The graduate was put in a battery jar filled with cold water, and the mixture agitated for thirty minutes by means of compressed air. The mixture was then allowed to settle for six hours. It was found that the acid had increased in volume to 216 cc. and the oil had decreased in volume to 64 cc., thus giving a loss of 58% to the acid and 10% by evaporation,or a total loss of 68%. The oil remaining was decanted off and agitated for thirty minutes with 8 cc. (4% of the original volume) of caustic soda of 14° Be. After settling a further loss of 2 cc, or 1% was noted. The oil was very clear, but showed a slight yellow tint by reflected light. Upon standing, the oil developed a lemon-yellow color, which persisted for several weeks.

This test would indicate that 68% of the gasoline fraction was composed of unsaturated hydrocarbons, or compounds soluble in concentrated sulphuric acid, and 1% of acid or phenolic compounds.

### Test No. 2

100 cc. of the gasoline was agitated for thirty minutes with 4% of concentrated sulphuric acid. After settling the sludge was drawn off, the gasoline washed, and then agitated with 4% of caustic soda ($14^{\circ}$Be.) for thirty minutes. The resulting oil was yellow in color, and, on standing, developed a deep red color which persisted. The total loss was 22%.

### Test No. 3

100 cc. of the gasoline was agitated for thirty minutes with 10% of hydrochloric acid (1.18), settled, washed with water, and again agitated with 4% of caustic soda. The resulting gasoline was ddep brown in color, and after a few days had turned to black. No deposit was left on the glassware after treatment. Total loss in refining was 12%.

### Test No. 4

100 cc. of the gasoline was agitated for thirty minutes with 10% of nitric acid (1.42), settled, washed with water, and agitated with 4% caustic soda. The resulting gasoline was of a bright red color, which changed to a darker red on standing. No deposit was left on the glassware. Total loss in refining was 14%.

### Test No. 5

100 cc. of the gasoline was agitated for thirty minutes with 6% "aqua regia", made by mixing one part of strong nitric acid with three parts of strong hydrochloric acid. The gasoline was then washed with water, and again agitated with 4% caustic soda. The resulting oil was deep brown in color, the color persisting on standing. The total loss in refining was 8%.

### Test No. 6.

This is a repetition of test No. 5, using 10% of "aqua regia". The resulting oil was light brown in color, changing but slightly on standing. The loss in refining was, however, 13%.

### Test No. 7

100 cc. of the gasoline was agitated for thirty minutes with 10% of strong acetic acid, and settled. No action

was apparent. The gasoline was then agitated with 4% caustic soda for thirty minutes. A loss of 2% was discernible. The color of the gasoline was not changed.

Test No. 8

100 cc. of the gasoline was agitated for thirty minutes with 10% of aqueous solution of tannic acid. After settling, no change was noted.

Test No. 9

The same oil used in test No. 8, was agitated for thirty minutes with 10% of sodium acid carbonate, $NaHCO_3$. At the end of this time no change in the oil was noted.

Test No. 10

100 cc.of the gasoline was agitated for thirty minutes with 10% of a concentrated solution of potassium dichromate. The oil was settled and a reddish color in the oil noted. The oil was then agitated for thirty minutes with 4% of caustic soda. The oil had acquired a dark-brown color, which persisted. The loss in refining was 4%.

Test No. 11.

100 cc. of the oil was agitated for thirty minutes with 10 % of an aqueous solution of potassium permanganate. The oil assumed a purple color from the permanganate, which was changed to a dark brown color upon agitation with 4% of caustic soda for thirty minutes. This color persisted. The loss in refining was 4%.

Test No. 12

100 cc. of the oil was agitated with 10% of a concentrated solution of potassium dichromate for thirty minutes. After settling, it was again agitated with 4% of concentrated sulphuric acid, settled, washed with water, and agitated with 4% of caustic soda solution. The color was yellow changing to a dark brown on standing. The loss in this treatment was 20% of the oil.

Test No. 13

100 cc.of the gasoline was agitated for thirty minutes with 10% of a solution of potassium permanganate, washed and agitated with 4% of concentrated sulphuric acid for thirty minutes. It was again washed, then agitated with 4% of caustic soda. The results were the same as those obtained in test No. 12.

Test No. 14

100 cc. of the oil was agitated for thirty minutes with 6% of an oxidizing mixture made up of six parts

of potassium dichromate, five parts of concentrated sulphuric acid and thirty parts of water. After settling the oil was washed and agitated with 4% of caustic soda. The resulting oil was brown in color. The loss by this treatment was 14%.

Test No. 15

100 cc. of gasoline was agitated for thirty minutes with 6% of an oxidizing mixture made up of thirteen parts of potassium permanganate, twelve parts of concentrated sulphuric acid and 100 parts of water. The oil was agitated, after settling, with 4% of caustic soda. The resulting oil was dark brown in color. The loss was 14%.

Test No. 16

100 cc. of the oil was agitated for thirty minutes with 10% of strong chlorine water. The oil was washed, and agitated with 4% of caustic soda. The resulting oil was yellowish brown in color, and darkened upon standing. The loss was 5%.

Test No. 17.

100 cc. of the oil was agitated for thirty minutes with 2% of concentrated (1.84) sulphuric acid, washed, and agitated with 4% of caustic soda. The oil was yellow in color, and deepened slightly on standing, turning finally to a brownish red. Loss in treatment was only 7%.

Test No. 18

100 cc. of gasoline were put into the agitator and 4 grams of zinc dust added. This was then treated with 10 cc. of strong acetic acid. The evolution of hydrogen was allowed to continue for about twenty minutes, and the mixture was agitated with air for fifteen minutes. No change in the oil was noticeable. The oil was then washed and agitated with caustic soda. The resulting oil was still very dark. The loss in this treatment was 4%.

Test No. 19

100 cc. of the oil was agitated for thirty minutes with 2 grams of potassium nitrite and 4 cc. of sulphuric acid. The oil appeared green on account of the dissolved nitrous acid fumes. Upon washing, or standing for a short time, this color is removed. The oil was then washed with 4% of caustic soda. The resulting oil was dark brown in color. Loss was 20%.

Test No. 20

100 cc. of the oil was agitated for thirty minutes with 10% of an aqueous solution of stannous chloride. The oil was then washed with water and agitated with 2% of concentrated sulphuric acid, after which it was washed and agitated with 4% of caustic soda. The resulting oil was brown in color and darkened on standing. The loss was 8%.

Test No. 21

100 cc. of the oil was agitated for thirty minutes with 10% of hydrogen peroxide. It was then given the usual treatment with 2% of sulphuric acid and 4% of caustic soda. The resulting oil was dark yellow, which color changed to a dark brown on standing. The loss was 8%.

Test No. 22

100 cc. of the gasoline was agitated for thirty minutes with 6% of a solution of ferrous and copper sulphates, and sodium chloride, made acid with sulphuric acid. The oil was then agitated with 4% of caustic soda. The resulting oil was dark brown in color. Loss was 10%.

These foregoing tests differ mostly in the acid constituent used in the treatment. The alkali treatment was, in each case, accomplished with 4% of caustic soda of a gravity of 14°Be. Tests 10 to 16, inclusive, and 21, were tests using oxidizing agents along with the usual acid and alkali treatment, while tests 18, 19, and 20, were made using reducing agents.

Of all of these, the treatment given in test No. 17 seems to be the most satisfactory. The color of the gasoline obtained was as good as that obtained in any other test, the loss in treatment was comparatively small, due to the use of only a small amount of acid, which also reduces the cost of the treatment.

The acid treatment of test 17, i.e. 2% of concentrated sulphuric acid, is therefore taken as the basis for the following tests, which differ in the basic constituent used.

Test No. 23

100 cc. of the gasoline was agitated with 2% of concentrated (1.84) sulphuric acid, settled, washed with water and agitated for thirty minutes with 4% of caustic soda of a gravity of 6° Be. The resulting oil was decidedly yellow in color, and, on standing, darkened to a brown. The loss in refining was 6%.

Test No. 24

100 cc. of the gasoline was agitated with 2% of concentrated sulphuric acid, settled, washed with water, and then agitated for thirty minutes with 4% of a caustic soda solution of 40° Be. The resulting oil was light yellow in color, and changed to a yellowish red on standing. The loss was 7%.

Test No. 25

100 cc. of the gasoline was agitated for thirty minutes with 2% of concentrated sulphuric acid, settled, washed, with water, then agitated with 20% of a caustic soda solution of 40°Be. The resulting oil was of a light yellow color, deepening to a yellowish red, as in the preceding test. The loss was 7%.

Test No. 26

100 cc. of the gasoline was agitated with 2% of concentrated sulphuric acid, settled, washed, and then agitated with 10% of a saturated solution of salt. The resulting oil was brown, the color deepening on standing. The loss by this treatment was 7%.

Test No. 27

100 cc. of the gasoline was agitated with 2% of concentrated sulphuric acid, settled, washed, and agitated for thirty minutes with 10% of a saturated solution of sodium bicarbonate. The oil was of a brown color, which deepened on standing. The loss was 6%.

Test No. 28

100 cc. of the gasoline was agitated for thirty minutes with 2% of concentrated sulphuric acid, then settled and washed wth water. 10 cc of a saturated solution of sodium carbonate was added, and the mixture agitated for thirty minutes. The resulting oil was brown in color, which color persisted. The loss was 8%.

### Test No. 29

100 cc. of the gasoline was agitated for thirty minutes with 2% of concentrated sulphuric acid, then settled, washed with water, and agitated for thirty minutes with 10% of a saturated solution of ferric chloride. No change was noted, which was due to the ferric chloride, although the oil has assumed a red color.

### Test No. 30

The remaining oil from test 29, was further agitated with 4% of caustic soda solution (14 Be.) The resulting oil behaved like that in test 17, as if no other reagent has been used, but the acid and soda. The loss was 7%.

### Test No. 31

100 cc. of the gasoline was agitated for thirty minutes with 2% of concentrated sulphuric acid, settled, and washed with water. It was then agitated with 10% ofa solution of sodium phosphate. No action due to this latter reagent was noticed.

### Test No. 32.

The oil from test 31 was washed with water, and further agitated with 10% of lead acetate solution. No action resulted.

### Test No. 33

The oil from test 32 was washed with water, and again agitated with 10% of an ammonium molybdate solution. No reaction resulted.

### Test No. 34.

The oil from test 33 was washed with water, and again agitated with 10% of a solution of potassium ferri-cyanide. No action resulted.

### Test No. 35.

The oil from test 34 was thoroughly washed with water and agitated with 10% of a potassium ferrocyanide solution. No action resulted.

### Test No. 36.

The oil from test 35 was again washed with water and agitated with 10% of a solution of potassium cyanide. No action resulted.

### Test No. 37

The oil from test 36 was agitated with 4% of a caustic soda solution. The resulting oil was the same as that obtained in test 17, showing that the only reagents affecting the oil, were the concentrated sulphuric acid and caustic soda.

Test No. 38

100 cc. of the gasoline was agitated for thirty minutes with 2% of concentrated sulphuric acid, settled, washed with water, then agitated with 10% of ammonium hydroxide (0.90). The resulting oil was brown in color, and darkened on standing. The loss was 7%.

Test No. 39

100 cc. of the oil was agitated for thirty minutes with 2% of concentrated sulphuric acid, settled, washed with water, and agitated with 10% of dimethyl glyoximee. A slight brown precipitate, insoluble in alkali, but soluble in hydrochloric acid, was formed. On further treatment with the dimethyl glyoxime,, the precipitate was not formed. The oil was then agitated with 4% of a caustic soda solution. The resulting oil was like that obtained in test 17. The loss noted was 7%.

Test No. 40

100 cc. of the oil was agitated for thirty minutes with 2% of concentrated sulphuric acid, settled, washed with water, and then agitated with 10% of a solution of mercuric chloride. A white precipitate, soluble in hydrochloric acid was formed. On further treatment with 4% of a caustic soda solution, the oil became yellow in color, but darkened on standing. Loss, 8%.

These foregoing tests tend to show that the alkaline reagent in the treatment of the gasoline from shale-oil, have but little effect on the oil. Of those tried, a solution of caustic soda of 14°Be. proved to be as suitable as any. The amount of this reagent seems immaterial, but about 4% is about the best amount to use. More than this is unnecessary, and less does not seem to give an oil which will not darken to a great extent.

TESTS BY FILTRATION

The second of the methods of decolorizing oils, as given on page 23 was then tried. A filter made of glass was constructed as follows:- A glass tube 1 inch in diameter and four feet long was covered at the one end by means of a piece of cloth fastened tight with a wire ring. In the other end was inserted a rubber stopper carrying a small glass tube, which led to a reservoir for the oil. To set up for operation, the large tube is supported vertically by a ring stand, the filtering medium is poured in at the top and the cork and reservoir put on. The joints at the corks are covered with paraffin. The oil to be filtered is then put in the reservoir and allowed to filter down the tube. The distillate is caught at the bottom, in some suitable receiver.

The oils filtered were used, for the most part, in making the other tests described.

Test No. 41

The tube was filled with the variety of fuller's earth known as "Floridine". This is a granular earth of a light grey color. The reservoir was filled with 500 cc. of gasoline, and a receiver put at the bottom of the tube. After two days, the filtrate was examined. Its color had not been changed to a degree that could be noticed. The amount recovered was 405 cc., the loss being due to the oil absorbed by the earth.

Test No. 42

500 cc. of the gasoline was treated as above, using animal charcoal in place of the fuller's earth. No decolorization was discernible.

Test No. 43

500 cc. of the gasoline was treated as above, using pulverized English fuller's earth. No decolorization was discernible.

### Test No. 44

500 cc. of the gasoline was treated as above, using the Floridine fuller's earth, which had been calcined at $400^{\circ}$C. for two hours. No effect was noticed.

### Test No. 45

500 cc. of the gasoline was treated as above, using English fuller's earth which had been calcined as $400^{\circ}$C. for two hours. No effect was discernible.

### Test No. 46

500 cc. of the oil was treated as above, using "kieselguhr" a diatomaceous earth. No decolorization was noticed.

### Test No. 47

500 cc. of the gasoline was treated as above, using "spent" shale, which would pass a 20 mesh screen for the filtering medium. This had no effect.

### Test No. 48

500 cc. of the gasoline was treated as above, using "raw" shale. No effect could be noticed.

## TESTS BY DISTILLATION

The following series of tests, was preformed to investigate the action of various substances on the gasoline fraction with the aid of heat. As the gasoline is very volatile, these tests were carried out as distillation tests. The oil was put into a 500 cc. Engler distilling flask, the substance whose action is to be investigated is added, a thermometer placed in the neck of the flask by means of a cork, and the gasoline distilled. After distillation, the collected oil was treated with 2% of concentrated acid and 4% of caustic soda solution of $14^{\circ}$Be. strength. No losses are recorded, as it was considered that any oil left in the distilling flask would be added to the kerosene fraction. The losses by the acid and alkali treatment would be approximately those noted before.

Test No. 49

200 cc. of the gasoline was distilled over 20% of Florida fuller's earth. After the usual treatment, no change was noted.
(When "no change" is noted, it means no change except that caused by the acid and alkali treatment).

Test No. 50

200 cc. of the gasoline was distilled over 20% of animal charcoal. No change was noted.

Test No. 51

200 cc. of the gasoline was distilled over 20% of the English fuller's earth. No change was noted.

Test No. 52

200 cc. of the gasoline was distilled over 20% of kiesel-guhr. No change was noted.

Test No. 53.

200 cc. of the gasoline was distilled over 20% of calcined Florida earth. No change was noted.

Test No. 54

200 cc. of the gasoline was distilled over 20% of calcined English fuller's earth. No change was noted.

Test No. 55.

200 cc. of the gasoline was distilled over 10% of stick caustic soda. On account of the water contained in the caustic soda, the first part of the distillation was accompanied by violent "bumping". However, no change was noted.

Test No. 56

Test 55 was repeated using caustic potash in place of the caustic soda. No change was noted.

Test No. 57

200 cc. of gasoline was distilled over 20% of solid phosphoric acid. No change was noted.

Test No. 58.

200 cc. of gasoline was distilled over 20% of anhydrous aluminum chloride. No change was noted.

Test No. 59

200 cc. of gasoline was distilled over 20% of common salt. The distillate was nearly clear, showing only a slight yellow color, which deepened somewhat on standing. This color changed to a light cherry red, which persisted.

At this point the gasoline was tested, qualitatively for sulphur.[1] A positive test was obtained, but the quantity seemed to be small. The gasoline was therefore subjected to the following treatments for the purpose of removing the sulphur.

Test No. 60

200 cc. of the gasoline was distilled over 20% of powdered copper.[2] The distillate was lighter in color, than usual, but after treatment with acid and alkali, no change was noted.

Test No. 61

200 cc. of the gasoline was distilled over 20% of iron turnings[3]. No change was noted.

Test No. 62

200 cc. of the gasoline was distilled over 20% of black copper oxide. No change was noted.

Test No. 63

200 cc. of the gasoline was distilled over 20% of litharge or lead oxide. No change was noted.

Test No. 64

200 cc. of the gasoline was distilled over 20% of a mixture composed of 75 parts of black copper oxide. 10 parts of litharge, and 15 parts of ferric oxide.[4] No change was noted.

Test No. 65

200 cc. of the gasoline was distilled over 20% of ligharge. The distillate was agitated with 2% of concentrated sulphuric acid and washed with 2% of It was then agitated with 6% of a "doctor" solution

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1. See Noyes and Milliken "Laboratory Manual of Organic Chem".
2. Thorpe "Dictionary of Chemistry", 1913, vol. IV, p. 128
3. Mabery,"Composition of American Petroleum,J.Am.Chem.Soc.1906
4. Frasch "Desulphurizing Petroleum",J.Ind.and Eng.Chem.,1908

which was a concentrated solution of sodium plumbite in 14°Be. caustic soda[1] The oil after settling was treated with a small quantity of flowers of sulphur to settle the "doctor". The resulting oil was light brown in color. The color deepening on standing. The loss was 14%.

These tests indicate that the color of the gasoline is caused by some compound other than a sulphur compound.

MISCELLANEOUS TESTS

These tests, for the most part, were performed in search of definite compounds, which were suspected to be present in the gasoline.

The white precipitate cuased by agitation of the gasoline with mercuric chloride indicated quinoline, pyridine or pyrrole. Confirmatory tests for these compounds were therefore performed.

Test No. 66

Test for pyridine or pyrrole. One drop of an aqueous solution of isatin was put upon a test tile and a drop of concentrated sulphuric acid added. To this was added two drops of the gasoline. Pyrrole or pyridine would have given a dark-blue or indigo precipitate[2] but the test was negative.

Test No. 67

Test for quinoline. 100 cc. of the gasoline was agitated with 10% of a solution of 5 grams of iodine in 100 cc. of a 7% solution of potassium iodide. Quinoline would have caused a red-brown precipitate, insoluble in hydrochloric acid. No precipitate was formed, however.

Test No. 68

As a confirmatory test of nos. 66 and 67, the gasoline was tested qualitatively for nitrogen[3] The gasoline gave a negative test for nitrogen.

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1. "Petroleum and Petroleum Products", I.C.S., Scranton, Pa.
2. "Watts Dictionary of Chemistry", Morley and Muir.
3. "Laboratory Manual of Organic Chemistry". Noyes and Milliken

The action of the gasoline in turning red or brown upon standing indicated that furfuran or furfuraldehyde might be present. These were accordingly tested for.

Test No. 69

100 cc. of the oil was agitated with 20% of a solution of ammonium sulphide. Furfuran or furfuraldehyde[1] would have caused a yellow crystalline precipitate, however, the precipitate was a dirty green. Upon further treatment, the oil resumed the red-brown color, indicating the absence of these compounds.

Owing to the peculiar odor of the gasoline, and its tendency, in some cases to become yellow, terpenes were suspected. Test 5, using "aqua regia" might have been used, but a more decision test was needed. Therefore, the following were made:-

Test No. 70

100 cc. of the gasoline was treated with hydrochloric acid gas, and the oil cooled. After continuing the process for one hour, the oil was filtered. No precipitate was formed. The oil was a dark blue color, but upon the destruction of the hydrochloric acid it contained, either by distillation or by treatment with 4% of caustic soda, it resumed a deep brown color.

The peculiar characteristics of the gasoline suggested that the color could possibly be due to colloidal particles, perhaps colloidal carbon. As colloids are coagulated under the influence of electrolytes or any source of direct current, the following test was performed.

Test No. 71

200 cc. of the gasoline was put into a beaker, and two copper plates, attached to the terminals of a static machine, were placed in the gasoline. An electrostatic stress of about 50,000 volts was maintained for one and one-half hours. At the end of that time, the copper plates were examined, as well as the color of the oil, but no change had taken place.

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[1] "Watts Dictionary of Chemistry" Morley and Muir

Test No. 72

A portion of the gasoline obtained by distillation was shaken with successive quantities of alcohol, to see if the colored substance would go into the alcohol. While each fraction of the alcohol became colored red, it did not seem to detract from the color of the gasoline.

Test No. 73

An effort was made to "salt out" the coloring matter. A sample of 200 cc. of the gasoline was shaken up with 50 grams of dry sodium chloride, or common salt. It was then covered and allowed to stand for two days, after which it was filtered from the salt. No change in the color could be noticed.

Test No. 74

500 cc. of the gasoline was fractionated in a 1000 cc. distilling flask, over 20% of common salt. The fractions were collected as follows--(1) up to 100 C., (2) 100-110°C., (3) 110-120°C., (4) 120-130°C., (5) 130-140°C., (6) 140-150°C., (7) 150-160°C., (8) 160-170°C., (9) 170-180°C., (10) 180-190°C., and, (11) 190-200°C. All fractions except the last two come over very clear. The last two were slightly yellow. Upon standing for two weeks, all the fractions developed a color ranging from yellow in the first fraction to a deep reddish-brown in the last. At the same time a brownish-black deposit was formed on the glass. This deposit is the same one that was noted before, and is soluble in alcohol. Each of these fractions was refined separately with 2% of concentrated sulphuric acid and 4% of caustic soda. The color ranged from a light lemon yellow in the first fraction, through yellow to an orange color in the last two fractions.

A large quantity of the gasoline was then prepared for the following tests by distilling over 20% of common salt, and refining with 2% of concentrated sulphuric acid and 4% of caustic soda. The gravity of the refined gasoline was 51° Be.

Test No. 75

200 cc. of the gasoline was fractionated according to the method prescribed by the American Society for Testing Materials. The following results were obtained.

| Temperature |  | Amount of Oil | Difference in |
|---|---|---|---|
| Centigrade | Fahrenheit | Distilled Over | Percentage |
| 60 | 140 | First drop |  |
| 100 | 212 | 5.0 % | 5.0 % |
| 110 | 230 | 8.0 % | 3.0 % |
| 120 | 248 | 18.0 % | 10.0 % |
| 130 | 266 | 35.5 % | 17.5 % |
| 140 | 284 | 52.0 % | 16.5 % |
| 150 | 302 | 68.0 % | 16.0 % |
| 160 | 320 | 79.0 % | 11.0 % |
| 170 | 338 | 87.0 % | 8.0 % |
| 180 | 356 | 94.0 % | 7.0 % |
| 190 | 374 | 97.5 % | 3.5 % |
| 200 | 392 | 100.0 % | 2.5 % |

These results are plotted in figure 5.

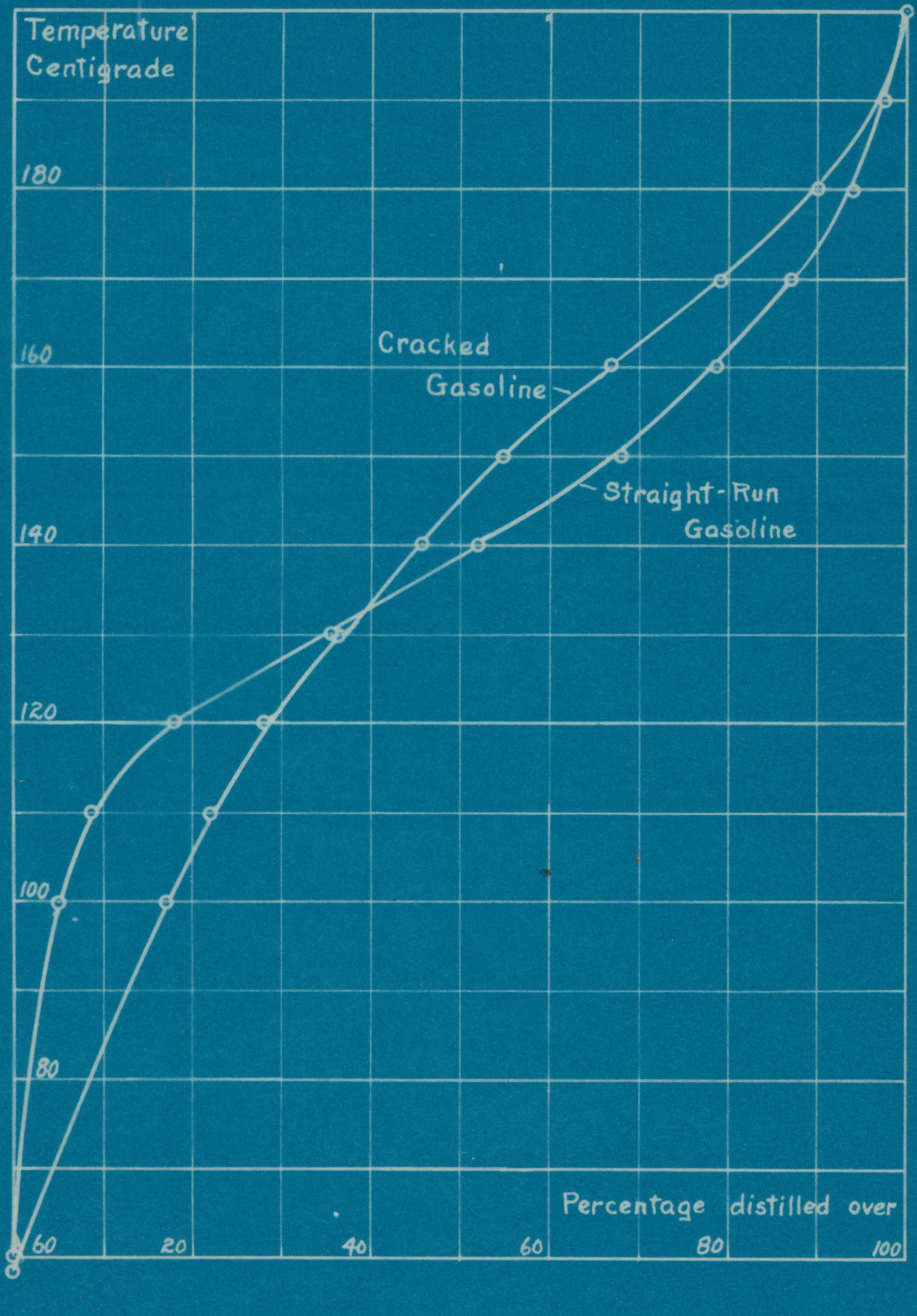
Test No. 76

A calorimeteric test was made on the gasoline, to determine the heating value per pound and per gallon. The instrument used was the Junker's calorimeter at the State Oil Laboratory. The following is the record of the test.

| Date | Time | Water |  |  |  | Gasoline |
|---|---|---|---|---|---|---|
|  |  | Temperature |  |  | Weight | Weight |
|  |  | IN | Out | Rise |  |  |
| 6-19 | 4:00 | 62.8- | 68,7 |  | 8.5 | 257 grams |
|  | 4:03 | 63.0- | 68.8 |  |  |  |
|  | 4:06 | 63.2- | 68.8 |  |  |  |
|  | 4:09 | 63.8- | 70.9 |  |  |  |
|  | 4:12 | 64.2- | 71.4 |  |  |  |
|  | 4:15 | 64.2- | 71.4 |  |  |  |
|  | 4:18 | 63.8- | 71.0 |  |  |  |
|  | 4:21 | 62.8- | 70.0 |  |  |  |
|  | 4:24 | 62.2- | 70.9 |  |  |  |
|  | 4:27 | 62.2 - | 70.5 |  |  |  |
|  | 4:30 | 62.0 | 70.5 |  |  |  |
|  | 4:33 | 62.0 | 69.0 |  |  |  |
|  | 4:36 | 62.0 | 68.7 |  | 67.5 | 247 grams |
| Average |  | 63.0 | 70.7 | 7.0 | 59.0 | 10 grams |

# PLATE VI.

## DISTILLATION TESTS OF GASOLINE



Calculation of Heat Value

$$\text{B.T.U. per pound} = \frac{\frac{7 \times 59}{10}}{28.31 \times 16} = \frac{28.31 \times 413 \times 16}{10} = 18{,}660$$

$$\text{B.T.U. per gallon} = 18{,}660 \times 6.46 = 120{,}540$$

(6.46 is number of pounds per gallon for 51°Be gasoline)

These results wil be discussed later.

TESTS ON KEROSENE

A portion of the combined kerosene fractions was refined to observe its behavior. Several portions were also treated by different filtering mediums. However, the most important tests were the cracking tests.

Test No. 77

500 cc. of the kerosene was filtered in the filter described under test 41, using Florida fuller's earth. No decolorization was noted.

Test No. 78

500 cc. of the kerosene was filtered using the English fuller's earth. No decolorization was noted.

Test No. 79.

500 cc. of the kerosene was filtered using animal charcoal. No decolorization was noted.

Test No. 80

500 cc. of the kerosene was filtered using kiesel guhr. No decolorization was noted.

Test No. 81

500 cc. of the kerosene was filtered using "spent" shale. No decolorization was noted.

Test No. 82

500 cc. of the kerosene was filtered using "raw" shale. No decolorization was noted.

Test No. 83

500 cc. of the kerosene was filtered using calcined Florida fuller's earth. No decolorization was noted.

### Test No. 84

500 cc. of the kerosene was filtered using calcined English fuller's earth. No decolorization was noted.

### Test No. 85

200 cc. of the kerosene was distilled over 20% of common salt. The distillate was then treated with 2% of concentrated sulphuric acid and 4% of caustic soda. The resulting oil was of a deep cherry red color, which color persisted. The loss was 9%.

### Test No. 86

95 cc. of the kerosene obtained in test 85 was heated on a water-bath for two hours with 20% of Florida earth, at the end of which time, the oil was filtered while hot. The resulting oil was somewhat lighter than before this treatment, but the change was only slight.

### Test No. 87

95 cc. of the kerosene obtained in test 85 was heated on a water-bath for two hours with 20% of English fuller's earth, and then filtered hot. No change in color was noted. Test No. 85 was repeated in order to obtain more oil for the following tests.

### Test No. 88

100 cc. of the kerosene obtained in test 85, was heated on a water bath for two hours with 20% of animal charcoal, and then filtered hot. The resulting oil was lighter in color, being little different than that obtained in test 86.

### Test No. 89

100 cc. of the kerosene obtained in test 85, was heated on a water bath for two hours with 20% of kiesel guhr and filtered hot. No decolorization was noted.

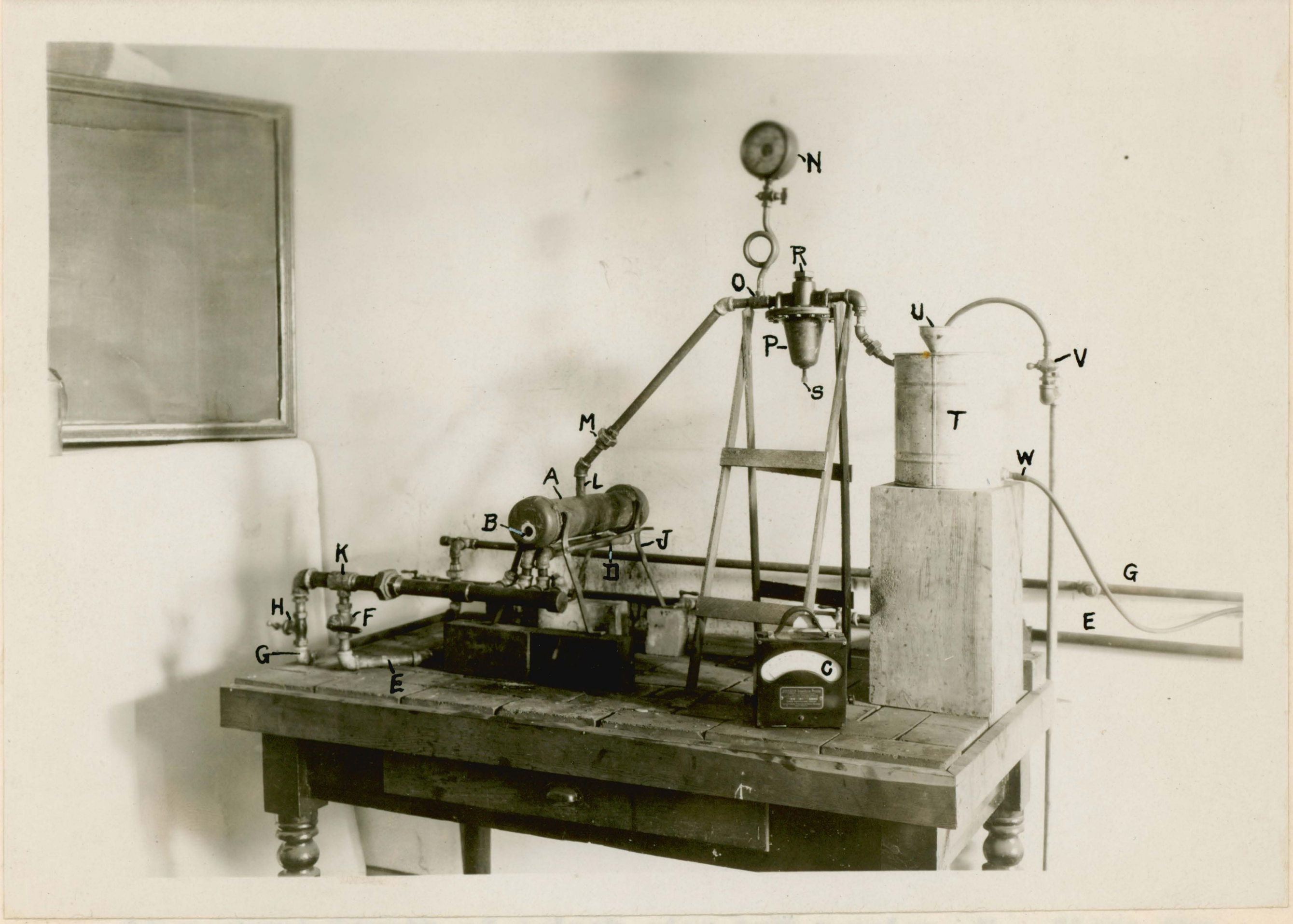
### Test No. 90.

100 cc. of the kerosene obtained in test 85, was heated on a water bath for two hours with 20% of calcined Florida earth, and filtered hot. The resulting oil was the same as obtained in test 86.

### Test No. 91

100 cc. of the kerosene obtained in test 85 was heated on a water bath for two hours with 20% of calcined English fuller's earth, and filtered hot. No change in color was noted.

FIGURE VI



APPARATUS USED FOR CRACKING TESTS

Tests of Kerosene by Cracking.- The combined kerosene fractions were tested by cracking under various pressures and the amount and character of the gasoline obtained was noted. A description of the cracking plant, which is pictured in figure 6, will be given here.

The cracking still, A, is a piece of 3-inch steel pipe, 15 inches long, with a cast-iron cap screwed on each end. In the cap on the front a piece of 3/4" pipe, 5 inches long is screwed in from the indide, before it is put on. This pipe is capped at the inner end, while the outer end, B, is left open so that an electric pyrometer can be inserted. The temperature is read from scale, C, to which the pyrometer is attached. The still is supported by a strap iron stand, J, and is heated by means of burners, D, which are supplied with gas from main E, and controlled by valve, F. Compressed air is supplied from main, G, and the flow controlled by means of valve, H. In the fitting, K, is a cone which aids in ~~of~~ the mixing of the air and gas.

Midway between the ends of the still, and at the top, a ½-inch nipple, to which is attached a 45-fitting leading to a short length of ½-inch pipe and a union, M. Since the photograph was taken, this union has been replaced by a right-and-left coupling, which makes a much tighter joint. From the union a piece of ½-inch pipe leads to another 45-fitting, which connects to the pressure-relief valve, P, by means of a horizontal pipe. The length of pipe between the two 45-fittings is 20 inches. A pressure gauge, N, to record the pressure in the system, is placed at O. The pressure relief valve, P, is of the Moeller type, discharging at atmospheric pressure, the pressure on the inlet side, being controlled by the cap, B, and small adjustment screw, S. The discharge pipe from the valve, leads to a small block-tin condenser, T, which is supplied with running water through reservoir, U, from faucet, V. The water is wasted through W. The outlet for the condensed liquid from the condenser, is at the back, and is not shown in the photograph. This block-tin condenser had to be replaced later, by one in which the coils were iron, because the tin would not stand up under the high temperatures at which the vapors entered it.

All permanent joints were put together with a mixture of "Smooth-On" Iron Cements Nos. 3 and 1. Temporary joints were made using litharge and glycerine.

Many difficulties were encountered before this apparatus could be run without serious leaks. All joints must be carefully made, and the material used in the making of the apparatus must be flawless. It might be stated here, that this small still replaced one which had a capacity of four gallons. The large still was very difficult to manage on account of its weight and the amount of oil required, and it leaked very badly, therefore, necessitating a still much smaller in size.

Operation of the Cracking Still.-In order to put the kerosene into the still, the right-and-left coupling at M, was unfastened, and the still turned over to the left slightly. 500 cc. of the oil to be cracked was then poured in by means of a funnel, the still turned back, and the coupling at M, tightened up using litharge and glycerine on the threads. The pressure-relief valve, P, was next adjusted to about the desired pressure by turning the threaded cap, R. Water was then started through the condenser T, from V, and the burners D, were then lighted. The flame was adjusted by means of valves F and H. The still, A, was covered with a sheet of asbestos, and an electric pyrometer attached to C, inserted in the pipe B. The temperature and pressure were recorded every five minutes. When the pressure reached the desired maximum as shown at H, the screw, S, was turned up until the relief-valve began to discharge. Thus the pressure was definitely controlled. When the oil ceased to come over the heat was raised for a short time, and then discontinued. The resulting oil was then measured and its gravity recorded. It was then fractionated, and the gasoline portion saved for further tests.

Test No. 92

500 cc. of the kerosene was tested according to the foregoing procedure, using a maximum pressure of 70 pounds. The following is a record of the test.

| Time | Temperature | Pressure |
|---|---|---|
| 7:45 | 50° C. | 0 lbs |
| 7:50 | 200° C. | 0 lbs |
| 7:55 | 280° C. | 5 lbs |
| 8:00 | 340° C. | 10 lbs |
| 8:05 | 360° C. | 15 lbs |
| 8:10 | 380° C. | 20 lbs |
| 8:15 | 400° C. | 30 lbs |
| 8:20 | 420° C. | 40 lbs |
| 8:25 | 420° C. | 50 lbs |
| 8:30 | 420° C. | 65 lbs |
| 8:35 | 420° C. | 70 lbs |
| 8:40 | 420° C. | 60 lbs |
| 8:45 | 420° C. | 50 lbs |
| 8:50 | 420° C. | |
| 8:55 | 420° C. | 0 lbs |

| | Amount | Percentage | Gravity |
|---|---|---|---|
| Oil Used | 500 cc. | 100.0% | 35.5° Be. |
| Oil Received | 475 cc. | 95.0% | 42.0° Be. |
| Amount of Gasoline (Up to 200°C.) | 69 cc. | 13.8% | 52.0° Be. |
| Residue | 406 cc. | 81.2% | 35.0° Be. |
| Loss | 25 cc. | 5.0% | |

Test No. 93

Test No. 92 was repeated with the same maximum pressure. The following is the record.

| Time | Temperature | Pressure |
|---|---|---|
| 4:30 | 50°C. | 0 lbs |
| 4:35 | 210°C. | 0 lbs |
| 4:40 | 300°C. | 5 lbs |
| 4:45 | 330°C. | 10 lbs |
| 4:50 | 360°C. | 20 lbs |
| 4:55 | 390°C. | 35 lbs |
| 5:00 | 410°C. | 50 lbs |
| 5:05 | 420°C. | 65 lbs |
| 5:10 | 420°C. | 70 lbs |
| 5:15 | 420°C. | 50 lbs |
| 5:20 | 420oC. | 0 lbs |

| | Amount | Percentage | Gravity |
|---|---|---|---|
| Oil Used | 500 cc. | 100% | 35°Be. |
| Oil Recovered | 460 cc. | 92% | 43°Be. |
| Gasoline (Up to 200°C.) | 73 cc. | 14.6% | 51°Be. |
| Residue | 387 cc. | 77.4% | 35°Be. |
| Loss | 40 cc. | 8.0% | |

Test No. 94

The cracking test was repeated using 100 pounds as maximum pressure. The record follows.

| Time | Temperature | Pressure |
|---|---|---|
| 2:30 | 50°C. | 0 lbs |
| 2:35 | 190°C. | 0 lbs |
| 2:40 | 240°C. | 10 lbs |
| 2:45 | 280°C. | 20 lbs |
| 2:50 | 310°C. | 40 lbs |
| 2:55 | 340°C. | 60 lbs |
| 3:00 | 380°C. | 80 lbs |
| 3:05 | 420°C. | 100 lbs |
| 3:10 | 460°C. | 100 lbs |
| 3:15 | 460°C. | 80 lbs |
| 3:20 | 460°C. | 10 lbs |
| 3:25 | 460°C. | 0 lbs |

| | Amount | Percentage | Gravity |
|---|---|---|---|
| Oil Used | 500 cc. | 100.0% | 33.5°Be. |
| Oil Recovered | 460 cc. | 92.0% | 41.5°Be. |
| Gasoline(Up to 200°C.) | 100 cc. | 20.0% | 51.0°Be. |
| Residue | 360 cc. | 72.0% | 32.0°Be. |
| Loss | 40 cc. | 8.0% | |

## Test No. 95

Test 94 was repeated, using the same maximum pressure.

| Time | Temperature | Pressure |
|---|---|---|
| 11:05 | 50°C. | 0 lbs |
| 11:10 | 200°C. | 0 lbs |
| 11:15 | 280°C. | 0 lbs |
| 11:20 | 320°C. | 10 lbs |
| 11:25 | 340°C. | 30 lbs |
| 11:30 | 370°C. | 45 lbs |
| 11:35 | 400°C. | 60 lbs |
| 11:40 | 420°C. | 80 lbs |
| 11:45 | 460°C. | 95 lbs |
| 11:50 | 470°C. | 100 lbs |
| 11:55 | 470°C. | 90 lbs |
| 12:00 | 470°C. | 30 lbs |
| 12:05 | 470°C. | 0 lbs |

| | Amount | Percentage | Gravity |
|---|---|---|---|
| Oil Used | 500 cc. | 100.0% | 35°Be. |
| Oil Recovered | 465 cc. | 93.0% | 40°Be. |
| Gasoline ( Up to 200°C.) | 105 cc. | 21.0% | 51°Be. |
| Residue | 360 cc. | 72.0% | 33 Be. |
| Loss | 35 cc. | 7.0% | |

## Test No. 96

Test No. 94 was again repeated using the same maximum pressure. The record follows.

| Time | Temperature | Pressure |
|---|---|---|
| 3:40 | 50°C. | 0 lbs. |
| 3:45 | 200°C. | 0 lbs. |
| 3:50 | 250°C. | 10 lbs. |
| 3:55 | 300°C. | 30 lbs. |
| 4:00 | 340°C. | 50 lbs. |
| 4:05 | 380°C. | 75 lbs. |
| 4:10 | 400°C. | 90 lbs. |
| 4:15 | 440°C. | 100 lbs. |
| 4:20 | 450°C. | 80 lbs. |
| 4:25 | 460°C. | 0 lbs. |

| | Amount | Percentage | Gravity |
|---|---|---|---|
| Oil Used | 500 cc. | 100.0% | 35°Be. |
| Oil Recovered | 360 cc. | 72.0% | 39°Be. |
| Gasoline (Up to 200°C.) | 90 cc. | 18.0% | 51°Be. |
| Residue | 270 cc. | 54.0% | 35°Be. |
| Loss | 140 cc. | 28.0% | |

The large loss in this test was due to a leak which developed around the pressure gauge, near the end of the test.

Resumé of Cracking Tests.-

| Test | 92 | 93 | 94 | 95 | 96 |
|---|---|---|---|---|---|
| Oil Used, cc. | 500 | 500 | 500 | 500 | 500 |
| Gravity, Be. | 35.5 | 35.0 | 33.5 | 35.0 | 35.0 |
| Gasoline Recovered, cc. | 69 | 73 | 100 | 105 | 90 |
| Percentage | 13.8 | 14.6 | 20.0 | 21.0 | 18.0 |
| Gravity, °Be. | 52.0 | 51.0 | 51.0 | 51.0 | 51.0 |
| Maximum Pressure | 70 | 70.0 | 100 | 100 | 100 |
| Maximum Temperature | 420 | 420 | 460 | 460 | 460 |
| Loss,[1] % | 5.0% | 8.0 | 8.0 | 7.0 | 28.0 |

Average of Cracking Tests. (Test 96 is omitted.) These are averaged for the maximum pressure used.

| | | |
|---|---|---|
| Maximum Pressure | 70 | 100 |
| Maximum Temperature | 420oC. | 460°C. |
| Gravity of Oil Used | 35.25°Be | 34.25°Be |
| Gasoline Recovered, cc. | 71 | 102.5 |
| Percentage | 14.2 | 20.5 |
| Gravity | 51.0oBe. | 51.0°Be |
| Loss | 6.5% | 8.5% |

## Test No. 97

The gasoline recovered from the foregoing tests was recovered and subjected to a distillation test in the same manner as Test 75. The results follow.

| Temperature<br>Centigrade | <br>Fahrenheit | Amount of oil<br>Distilled Over | Difference in<br>Percentage |
|---|---|---|---|
| 57 | 134 | First drop | |
| 100 | 212 | 17 | 17 |
| 110 | 230 | 22 | 5 |
| 120 | 248 | 28 | 6 |
| 130 | 266 | 37 | 9 |
| 140 | 284 | 46 | 9 |
| 150 | 302 | 55 | 9 |
| 160 | 320 | 67 | 12 |
| 170 | 338 | 79 | 12 |
| 180 | 356 | 90 | 11 |
| 190 | 374 | 98 | 8 |
| 200 | 392 | 100 | 2 |

The results of this test are also plotted in figure 5, with the distillation test of the straight-run gasoline.

## TEST ON GAS OIL

Nothing was done with the gas oil, except a few tests of decolorization by filtration. These all proved negative and are not recorded. As stated before, the gas oil, would be used to enrich water-gas, or would be cracked for the pro-

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[1] Cross in Bulletin 14, Kansas City Testing Laboratories, calls this "shrinkage".

duction of gasoline

## TESTS ON LUBRICANTS

The lubricating oil fractions were refined by treating with 4% of concentrated sulphuric acid then with 6% of caustic soda. This was done at a temperature of 40°C. (94°F.), which was maintained by means of a water jacket around the agitation flask. After this treatment the oil was heated for two hours on a water bath with 20% of Florida earth, and then filtered hot.

The light lubricating oil was noticeably lighter in color than before this treatment, being a deep cherry red, while the heavy lubricating oil was still black, appearing red only in thin layers. These oils were then subjected to the ordinary tests to determine their properties.

Test No. 98

Light Lubricating Oil
Gravity--31.0°Be. (.8696)
Flush Point--220°F.
Burning Point--240°F.
Viscosity by the Saybolt Universal Viscosimeter.
at 60°F.,-53, at 100°F.,-44.5, at 200°F.,-34.6

Test No. 99

The light lubricating oil was subjected, to a friction test on a Tinius Olsen friction machine. The recor
of the test follows-
Journal- Steel Bearing - Brass.
Area of bearing,-7 sq.in. - Total load on bearing-300 lbs.
Pressure per square inch.-42.9
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 results of test are plotted in figure 7.

### Test No. 100

The heavy lubricating oil was tested for its properties. Results are as follows.

Gravity-19.7°Be (.9352
Flash Point-448°F.
Burning Point-490°F.
Viscosity by the Sayboldt Universal Viscosimeter at
60°F.-Too thick to test. 100°F.-3340 200°F.-732

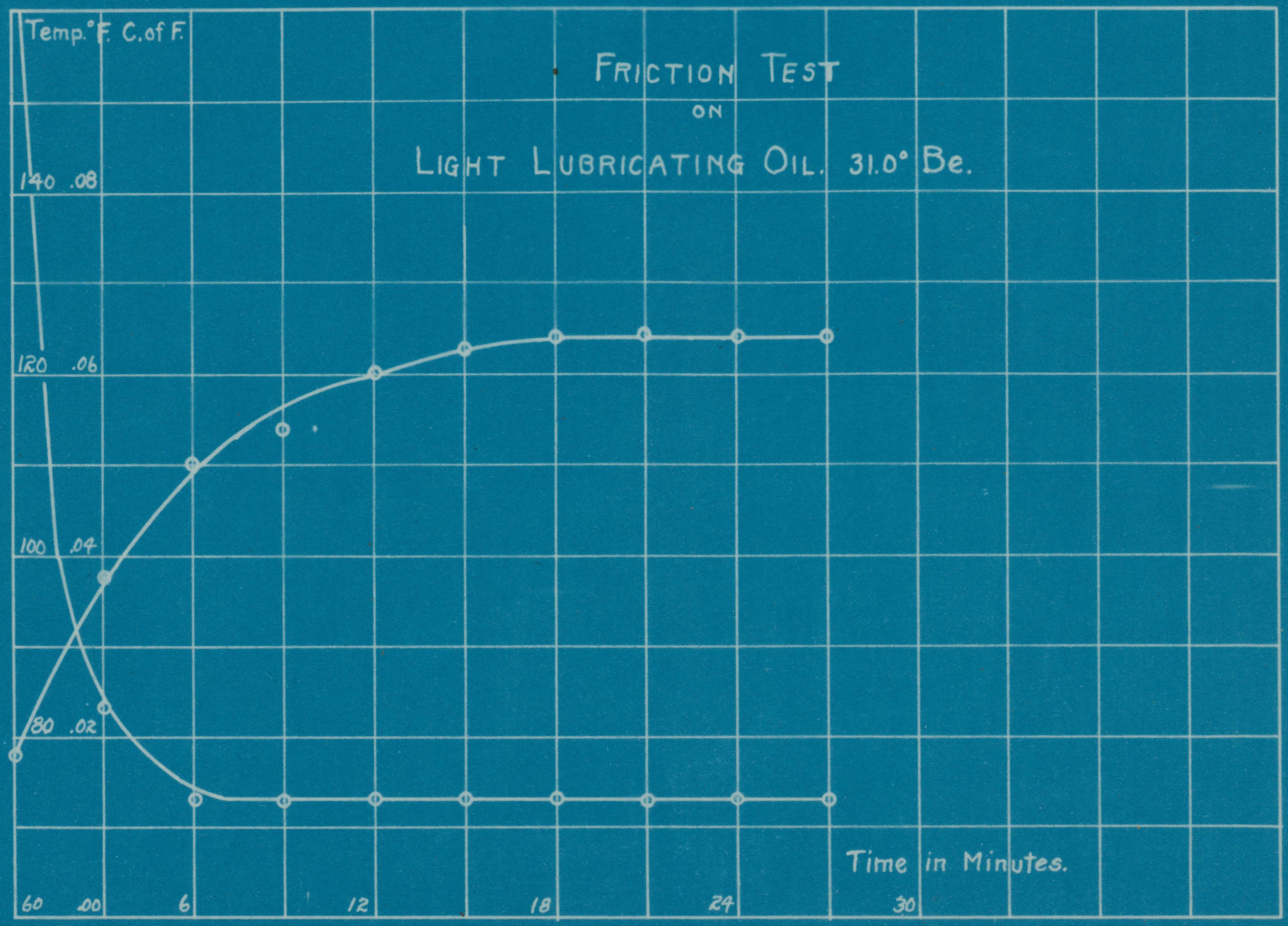
### Test No. 101

This heavy oil was then tested on a Tinius Olson friction machine, and the following results recorded.
Journal - Steel Bearing - Brass
Area of Bearing-7 square inches. Total Load on Bearing 300 pounds. Pressure per square inch.-42.9
Revolutions per minute of Journal - 800.

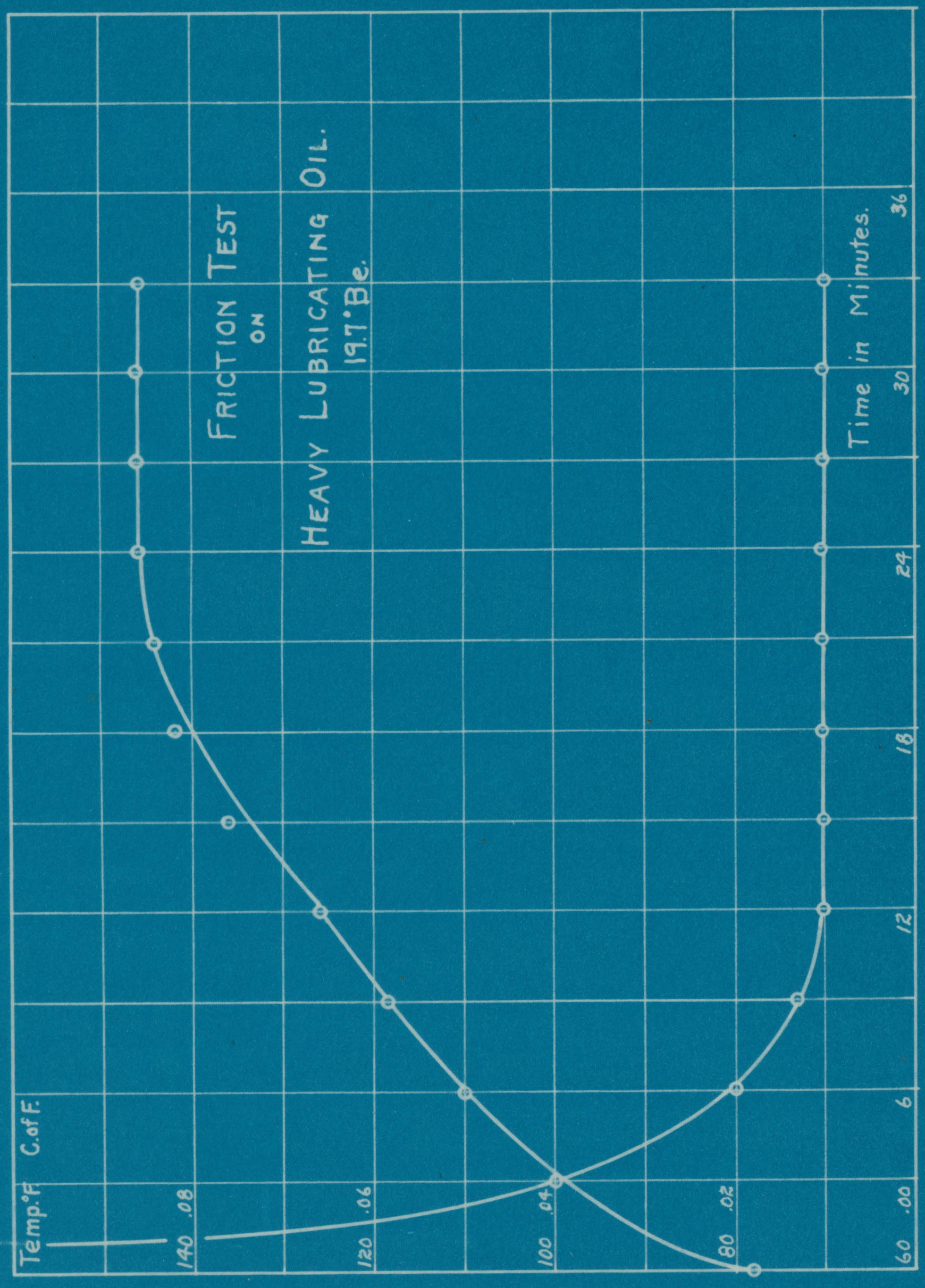
| Time | Temperature of Bearing | Load | Friction | |
|---|---|---|---|---|
| | | | Total | Coefficient |
| 7:08 | 78 | 300 | 60 | 0.2 |
| 7:11 | 100 | 300 | 12 | 0.04 |
| 7:14 | 110 | 300 | 6 | 0.02 |
| 7:17 | 118 | 300 | 4 | 0.013 |
| 7:20 | 126 | 300 | 3 | 0.01 |
| 7:23 | 136 | 300 | 3 | 0.01 |
| 7:26 | 142 | 300 | 3 | 0.01 |
| 7:29 | 144 | 300 | 3 | 0.01 |
| 7:32 | 146 | 300 | 3 | 0.01 |
| 7:35 | 146 | 300 | 3 | 0.01 |
| 7:38 | 146 | 300 | 3 | 0.01 |
| 7:41 | 146 | 300 | 3 | 0.01 |

Maximum temperature-146°F.
Minimum Coefficient of Friction-0.01
The results of this test are plotted in figure 8.

# PLATE VII.


FRICTION TEST
ON
LIGHT LUBRICATING OIL. 31.0° Be.
Temp. °F. C. of F.
140 .08
120 .06
100 .04
80 .02
60 .00
6
12
18
24
30
Time in Minutes.

# PLATE VIII.



FRICTION TEST ON HEAVY LUBRICATING OIL. 19.7° Be.

## BY-PRODUCTS

So much has been said of the various by-products obtained from oil-shale that a few remarks concerning them should be given here. A great many advertisements of the companies exploiting the shales give a long list of by-products, which they claim can be easily taken from the shales. This, however, is not the case. The products are comparatively few, and the only justification for the long lists of products is the fact that there are derived from the shale certain substances which, are used in more or less elaborate processes, for the manufacture of certain important drugs or chemicals. For example, a small percentage of benzene is found in the gasoline, but the expense of nitrating it and removing it from the gasoline would probably be so great that the product could not compete, as yet, with coal-tar benzene. Many other instances could be given.

The more important by-products are, (1) ammonium sulphate, (2) paraffin wax, (3) potash, (4) petroleum coke, and, (5) the spent shale. Of these, the coke has been mentioned.

The ammonium sulphate is valuable as a fertilizer. It can easily be obtained by treating the "ammonia water" with sulphuric acid, and separating the product by crystallization. It would also be obtained in the ammonia scrubber. The cost of recovering this substance is very slight, as the process is extremely simple.

Paraffin wax can easily be obtained by any of the commercial methods, from the heavier oils. No specific tests were made on this, but a pure white wax was obtained, which seemed to have a rather high melting point.

The spent shale can be used for a pigment in such paints as roofing paints, protective coatings for iron and steel, and other places where an inert mineral constituent is desirable. The coke would be black on account of the carbon, and the mineral content would give it the necessary wearing qualities. The spent shale also contains about 1% of water-soluble potash, which could easily be lixiviated at a very small cost.

## SUMMARY AND CONCLUSION

In this work, a quantity of the oil-shale was distilled, the resulting crude oil fractionated, and the various fractions tested.

The gasoline was subjected to the action of different acid and basic substances, both with and without the aid of heat. It was also treated with oxidizing and reducing agents, and with different filtering mediums. A slight amount of sulphur was found in the gasoline, but it was proven that this did not cause the color. No nitrogen was found, so substances such as pyridine and pyrrole were absent. Terpenes and derivations of furfural were tested for, but not found. The best method of treatment found was by distillation over 20% of common salt, then agitation with 2% of concentrated sulphuric acid and 4% of caustic soda (14° Be) successively. This yielded a gasoline which was light cherry-red in color.

This gasoline was tested and found to have no effect on iron or brass, which had been left in it for two weeks. In heating value, this gasoline was found to be up to the standard of ordinary gasoline, although lower in gravity. This, however, does not give any indication of the comparative power value when used in an internal combustion engine. By the distillation test, it was shown that this gasoline had about the same proportion of low boiling constituents as ordinary gasoline, thus insuring easy ignition. When burned side by side in the open air, no difference could be noticed between the gasoline from shale and from ordinary petroleum.

These tests would indicate that the gasoline from shale is as good, in every respect, as the gasoline from petroleum, although its color might be objectionable.

The kerosene derived from shale was proved to be valuable as the basis for cracked gasoline. The tests show that by distilling at 100 pounds pressure, 20% of the kerosene is converted into gasoline, which has even a higher percentage of low-boiling products than the straight-run gasoline. It is probable that even higher recovery could be obtained under greater pressure.

The gas oil would also be used in cracking, and would probably yield a larger percentage of gasoline than did the kerosene fraction.

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