May 31, 1932. E. H. LESLIE 1,860,838

METHOD OF PREPARING LUBRICANTS

Filed March 7, 1928 3 Sheets-Sheet 1

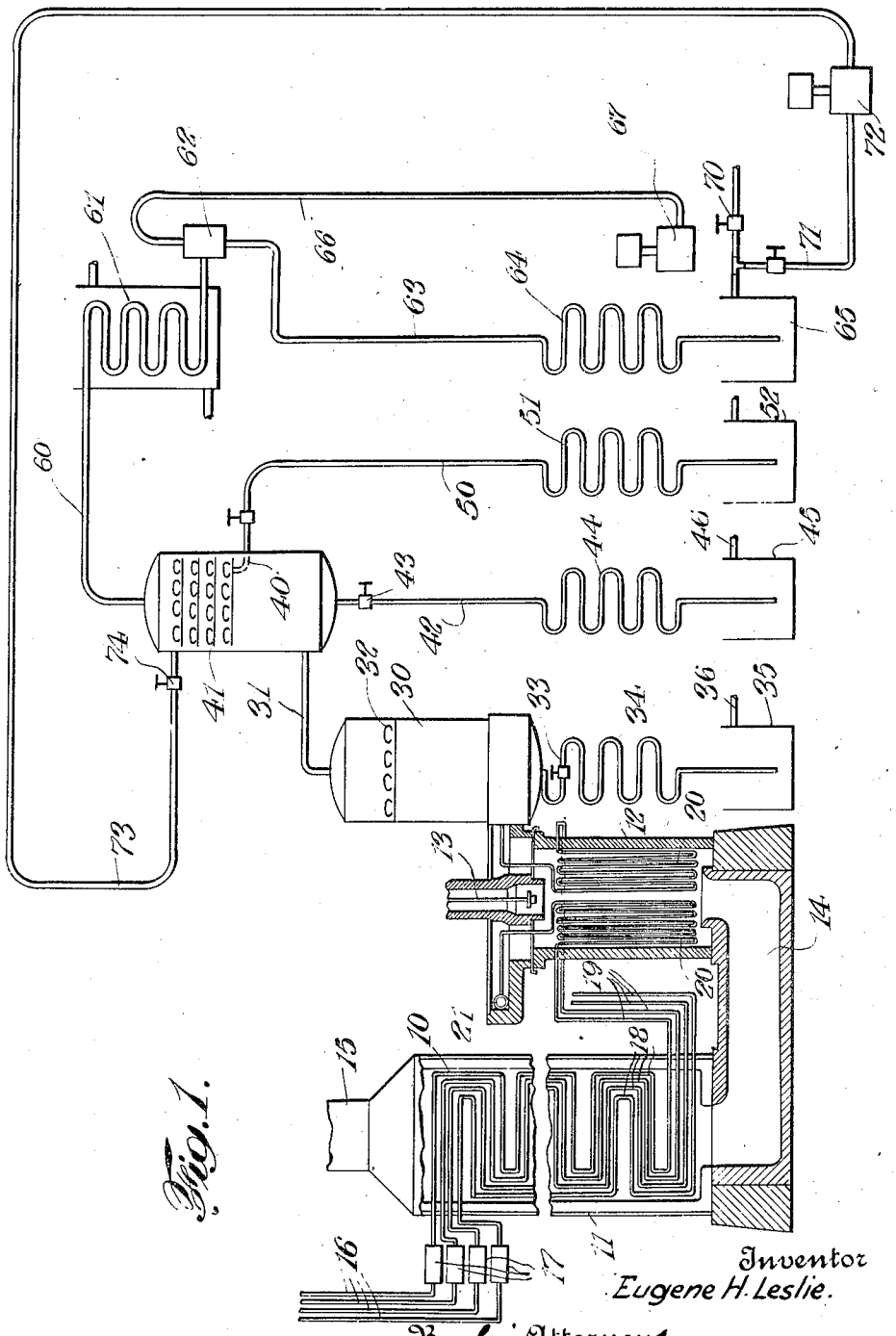


Fig. 1.

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Filed March 7, 1928 3 Sheets-Sheet 2

Fig. 2.

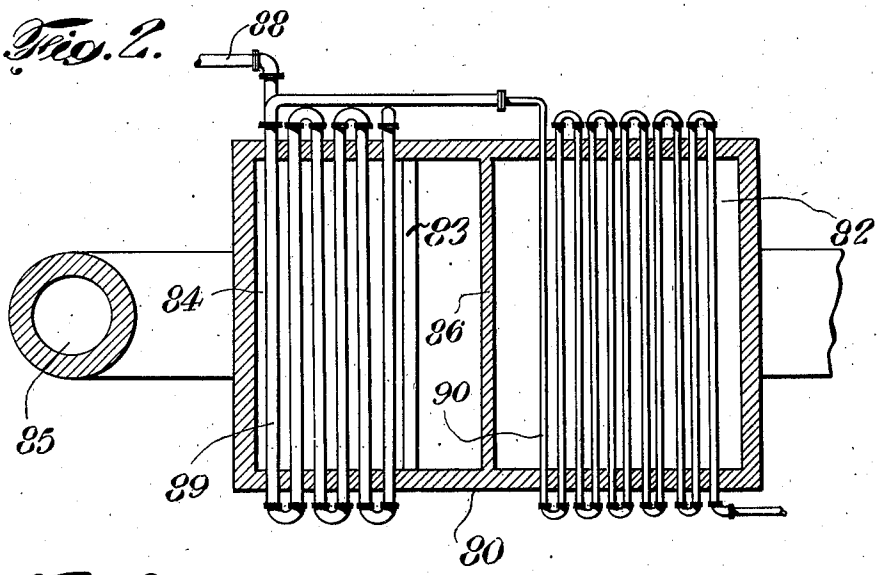
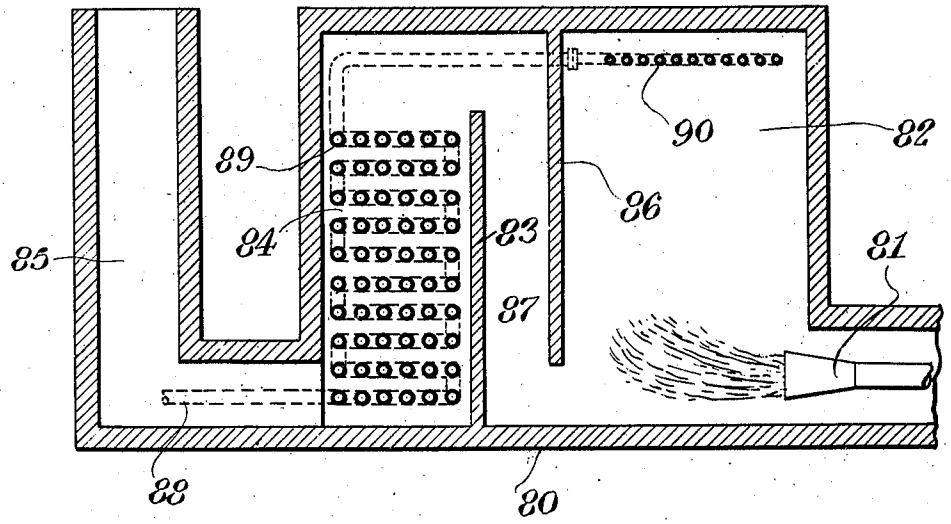


Fig. 3.



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Filed March 7, 1928 3 Sheets-Sheet 3

Fig. 4.

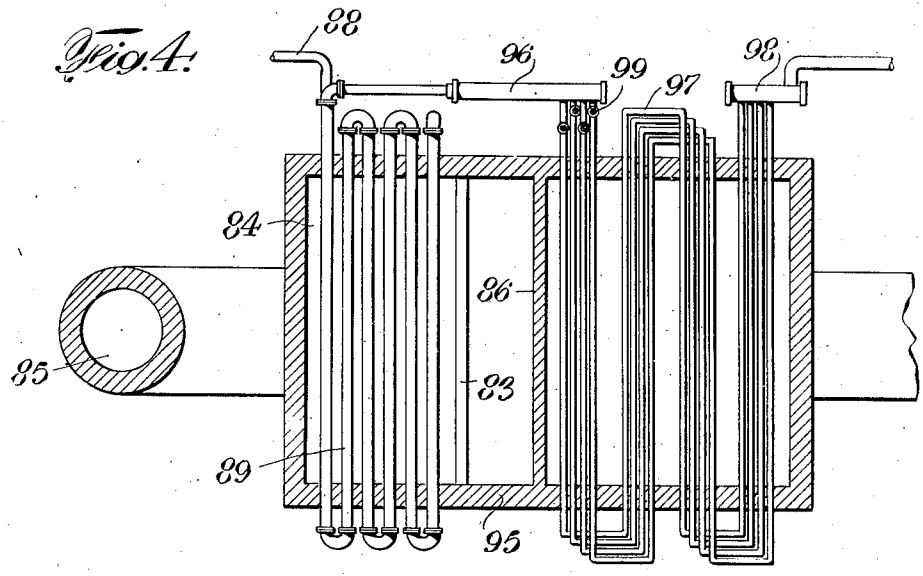
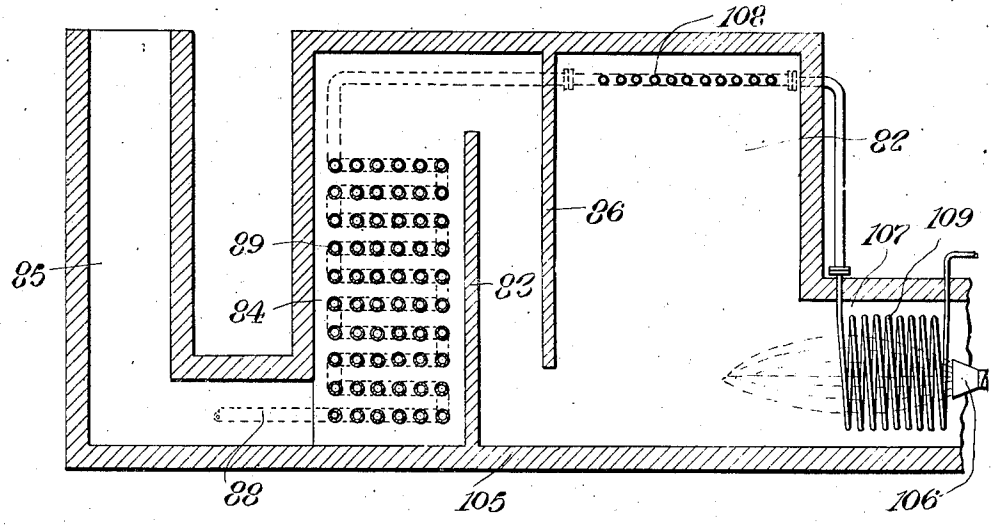


Fig. 5.



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UNITED STATES PATENT OFFICE

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METHOD OF PREPARING LUBRICANTS

Application filed March 7, 1928. Serial No. 259,694.

My invention relates to a method for economically securing a high yield of lubricants from a petroleum or like material.

Many crude petroleum materials from which lubricants are derived contain a considerable amount of tarry or asphaltic materials, which are either practically nonvolatile or have boiling points higher than those of the highest boiling lubricants. These tarry or asphaltic materials must not be present in the finished lubricants and in order to remove them by distillation the temperature must be sufficiently high to distill and separate from them the desired lubricants. The lubricants of the highest viscosity which in many instances are the most valuable, are those having the highest boiling points, and it is therefore desirable to use a maximum distillation temperature and at the same time to avoid substantial cracking.

It is well known that the rate of cracking is a function of temperature, the amount of cracking being generally directly proportional to the time and increasing rapidly with increase in temperature. Extended researches have been made by others to determine the time and temperature factors contributing to a maximum amount of cracking, as this information has been of great importance to the tremendous industry of producing gasoline by cracking. I am concerned on the other hand with obtaining a minimum of cracking of the lubricating portions of a material from which lubricants are derived, in order to prevent loss of the lubricants, and at the same time with utilizing temperatures that will permit distilling a maximum of lubricants in an economical manner. The precise influence of the factors of time and temperature on the loss of lubricants by cracking has not been previously determined, and the method of this invention is based on careful investigation and experimental work in this field.

In the ordinary present day commercial practice, the yields of lubricants obtained from crude petroleum are low because of plant procedures that destroy the viscous oil by thermal decomposition or cracking. Usually one or more distillations from large shell stills are employed, and the petroleum is heated to a relatively high temperature for a period of many hours, thereby causing cracking in the body of the oil. Moreover, the temperature of the oil close to the heating surfaces is necessarily materially higher than that of the body of the oil, and hence considerable cracking takes place in the relatively quiescent film next to the heating surfaces. Endeavors have been made to reduce the decomposition of the lubricants by reducing the temperature or time of distillation or both. One process employs shell stills and lowers the temperature required by distilling under a low absolute pressure. The long time of heating and the film overheating effect referred to above, are necessary consequences of the use of shell stills, hence it follows that it is essential, if any substantial cracking is to be avoided, to maintain a very low absolute pressure, thereby permitting the use of a relatively low temperature. The use of shell stills under such very low absolute pressures involves relatively high initial installation costs and operating expenses. Another expedient in common use is to distill in shell stills and to inject steam into the oil. The effect of the steam is to lower the distillation temperature in accordance with well known principles. In addition to the disadvantages of a long time of heating and overheating of the oil next to the heating surfaces, such practice involves a relatively high cost for steam and the use of large condensing surfaces.

Tubular heaters or pipe stills have recently been used in the distillation of lubricating oils. In these the material to be distilled is forced by pumps or otherwise through tubes heated externally, the time of

heating and therefore the amount of cracking being less for any given temperature than in shell stills at the same temperature. In all such heaters, however, so far as I am aware, the viscous material or lubricant has been subjected to a time of heating such that substantial cracking of lubricants results when raising the temperature to a point where the desired amount of lubricant will be distilled. In some cases steam has been introduced into the heating tubes to lower the distillation temperatures, and to increase velocity, thus decreasing the time element. In one installation a tubular type of heater has been employed in which the tubes are not filled with the material being distilled and provision is made for the removal of vapor as formed.

In contrast to present practice, my procedure is designed to secure larger yields of lubricants economically from material containing the same by distillation in such manner as to prevent any substantial amount of cracking. By my process, average distillation temperatures higher than those that have previously been considered possible may be employed without diminishing the yield of lubricants or impairing their viscosity.

According to my invention, I heat the lubricant containing material in a tubular heater to the temperature required to vaporize the desired lubricating fractions, which temperature is normally considerably above the cracking temperature of the material, in such a manner that the period of time during which the material is subjected to temperatures above its cracking temperature is too short to permit any substantial cracking to take place, and that the oil next to the heating surface is not heated to a temperature considerably exceeding that of the body of the oil. After heating the material to the required temperature I subject it to flash vaporization in a suitable vaporizer or flash chamber. I have demonstrated by complete experimental work the advantages of flash vaporization as compared with incremental or stepwise vaporization. By means of flash vaporization approximately 30% more lubricants can be vaporized by heating to any given temperature than by stepwise vaporization, or the same percentage can be vaporized by heating to a lower temperature. Furthermore, at a given temperature the desired lubricants will be more sharply separated from the viscous bottom by flash vaporization than by stepwise vaporization. To reduce the temperature required to vaporize a large part or substantially all of the viscous oils I preferably operate under a vacuum as described more fully hereinafter. After separating the desired lubricants from the heavy residues by flash vaporization, I preferably employ counter current fractionation in a suitable plate column to separate the vapors from the vaporizer into the desired fractions. The use of a plate fractionating column is essential to secure the highest yields of lubricants and the advantages thus obtained will be pointed out in detail hereinafter.

My process is particularly advantageous in connection with the treatment of wax-bearing oils, and an important feature of my invention resides in the treatment of these oils in such a manner as to secure in a single operation a maximum amount of so-called pressable wax distillate. In the commercial shell still processes heretofore employed, the wax distillate cut either does not contain the wax in a form suitable for pressing or is divided into two portions during distillation, the first portion to go overhead being pressable, and the second and usually larger portion being non-pressable. The customary procedure is to redistill the wax distillate or the non-pressable part thereof in shell stills under cracking conditions, whereby a secondary wax distillate, which is pressable, is produced. Obviously, however, this procedure is wasteful of heat and destroys some of the valuable lubricants. According to my invention, on the other hand, as will appear from the detailed description given hereinafter, a larger yield of pressable wax distillate may be obtained directly from the original distillation, which is carried out under substantially non-cracking conditions as above described.

These and such other objects and advantages of my invention as may hereinafter appear will be best understood in connection with a description of suitable apparatus for carrying out my process, such as is illustrated diagrammatically in the accompanying drawings, which form a part of this specification and in which;

Figure 1 is a diagrammatic side elevation, partially in section, of a complete plant adapted to carry out my process;

Figure 2 is a horizontal section through a tubular heating unit which may be used in place of the heating unit shown in Figure 1;

Figure 3 is a central vertical section through the heating unit shown in Figure 2;

Figure 4 is a horizontal section through another form of heating unit; and

Figure 5 is a central vertical section through still another suitable form of heating unit.

Referring more particularly to Figure 1 of the drawings, 10 indicates generally a suitable tubular heater or pipe still for bringing the lubricant containing material to the desired distillation temperature. The pipe still 10 comprises a preheating or economizer section 11 and a high temperature or radiant heat section 12. The radiant heat section 12 is preferably constructed in the form of an upright cylinder and is provided at its upper

end with a centrally located oil burner 13. The hot products of combustion from the burner 13 pass downwardly through the center of the radiant heat section 12 and thence through the flue 14 to the economizer section 11 and out through the stack 15. In the embodiment shown in this figure, the oil to be heated is divided into a plurality of independent streams, four of such streams being illustrated. The oil is picked up from each of the four supply pipes 16, connected to a source of supply not shown, by a separate pump 17, and forced through a heating coil 18 in the economizer section 11. The four independent heating coils 18 are horizontally disposed in direct contact with the products of combustion. If desired, the oil may be preheated in suitable heat exchangers (not illustrated) before being introduced into the economizer section 11. From the economizer section, each of the four streams of oil is separately conducted through a line 19 to the radiant heat section 12, where it passes through a vertically disposed heating coil 20 arranged circumferentially around a portion of the inner wall of the radiant heat section. The four coils 20 taken together form a hollow vertical cylinder. By this construction, the hot products of combustion from the burner 13 pass centrally downward through the radiant heat section, and transmit their heat thereto mainly by radiation. The products of combustion are cooled materially before they come into direct contact with the heating coils located in the economizer section 11, thus preventing the burning or oxidation of the heating tubes. The ends of the four coils 20 in the radiant heat section 12 are all connected to the common outlet header 21 disposed above the radiant heat section.

I preferably design and operate the pipe still 10 in such manner that the oil undergoing treatment does not reach a cracking temperature until after it enters the radiant heat section 12. In other words, for a typical lubricant containing petroleum oil the maximum temperature reached in the economizer section 11 will be in the neighborhood of 600° F. It is thus feasible to heat the oil relatively slowly in the preheating or economizer section. Therefore, I employ relatively large heating tubes in the economizer section 11, thereby securing a cheaper and more durable construction and lessening the pressure required for forcing the material through the heating tubes in the economizer section.

The heater is preferably so designed that the material is under pressure during substantially all of its passage through both sections of the pipe still. The pressure at the pump may range from 200 to 500 lbs. Under these conditions the oil is probably in the liquid phase throughout the greater part of the heating tubes, although some vapor may exist in the portion of the tubes near the outlet header 21.

Although my method is independent of any specific design of tubular heater, I employ a pipe still the high temperature section of which is especially designed to obtain the necessary distillation temperature for the desired percentage of vaporization with a minimum amount of cracking. The final maximum temperature which must be obtained depends of course upon the character of the material and the percentage thereof which it is desired to vaporize. The first requirement in the design of the heater obviously is that the area of the heating surface must be large enough so that the amount of heat required to raise the oil to the desired distillation temperature can be put into the oil.

The maximum time during which the material is above its cracking temperature may be determined by dividing the material capacity of the portion of the heater in which the material is above its cracking temperature, in this case the radiant heat section 12, by the amount of said material which passes through this portion of the heater in a unit of time. For instance, if the material capacity of the radiant heat section 12 should be thirty gallons and the material put through one gallon per second, it would require a maximum of thirty seconds for the material to pass through this portion of the pipe still. Throughout this specification I have defined the time during which the material is held at a temperature above its cracking temperature in this manner, disregarding such variations as may result from the formation of gas or vapor.

The permissible time for this final stage of the heating depends upon the character of the material undergoing treatment and the maximum temperature to which it is to be heated. I have found that when a distillation temperature of about 850° F. is desired, it is possible to avoid substantial cracking if the oil is raised from its cracking temperature, or approximately 650° F., to the maximum temperature in about 60 seconds. A longer time is permissible if the distillation temperature is lower, and vice versa. However, in order to secure the best results, I prefer not to heat the oil at temperatures above its cracking temperature for more than 35 seconds.

I have further found that the velocity at which the material passes through the heating tubes is of great importance, and that this velocity must be high. In order to prevent any substantial cracking of the oil being heated, it is necessary that no portion of the oil stream be materially higher in temperature than the main body of the oil. The velocity of flow determines to a large extent the temperature gradient between the oil close to the tube walls and that at the center of the tubes. A high velocity of flow ap-

parently sets up a turbulent condition which aids in preventing any great differences of temperature in the oil stream.

There are thus three major factors which I consider in the design of the tubular heater: First, the area of the heating surface must be great enough so that the required amount of heat can be put into the oil; second, the material capacity of the heating tubes must be sufficiently small so that the time of passage of the oil will be 60 seconds or less; and, third, the velocity of the oil through the tubes must be such that high film temperatures on the tube walls are avoided. At the same time, care must be taken that the velocities and tube lengths used are not such as would give an excessively high pressure drop through the heater.

As a result of my experimental work, I have found that for commercial size plants, tubes of 1½″ inside diameter or less are desirable, and that velocities of 10 feet per second or over must be used. For large units in which a single tube of the required area would be so long that the pressure drop would be excessive, I may use several tubes in parallel.

The oil after being heated to the required final temperature is discharged into the flash vaporizer 30, which may be, as shown in Figure 1, interposed in the periphery of the outlet header 21. The material entering the vaporizer 30 is above its boiling point at the pressure existing therein, and a substantial portion of the material will therefore immediately flash into vapor, which leaves the vaporizer through the overhead pipe 31. One or more baffle plates 32, which may be of the usual bubble tower type, are, if desired, placed in the top of the vaporizing chamber 30 to prevent entrainment of liquid in the vapors leaving the chamber. I have illustrated the flash vaporizer 30 only diagrammatically as any suitable type thereof may be employed.

The liquid residue remaining in the vaporizer 30 may be drawn off through the valve 33 into a cooling coil 34, which discharges into a receiving tank 35 provided with a drawoff pipe 36.

The vapor leaving the vaporizer through the pipe 31 may be led to a suitable fractionating column 40 where the lubricating stock may be cut into fractions of desired viscosity and, if desired, the lighter non-viscous material may be stripped from the viscous lubricants. The column 40 is provided with a series of plates 41 which may be of the usual bubble tower type. The heavy lubricant collecting in the bottom of the fractionating column 40 may be drawn off through the pipe 42 controlled by the valve 43 into a cooling coil 44. The cooling coil 44 discharges into a receiving tank 45 provided with a drawoff 46.

One of the fractionating plates 41 is provided with a valve controlled intermediate drawoff pipe 40 which is connected to a cooling coil 51 discharging into a receiving tank 52. It will be understood that more than one intermediate drawoff such as 50 may be provided if desired.

The overhead vapors from the fractionating column 40 are conducted through the pipe 60 to a condenser 61. The condenser 61 discharges into a trap 62 from which the condensate is conducted through a pipe 63 to a cooler 64, discharging into a receiving tank 65. The vapor space of the trap 62 is connected by the pipe 66 to a vacuum pump 67, by means of which the desired low absolute pressure may be maintained in the vaporizing system.

The receiver 65 is provided with a valve controlled drawoff pipe 70 and also with a valve controlled pipe 71 leading to a pump 72, which pumps the desired quantity of condensate through the line 73 into the top of the fractionating column 40 to provide the necessary reflux to secure the proper operation of the fractionating column. The valve 74 may be used to control the amount of reflux introduced into the column.

The manner of carrying out the flash vaporization and the counter current fractionation steps of my method depends, to a considerable extent, upon the character of the oil being treated. A broad line of division may be made between the handling of non-wax-bearing oils and of oils containing wax, and the latter classification may be sub-divided again into those of mixed base and those of paraffin base.

In the treatment of non-wax-bearing oils, a temperature in the vaporizer 30 is preferably maintained such that the residue remaining therein and drawn off through the valve 33 forms but a small proportion of the stock charged, and consists largely of tarry, asphaltic, gummy, and coloring substances. The viscous stocks vaporized overhead and recovered in the bottom of the fractionating column 40 are oils of good color as well as of high viscosity and may be sold as cylinder stock without filtration and with very little refining treatment. In previous processes in which cylinder stocks have been made from various oils, on the contrary, these stocks or the most viscous lubricants remain as a still residue, and filtration or a heavy treatment with sulphuric acid, or both, are usually necessary, particularly in the production of bright stocks from mixed base oils such as Mid-Continent oils.

Non-wax-bearing oils may be cut in the fractionating column 40 directly into fractions of the desired viscosity. Thus, for example, gas oil and other non-viscous products may be taken overhead into the pipe 60, a light lubricating oil may be drawn off

through the pipe 50, and a heavy lubricant from the bottom of the column through the pipe 42. If the charging stock contains only enough gas oil or other light fractions to obtain an overhead product of the desired viscosity, a light lubricating material may be taken overhead from the column 40 and collected in the receiving tank 65.

Most present day commercial methods of lubricating oil manufacture are, I believe, based on the method of simple distillation or differential vaporization with little or no fractionation of the vapor so formed. Most stills have no fractionating devices or even partial condensers. In actual practice, the lubricating oil in the still may contain considerable gas oil. The presence of this gas oil lowers the viscosity of the lubricating oil, not only in proportion to the concentration of the gas oil but considerably more because viscosities are not additive. In other words, blending equal parts of two oils, one of 200 viscosity and the other of 100 viscosity will give a blend not of 150 viscosity but of considerably less. In the reduction of petroleum oils to required viscosity by simple distillation in a shell still, the distillation must be carried to such a point that the gas oil concentration in the still is low, which results, therefore, in a large loss of valuable lubricating oil in the gas oil vapors normally passed overhead. By employing a fractionating column according to my invention, on the other hand, I can cleanly strip the lubricating stock from inclusions of non-viscous lighter oils which may be separated overhead without loss of valuable lubricants, and can sharply separate heavy viscous lubricants from those of lower viscosity.

In the manufacture of lubricants from wax-bearing oils it is, of course, essential to separate the wax from the lubricating oil in some manner. The wax present in petroleum oils may be classified according to its state, as: (*a*) amorphous wax, (*b*) crystalline wax not in pressable form, and (*c*) crystalline wax in form suitable for pressing. In this classification amorphous wax is wax the crystal growth of which has been practically completely prevented by inhibiting substances. These substances are heat sensitive and can be destroyed by cracking. They are high boiling and can be removed from the lower boiling lubricating fractions by counter current fractionation. As an example of amorphous wax it may be noted that the well known "petrolatum" of commerce contains a large part of its wax in the amorphous or non-crystalline state. By crystalline wax not in pressable form is meant wax the crystal growth of which has been partially inhibited, that is, to such an extent that the crystals cannot easily be removed by filtration in a filter press. Amorphous wax and crystalline wax not in pressable form are referred to collectively in this specification as non-pressable wax. Pressable wax, as the term is used herein, is wax in the form of crystals that deposit on the press cloth and on the frame of the press to form a reasonably open crystal mass through the interstices of which oil can flow.

In handling wax-bearing lubricating oils the usual shell still practice is to distill off successively the gasoline, kerosene and gas oil and then to take overhead a so-called wax distillate, the different fractions being conducted to separate receiving tanks. The distillation is usually conducted at atmospheric pressure, steam being employed during the later stages to avoid excessive cracking. The wax distillate is ordinarily collected as a single fraction, but in some cases it is divided into two cuts, the first cut to go overhead being usually much smaller than the second. Where a single wax distillate cut is made, it is not pressable in the form in which it is originally obtained, and the same is true of the second cut where two cuts of wax distillate are made. This non-pressable wax distillate must be redistilled to secure a pressable wax distillate. In redistilling this wax distillate, which operation like the original distillation is usually carried out at atmospheric pressure, the heat treatment is intentionally made such that considerable cracking takes place. The substances inhibiting wax crystal growth are heat sensitive and are destroyed to such an extent that wax crystals of reasonable size will grow in the so-called "crystallized wax-distillate" taken overhead from the "crystallizing" still. This cracking operation has heretofore always been considered essential to convert the wax in the wax distillate into pressable form. The redistilled wax distillate contains the wax in pressable form and the still residues remaining from this second distillation is commonly sold as a cheap lubricant or black oil. The wax is removed from the pressable wax distillate by filtering through a filter press and the resulting pressed distillate is then redistilled into cuts of suitable viscosities or boiling points. It will be understood that the wax distillate contains sufficient gas oil so that its viscosity is low enough to enable it to be readily run through the usual filter presses.

I have discovered that substantially all wax-bearing oils contain certain wax crystallization inhibitors that tend to prevent the crystallization of wax that would otherwise crystallize in pressable form, and that it is the presence of these wax crystallization inhibitors that renders the original wax distillate in the usual shell still operation non-pressable. I have further discovered that these wax crystallization inhibitors are high boiling substances that can be separated from the wax distillate by counter current frac-

tionation, thus making it possible to obtain directly a pressable wax distillate without the step of redistilling and cracking. For example, traces of high boiling, resinous, tarry or asphaltic substances in the wax distillate may act as wax crystallization inhibitors and prevent obtaining the crystallizable wax in pressable form. These high boiling substances appear to interfere with the growth of the wax crystals by retarding the movement of the crystallizable wax molecules in the solution and by coating the wax crystal nuclei. Moreover, these same inhibiting substances or other high boiling substances in the oil increase the solubility of wax in oil and oil in wax, thus interfering with the separation and proper growth of wax crystals.

According to my invention wax crystallization inhibitors are partially left in the residue of the vaporizer. The vapors leaving the vaporizer are subjected to counter current fractionation in the fractionating column as described and the high boiling, resinous, tarry and asphaltic substances are removed from the wax distillate containing the crystallizable wax, thus obtaining a maximum yield of pressable wax distillate in a single operation. It will be apparent that by my process I avoid the destruction of valuable lubricants by cracking and also avoid the heat loss incident to a second distilling operation. Furthermore, I am enabled to get a sharper separation between the crystallizable wax and the amorphous wax than is possible in prior processes. This permits me in the primary distillation to obtain a larger part of the oil as directly pressable wax distillate than is ordinarily obtained as pressable distillate after the primary and crystallizing distillations. Since pressing is a less expensive operation than either of the other well known methods of wax removal, namely, cold settling and centrifuging, it is, of course, desirable to secure as large a proportion of the oil as is possible in the form of pressable wax distillate.

It will be seen that my process possesses three major advantages over the prior art processes in the treatment of wax-bearing oils, first, substantially no cracking takes place during the original distillation of the oil; second, the necessity of redistilling and cracking to produce a pressable wax distillate is eliminated; and third, a clean separation among the various cuts may be obtained. Each of these advantages assists in obtaining a final maximum yield of valuable lubricants.

When a mixed base oil containing wax is being treated, the vaporizer residue will be relatively small as described in connection with the treatment of non-wax-bearing oils, and will contain the asphalt and a large part of the tarry wax crystallization inhibitors. The fractionating column bottoms will comprise a heavy lubricant containing the high boiling, non-pressable waxes, but the larger proportion of the wax present in the crude will be contained in the light fraction drawn off through the pipe 50. This fraction is the so-called pressable wax distillate and the wax is subsequently removed therefrom by pressing. Low cold test bright stocks may be obtained from the bottoms collected in the tank 45 by cold settling or centrifuging this heavy lubricant in the manner well known to those skilled in the art.

Turning now to paraffin-base wax-bearing oils, it is usually advisable to permit a larger percentage of the charging stock to remain in the vaporizer residue. This fraction will then contain substantially all of the high boiling amorphous wax or mineral jelly referred to above, and in addition will contain some of the higher boiling paraffins which may be pressed only with great difficulty. The heavy lubricant drawn from the bottom of the fractionating column may be subjected to refining treatment only, or may be subjected to cold settling or centrifuging followed by refining treatment. In some cases, it is advisable to combine the heavy stocks collected in the tanks 35 and 45 and subject the mixture to cold settling or centrifuging to remove the wax, and, if desired, to subsequent refining treatment such as filtering.

During all of the operations above described, I preferably maintain an absolute pressure of about 25 mm. in the vaporizer 30 by operation of the vacuum pump 67. The absolute pressure at the pump itself will usually be appreciably less than the pressure in the vaporizer, the pressure drop through the fractionating column 40 and the connecting piping being responsible for the difference. It will be apparent that the pressures at the pump and in the fractionating column are absolutely immaterial except in so far as they determine the pressure in the vaporizer. In order words, the function of the low absolute pressure in the vaporizer is to enable the vaporization of the required percentage of the charging stock at a lower temperature than would otherwise be necessary, and once this object is accomplished and the vapors have dropped below their cracking temperature, the pressure under which the subsequent operations are conducted is a mere matter of convenience. If steam is introduced into the vaporizer, it is possible to work at higher distillation pressures because steam reduces the partial pressure of the oil vapors.

In most instances also, I prefer to use as a starting material for my process a reduced crude rather than a crude containing the natural gasoline and kerosene. This is advisable in order to reduce the burden upon the apparatus and to make the operation more economical.

The foregoing operations have been suc-

cessfully carried out by me on a semi-commercial scale in an experimental plant, corresponding in general layout to the plant illustrated in Figure 1. The following are illustrative runs made in accordance with this invention on two of the three types of charging stock above discussed:

I. Asphaltic base nonwax-bearing oil

Charging stock, Orange County Gulf Coast reduced crude (70% bottoms).

Charging rate, 50 gallons per hour.

Products	% of feed	Viscosity
Total vaporized	88.1	
Light overhead cut	34.6	
Medium cut	34.2	60 sec. @ 210° F.
Heavy viscous cut	19.3	161 sec. @ 210° F.
Tar bottoms	11.7	

Temperatures

Outlet of heater	845–866° F.
Vapor from vaporizer	719–731° F.
Vapor from column	390° F.

Pressures (absolute)

Top of column	20 mm.
Vaporizer	31–32 mm.
Velocity of oil in heater	1.79 ft./sec.
Total time of oil in heater	112 seconds
Probable time in heater at temperature above 650° F.	60 seconds

II. Mixed base wax-bearing oil

Charging stock, mixture of Powell and Prairie Pipe Line Midcontinent reduced crudes (47% bottoms).

Gravity, 24.5° A. P. I.

Charging rate, 61.5 gallons per hour.

Products	% of feed	Gravity A. P. I.	Viscosity
Total vaporized	82.5		
Wax distillate	63.2	29.3°	79.5 sec. @ 100° F.
Heavy viscous cut	19.3		138 sec. @ 210° F.
Tar bottoms	17.5		

Temperatures

Outlet of heater	826° F.
Vapor from vaporizer	724–728° F.
Vapor from column	530° F.

Pressures (absolute)

Top of column	20 mm.
Vaporizer	36–38 mm.
Velocity of oil in heater	2.20 ft./sec.
Total time of oil in heater	91 seconds
Probable time in heater at temperature above 650° F.	50 seconds

It will be understood that the pressures, temperatures and other data given in these runs are not in any sense limiting values, but are simply illustrative of suitable values for the particular charging stocks. In computing the velocity of the oil in the heater and the time which the oil is in the heater, it was assumed, first, that the oil remains in the liquid phase throughout its passage through the heater, and second, that the average density of the oil is its density at 500° F.

In each of these runs the distillation was effected without any substantial cracking taking place even though the final temperature at the outlet of the heater was well above the cracking temperature of the charging stock. Moreover, this result was accomplished without the use of steam in the tubular heater. It will be noted that the lowest absolute pressure used in the vaporizer in either of these runs was 31 mm., but I have found it advisable in some instances to utilize considerably lower pressures in the vaporizer. Ordinarily, however, I maintain an absolute pressure in the vaporizer in the neighborhood of 25 mm. of mercury.

At first glance the velocity of the oil in the heater in the foregoing runs, which is in the neighborhood of two feet per second, appears to be far outside the preferred range of velocity according to my invention as hereinabove defined. This apparent discrepancy is due to the fact that the tubes in this experimental heater were of very small internal diameter, the heater consisting of 200 lineal feet of three-eighths inch standard wrought iron pipe having an internal diameter of 0.49″. With pipe of this internal diameter, a velocity of about two feet per second is sufficient to avoid any injurious skin effect and to secure a turbulent flow. The velocity of ten feet per second referred to above is applicable to tubes of the ordinary sizes used in commercial pipe stills. In this connection it may be stated here that the velocity of ten feet per second is higher than that ordinarily used in such pipe stills. Knowing the velocity required with one size of pipe when heating a given oil to a specified maximum temperature to obtain a suitable temperature gradient between the oil at the outside of the pipe and that at the center, that is to say, the velocity required to avoid injurious skin effect, it is possible to compute by well known formulæ the velocity which must be maintained to obtain the same temperature gradient when using a pipe still having pipes of a different size. In other words, the relationship between velocity and temperature gradient has been already mathematically determined, and depends upon the diameter of the pipe, the temperature to which the oil is to be heated, and the physical characteristics of the oil.

Turning now to Figures 2 and 3 of the drawings, these figures disclose a modified form of pipe still which may be advantageously employed in carrying out my process. The furnace 80 is provided with a burner 81 arranged at one end of the furnace near the floor thereof and discharging into a radiant heat or combustion chamber 82. The products of combustion are forced to pass upwardly over a bridge wall 83, down through a convection heat chamber 84 and thence upwardly through a stack 85. A depending baffle 86 is arranged in front of the bridge wall 83, forming a passageway 87 between the bridge wall and the baffle through

which the products of combustion must pass, thus keeping them close to the bridge wall 83. The oil to be heated is supplied to the heating tubes through a pipe 88 and passes through a bank of heating coils 89 arranged in the chamber 84 and thence through a series of coils 90 located along the roof of the radiant heat chamber 82. The tubes comprising the preheating section 89 are preferably larger in diameter than those in the radiant heat section 90, as shown, and the furnace is preferably so designed that the oil does not reach a cracking temperature until it enters the coils 90.

It will be understood that in carrying out the process of my invention, the pipe still 80 is simply substituted for the pipe still 10 in the plant shown in Figure 1. The pipe still 80 has the advantage of being considerably simpler in construction than the pipe still 10 and may be used whenever it is not necessary to divide the oil to be heated into a plurality of streams as shown in Figure 1.

Figure 4 shows still another form of pipe still in which the oil is passed through the convection heat section in a single stream and is then split into a plurality of streams before passing through the radiant heat section in which it reaches a cracking temperature. In this figure, 95 represents the pipe still as a whole, and the constructional details of the furnace may be exactly the same as those shown in Figures 2 and 3 except for the arrangement of the heating tubes. The same reference characters are therefore used for similar parts. In the furnace illustrated in Figure 4, the oil to be heated is introduced into the heating coils 89 located in the convection heat chamber 84 and passes thence into a manifold 96 from which the oil is delivered to the radiant heat or high temperature section of the furnace. A plurality of high temperature heating coils 97, in this instance four, are located along the roof of the combustion chamber 82, and have their inlet ends connected to the manifold 96 and their outlet ends to a manifold 98. A valve 99 is located at the inlet end of each of the coils 97 to regulate the quantity of oil flowing therethrough. If desired, a separate pump may be placed at the inlet of each of the coils 97 to pick up the preheated oil from the manifold 96 and force it through the high temperature coils.

In the pipe still 105 shown in Figure 5, a burner 106 is located in a small combustion chamber 107 anterior to the main radiant heat chamber 82. The heating tubes 89 located in the convection heat chamber 84 discharge into a radiant heat coil 108 located along the roof of the chamber 82. The coil 108, is formed of tubes of smaller diameter than those in the coil 89, and is connected to a helical coil 109 located directly in front of the burner 106 and through which the products of combustion from the burner pass. The coil 109 is made of tubing still smaller in diameter than that of the coil 108. By this construction the oil is given a final very short exposure to the highest temperature of the products of combustion before being discharged to the vaporizer.

The use of the various pipe stills disclosed in Figures 2 to 5 inclusive when substituted for the pipe still shown in Figure 1 will be obvious from the description of Figure 1, and it will be understood that the same considerations of design apply to each of the forms of heaters.

It is of course well known that some cracking occurs even at relatively low temperatures if sufficient time is given for the reaction, and that the rate of cracking increases very rapidly as the temperature is raised. When, therefore, I refer to a temperature below the cracking temperature, I intend to define a temperature such that even though a normal time be allowed for the reaction, no substantial amount of cracking will occur, and when I refer to a temperature above the cracking temperature, I mean a temperature such that under such time conditions a substantial amount of cracking will occur.

Where the expression "temperature gradient" is employed in the claims, it is intended to mean the difference in temperature between the oil near the surface of the tubes of the tubular heater and that at the center of the tubes.

It will be understood that my invention is not limited to the specific embodiments described and that various deviations may be made therefrom. It will be apparent also that some of the features of my invention may be used entirely independently of the employment of other features herein disclosed. I desire to be limited, therefore, only by the scope of the appended claims.

1. The method of preparing lubricants from non-overhead wax-bearing lubricating oil which comprises passing the material under treatment under an initial pressure through a tubular heater, heating said material during its passage through said heater to a temperature above its cracking temperature, the period during which the material is heated above its cracking temperature and the velocity of the material during said period being such that no substantial amount of cracking of the material takes place, subjecting said material to flash vaporization under a low absolute pressure to vaporize a substantial portion thereof and to separate certain wax crystallization inhibitors therefrom, subjecting the resulting vapors to counter-current fractionation, and cutting said lubricants into a relatively light lubricating fraction containing wax in pressable form and a relatively heavy lubricating

fraction containing the balance of the wax crystallization inhibitors.

2. The method of preparing lubricants from wax-bearing lubricating oil which comprises passing the material under treatment under an initial pressure through a tubular heater, heating said material during its passage through said heater to a temperature of over 750° F., the period during which the material is heated above its cracking temperature being less than 35 seconds and the velocity of the material during said period being such as to produce a low temperature gradient, subjecting said material to flash vaporization to vaporize a substantial portion thereof and to separate certain wax crystallization inhibitors therefrom, subjecting the resulting vapors to counter-current fractionation, and cutting said lubricants into a relatively light lubricating fraction containing wax in pressable form and a relatively heavy lubricating fraction containing the balance of the wax crystallization inhibitors.

3. The method of preparing lubricants from wax-bearing lubricating oil which comprises passing the material under treatment under an initial pressure in the absence of steam through a tubular heater, heating said material during its passage through said heater to a temperature of over 750° F., the period during which the material is heated above its cracking temperature being less than 35 seconds and the velocity of the material during said period being such as to produce a low temperature gradient, subjecting said material to flash vaporization under a low absolute pressure to vaporize a substantial portion thereof and to separate certain wax crystallization inhibitors therefrom, subjecting the resulting vapors to counter-current fractionation, and cutting said lubricants into a relatively light lubricating fraction containing wax in pressable form and a relatively heavy lubricating fraction containing the balance of the wax crystallization inhibitors.

4. The method of preparing lubricants from non-overhead wax-bearing lubricating oil which comprises heating the oil in a confined flowing stream to a point well above its cracking temperature for a period of time insufficient to cause any substantial amount of cracking of the oil, subjecting said oil to flash vaporization under a low absolute pressure to vaporize a substantial portion thereof and to separate certain wax crystallization inhibitors therefrom, subjecting the resulting vapors to counter-current fractionation, and cutting said lubricants into a relatively light lubricating fraction containing wax in pressable form and a relatively heavy lubricating fraction containing the balance of the wax crystallization inhibitors.

5. The method of preparing lubricants from non-overhead wax-bearing lubricating oil which comprises passing the material under treatment through a tubular heater, heating said material during its passage through said heater to a temperature above its cracking temperature, the period during which said material is heated above its cracking temperature being such that no substantial amount of cracking of the material takes place, subjecting said material to flash vaporization under a low absolute pressure to vaporize a substantial portion thereof, subjecting the resulting vapors to counter-current fractionation to separate wax crystallization inhibitors therefrom, and separating from the remaining vapors a directly pressable wax distillate.

6. The method of preparing lubricants from non-overhead wax-bearing lubicating oil which comprises passing the material under treatment through a tubular heater, heating said material during its passage through said heater to a temperature above its cracking temperature, the period during which said material is heated above its cracking temperature being such that no substantial amount of cracking of the material takes place, subjecting said material to flash vaporization under a low absolute pressure to vaporize a substantial portion thereof, subjecting the resulting vapors to counter-current fractionation to separate wax crystallization inhibitors therefrom, withdrawing the remaining vapors, and condensing the same to form a directly pressable wax distillate containing the major portion of the crystallizable wax initially present in the material.

In testimony whereof, I have signed my name to this specification this 27th day of February, 1928.

EUGENE H. LESLIE.