Aug. 3, 1937. J. E. SCHULZE 2,088,616

PROCESS FOR THE MANUFACTURE OF LUBRICATING OILS

Filed May 5, 1932 3 Sheets-Sheet 1

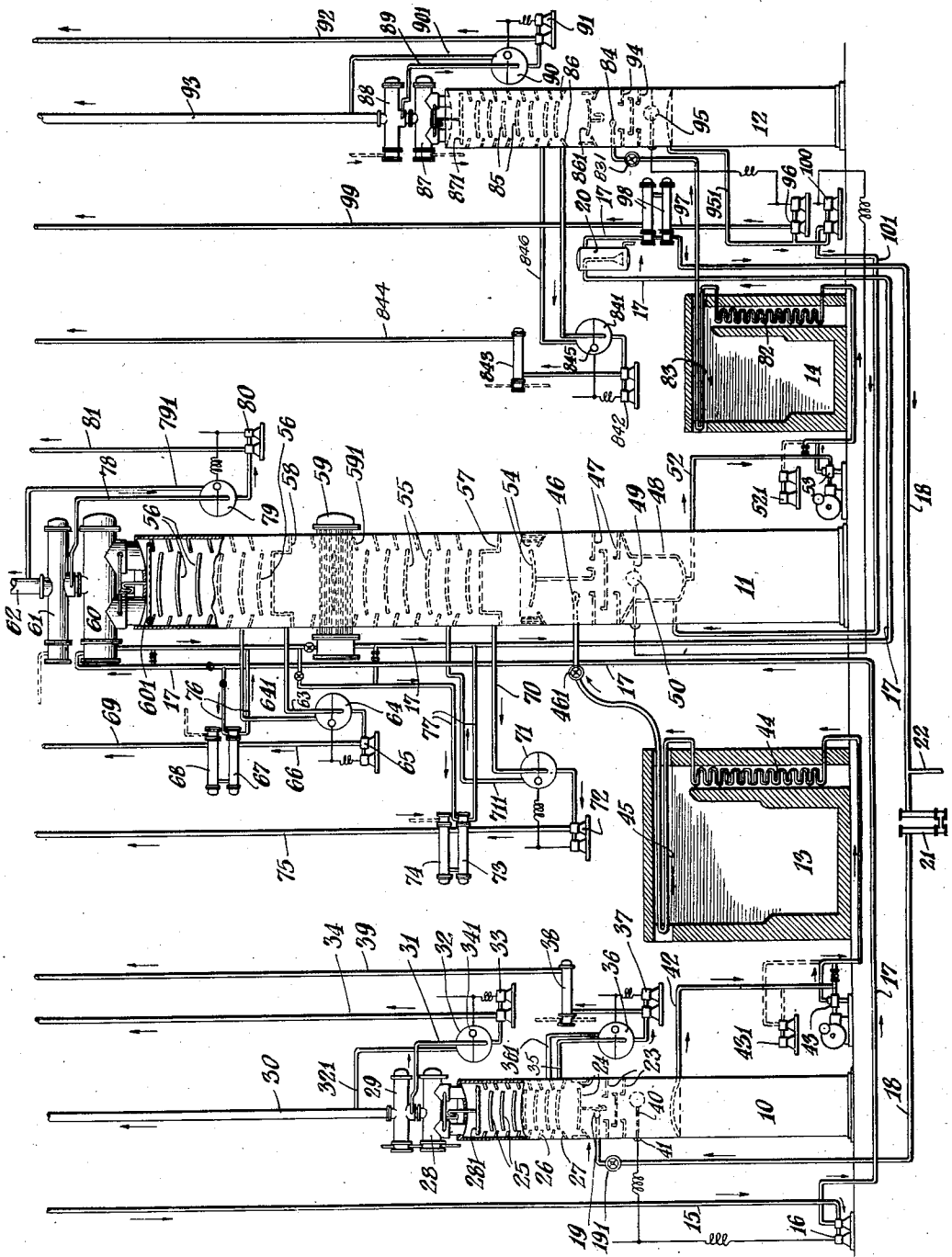


Fig. 1

INVENTOR
John E. Schulze
BY Stewart & McKay
his ATTORNEYS.

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PROCESS FOR THE MANUFACTURE OF LUBRICATING OILS

Filed May 5, 1932 3 Sheets-Sheet 2

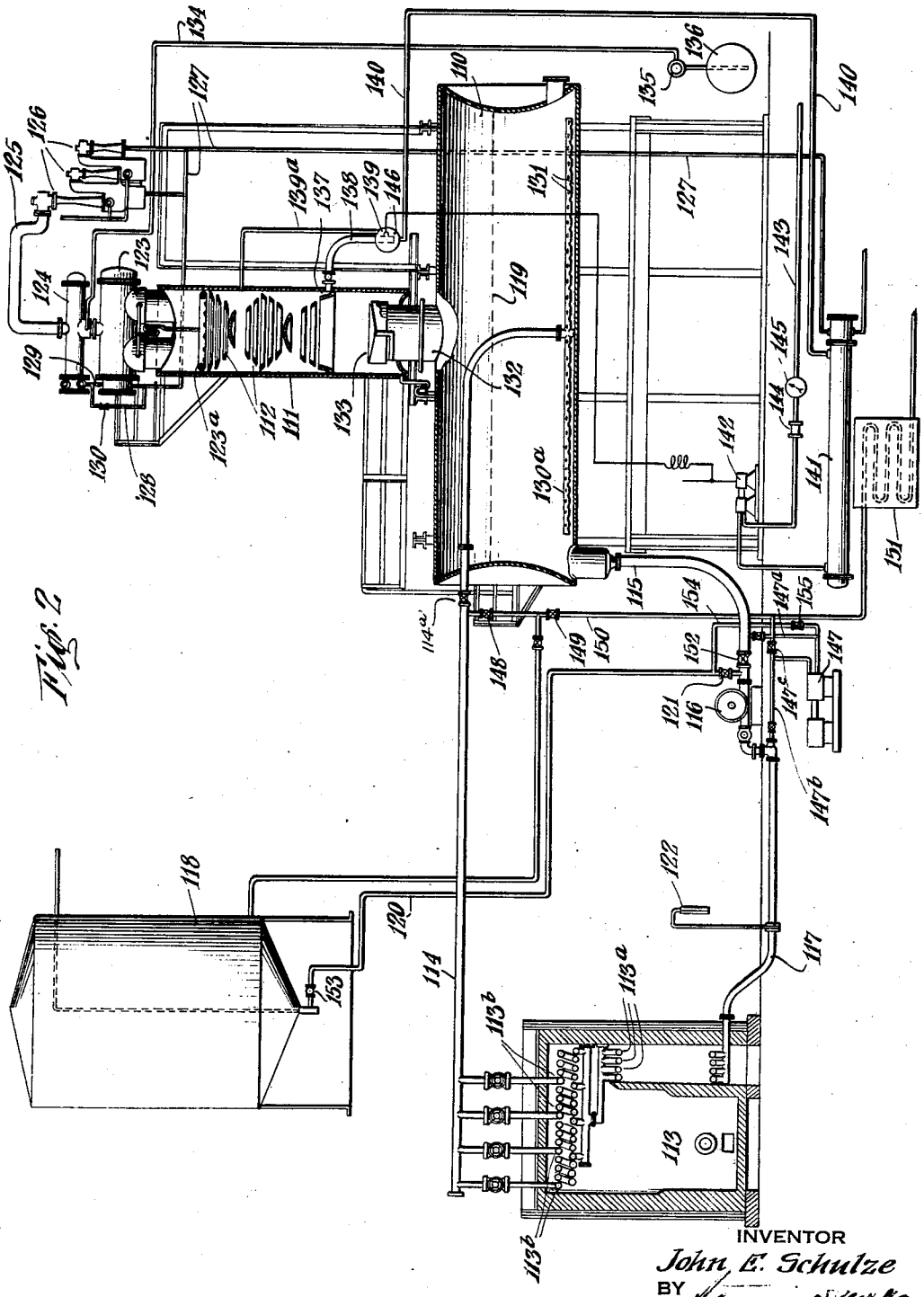


Fig. 2

INVENTOR
John E. Schulze
BY Lee and Mackay
his ATTORNEYS.

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PROCESS FOR THE MANUFACTURE OF LUBRICATING OILS

Filed May 5, 1932 3 Sheets-Sheet 3

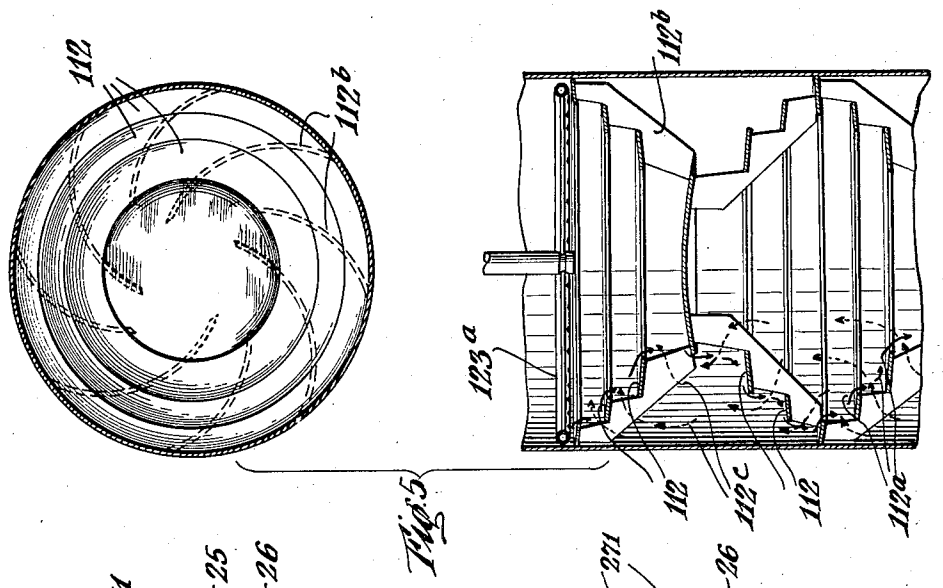


Fig. 5

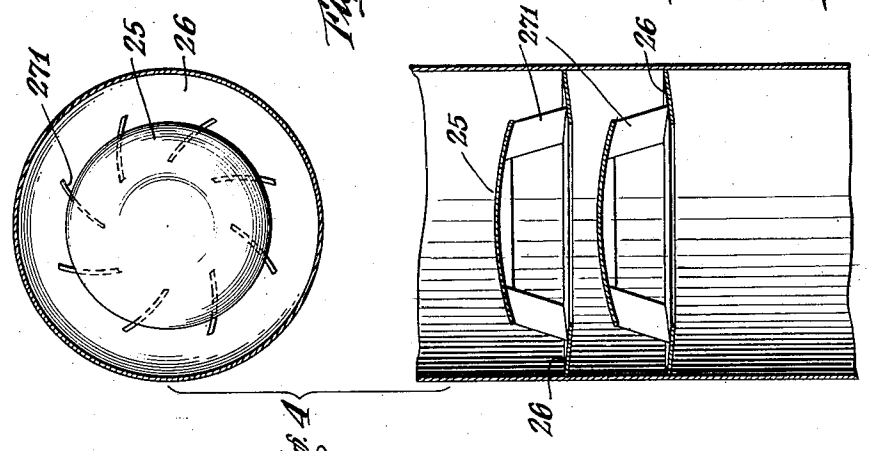


Fig. 4

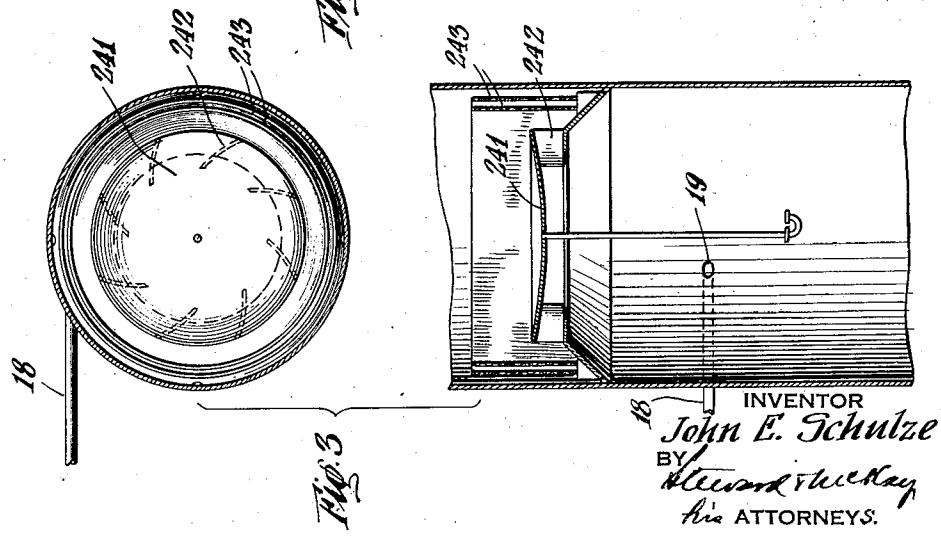


Fig. 3

INVENTOR
John E. Schulze
BY
Stevens & McKay
his ATTORNEYS.

Patented Aug. 3, 1937 2,088,616

UNITED STATES PATENT OFFICE

2,088,616

PROCESS FOR THE MANUFACTURE OF LUBRICATING OILS

John E. Schulze, Chicago, Ill., assignor, by mesne assignments, to Red River Refining Company, Chicago, Ill., a corporation of Delaware

Application May 5, 1932, Serial No. 609,508

23 Claims. (Cl. 196—77)

This invention relates to processes for the manufacture of lubricating oils. In respect to process, the invention relates more particularly to production in a novel manner of high grade lubricating oils directly by straight overhead distillation under high vacuum, the distillates thus obtained, when the invention is practiced in its best form, requiring no acid treatment or filtration to render them finished high grade lubricating oils of good color and stability. The invention further comprises novel apparatus useful in practicing the process of the invention effectively.

Broadly speaking, the manufacture of lubricating oils by so-called vacuum distillation of mineral oil starting material, such as crude petroleum or residues derived therefrom, has been known for many years. Prior to introduction of the process of the Schulze United States Patent 1,448,709, the degree of vacuum employed in the industry, under the most favorable conditions, had practically never exceeded 28 inches mercury gauge; that is, the absolute pressure under which distillation occurred was seldom less than 50 millimeters mercury in commercial work, and such lubricating oils as were distilled overhead had to be acid-treated and/or filtered to render them fit for use. Even these were usually of inferior quality, judged by present day standards. The heavier and more viscous lubricating oils could be prepared only by blending suitable "bottoms" or cylinder stock residues with lighter lubricating oils to meet required specifications, which were necessarily much less exacting then than now.

The commercial introduction of the aforesaid process of Patent No. 1,448,709, however, immediately gave a great impetus to vacuum distillation of lubricating oils. Refiners quite generally soon began to appreciate the commercial feasibility of operating at the much lower absolute pressures characterizing the method of that patent, and to realize that distillation at materially lower absolute pressure than had previously been used offered an entirely new and highly advantageous way of manufacturing high grade lubricating oils far superior in character to those previously available in the industry. There has accordingly been a marked tendency during recent years to depart from the so-called vacuum distillation methods of earlier years and to adopt the more modern and truly high vacuum method aforesaid. This also involved, in contradistinction to the earlier so-called vacuum distillation methods, rigid exclusion of oxidizing conditions within the distillation system.

The commercial development of the process of the aforesaid United States Patent 1,448,709 has been along the lines both of batch or discontinuous distillation, and also of continuous distillation. Batch or discontinuous distillation may be generally described as that type of distillation in which a given body of oil is gradually heated, without being replenished, to successively higher temperatures to distil off progressively heavier lubricating constituents in the order of their boiling points, until the original body of oil has been reduced to "bottoms" or residue. In continuous distillation, on the other hand, the charging stock to be distilled is continuously fed to the vaporizing system and is usually heated to progressively higher temperatures as it passes through successive units of the vaporizing system, all the lubricating constituents vaporizable under the conditions of operation, from the lightest to the heaviest, being simultaneously distilled off in one or more cuts at a series of predetermined temperatures corresponding, respectively, to the several vaporizing units of the system. Operation by either batch or continuous distillation is contemplated by the aforesaid United States Patent 1,448,709, but batch distillation is more specifically referred to therein by way of illustration. Furthermore, until the present invention, batch distillation has in point of fact remained the only way by which high grade lubricating oils of satisfactory color and stability could be produced directly as overhead distillates, even by distillation at absolute pressures below 25 millimeters of mercury, without the necessity of acid-treating and filtering.

The apparent advantages of continuous distillation from certain operating standpoints have led to much activity in an attempt to produce in this manner high grade lubricating oils of good color and stability, operating at absolute distilling pressures of the order of magnitude characterizing the process of the patent aforesaid, or approximating the same. While such attempts have indeed resulted in some cases in the production of lubricating oils notably superior to those obtainable by the old so-called vacuum methods that were in vogue prior to the last decade, it is a well known fact that they have not been successful to the desired extent. That is, they have not produced by direct overhead distillation, without subsequent acid treatment and/or filtration, finished lubricating oils of the good color and stability now recognized as necessary for modern lubricating oils of really high grade. This is especially true where the crude starting material employed as charging stock is

characterized by a substantial sulfur content, but it is also true for any charging stock. As will presently appear, the limitations inherently characterizing any practical form of continuous distillation system are such as to militate strongly against the feasibility of producing, by any method of continuous high vacuum distillation alone, thoroughly satisfactory finished lubricating oil distillates without the necessity for subsequent acid treatment and filtration. Discovery of this fact and appreciation of its practical significance have given rise in part to the present invention.

By the batch or discontinuous system, it is possible to produce, in the manner described in the aforesaid United States Patent 1,448,709, from some types of crude charging stock not characterized by high content of sulfur or other contaminating constituent causing too great complications, straight overhead distillates requiring no acid treatment or filtration to render them finished lubricating oils of satisfactory stability and color; and large amounts of lubricating oil have been actually produced commercially in this manner. That process does not, however, produce lubricating oils of equally high grade when applied to the treatment of mineral oil charging stock containing relatively high percentages of sulphur compounds and other contaminating constituents of malodorous and unstabilizing character. In order to handle such very low grade crude charging materials to the best advantage, this applicant has heretofore further developed and improved the process disclosed in said U. S. Patent 1,448,709 to provide a method of and means for distilling such low grade crude material to produce directly therefrom deodorized and stable overhead lubricating distillates at the very low absolute distilling pressures in question; see Canadian Patent No. 295,592, December 10, 1929. This improved method is highly effective for the intended purpose. In commercial practice, however, it has been found to give thoroughly satisfactory results only when operated on the batch or discontinuous principle, and it has not proved wholly satisfactory as applied to certain crudes of very low grade.

The manufacture of lubricating oils by direct overhead batch or discontinuous distillation from crude charging materials, such as topped crude petroleum, containing tarry or asphaltic matter or other high-molecular non-lubricants heavier and higher boiling than the lubricants which it is desired to obtain, is nevertheless open in all cases to certain objections, regardless of the character of the crude employed as starting material. The prolonged heating of the charge to increasingly high temperatures during vaporization of the lubricating fractions therefrom, which requires 12 to 14 hours or more in typical instances, involves more or less cracking or breaking down of these high-molecular compounds, which, as this applicant has now found, may go on to some extent substantially throughout the period during which the lubricating distillate is coming over. The products resulting from the breaking down are in part complex hydrocarbons or hydrocarbon derivatives which, even in very small percentages, have an unstabilizing effect upon lubricating oil. Some of them are unsaturates and, if the charging crude be high in sulphur, they often contain sulphur in complex combinations, and in both cases they are apt to be evil-smelling. Moreover, notwithstanding they are in general lighter and lower boiling than the lubricating constituents vaporized concurrently with them, it is often difficult, and with certain types of charging stock impossible, even when using the process disclosed in Canadian Patent 295,592 aforesaid, to free the lubricating distillates from them so completely as to render said distillates perfectly sweet and stable. The fact that, especially with certain types of charging crudes and residues, the formation of those malodorous and unstabilizing compounds goes on substantially throughout the entire period of lubricant vaporization renders the stated difficulty a continuing one and complicates the problem material. Formerly it was believed that they had to be contended with substantially only during the first part of the lubricant distillation period, and this indeed holds true for many charging stocks, but this applicant has discovered that for other kinds of charging stock it does not hold true.

A somewhat analogous difficulty is encountered in attempting to obtain sweet, stable lubricating oil distillates by continuous distillation under high vacuum. While it is possible to so conduct continuous distillation as to reduce materially the time during which the oil charge must be subjected to heat at the necessary vaporizing temperatures, it is not possible, in practice, to avoid superheating of both liquid and vapors to some extent, with consequent formation of lighter unstabilizing contaminants and further complication of the problem. Thus, in any given vaporizer of a series which is being operated on the continuous distillation principle, a certain distilling temperature is maintained, which temperature is a certain number of degrees higher than the temperature maintained in the next preceding vaporizer of the series, and a certain number of degrees lower than that maintained in the next succeeding vaporizer. Consequently, oil entering a given vaporizer is necessarily at a substantially lower temperature than the main body or pool of oil therein contained, so that the entering oil is necessarily subjected suddenly to contact with considerably hotter oil. As a result, portions of the entering oil which would actually volatilize at a somewhat lower temperature than that maintained in the given vaporizer under consideration, are nevertheless subjected in vaporizing to a temperature higher than is required to volatilize them, and they are therefore superheated. The extent of this superheating depends, of course, upon the amount which the temperature is stepped up from one vaporizer to the next. It would necessitate employing an infinite number of vaporizers, theoretically, to avoid this superheating effect altogether. It is obviously impractical to employ more than a rather limited number of vaporizers in such a series; and accordingly, in continuously operating systems of this kind, there is unavoidably a substantial step-up in temperature from one vaporizer to the next in series. This means that throughout the entire continuous distillation series there is constantly going on the described superheating with its attendant breaking down or cracking of the lubricating oil hydrocarbons, even though the absolute pressure be maintained well below 25 millimeters of mercury. At higher absolute pressures the injurious effect is still more pronounced. The products of the described cracking or breaking down include both light and heavy products. The light products of this decomposition commonly include unstabilizing agents of the character above discussed, while some of the heavy

products of the cracking are dark colored and even tarry in nature and, unless completely separated from the finished lubricating oil, materially lower its color grade.

Experience has shown that it is impossible to eliminate either the light or the heavy contaminants aforesaid with sufficient completeness even by the use of fractionating towers or columns in conjunction with the several vaporizing units of a continuous distillation system. Bubble tray columns and the like have been extensively tried in this connection, but in order for these or other fractionators to effect clean separation of desired distillates, the vapors must be bubbled through so many layers of liquid reflux, or must otherwise be brought into such intimate contact with the reflux, that relatively great resistance is offered to the vapor flow. Consequently the pressure drop through the fractionator is so great that the absolute pressure practical to maintain at the initial vaporization point (e. g. below the bubble plates) is undesirably high, necessitating employment of relatively high vaporizing temperatures with attendant liability to decomposition. If it be sought to get around this difficulty by increasing the capacity of the exhausting equipment, then the vapors "blow" through the layers of liquid reflux and escape adequate fractionation; and if resistance be cut down by reducing the number of trays in the fractionator, similarly ineffective fractionation results.

Thus, notwithstanding the greater rapidity of operation and consequent increased plant capacity characterizing continuous high vacuum distillation, these advantages have heretofore been outweighed by the disadvantage that the resultant lubricating distillates have had to undergo acid treatment and filtration before they could be marketed as finished lubricating oils.

A principal object of the present invention is to avoid the stated difficulties and objections and to provide a process which will enable production by direct overhead distillation at low absolute pressure, and more expeditiously and economically than has heretofore been possible, of stable lubricating oil distillates requiring no acid treatment or filtration to render them marketable as finished lubricating oils of high grade.

Another object of the invention is to provide improved procedures in the high vacuum distillation of lubricating oils, both continuous and discontinuous, whereby greater efficiency and economy of operation may be attained.

Still another object of the invention is to provide apparatus suitable for practicing the novel processes of the invention and novel in important respects.

The foregoing objects, as well as others which will presently become apparent, are attained by the present invention, of which certain typical practical embodiments will be described hereinafter for the purpose of illustrating the underlying principles involved, and which will then be more particularly pointed out in the appended claims.

Described generally, the complete process of the invention comprises two principal stages or phases:

In the first stage, there is effected a relatively quick separation of lubricating oil components from the heavier components of a given crude mineral oil material or charging stock by a continuous procedure which includes rapidly heating the crude material to temperatures sufficiently high to vaporize all the desired lubricating oil components under absolute pressure on the order of 50 millimeters of mercury or lower, vaporizing such components at such low absolute pressure, and condensing them under such low pressure as one or more lubricating oil distillates substantially free of tarry and other heavier constituents of the crude starting material employed, as well as from nearly all of the lighter constituents thereof. The lubricating oil distillates thus obtained by quick separation, while superior in some respects to those heretofore obtainable by comparable procedure, are nevertheless characterized by a small content of unstabilizing constituents, and they are not the finished, stable lubricating oils which it is one of the principal objects of the invention to produce. Said distillates are employed, either singly or together, as high grade charging stock in the second stage or phase of the complete process.

In the second stage or phase of the complete process of the invention, the quickly separated and unfinished lubricating oil distillates are subjected, either singly or in mixtures of two or more, to redistillation by batch or discontinuous distillation in which vaporization and subsequent condensation of the lubricating oil constituents is effected under absolute pressures not exceeding, and most desirably materially below, 25 millimeters of mercury, and at temperatures increasing only very gradually and uniformly, the lubricating oil constituents being thus progressively vaporized in the order of their boiling points and obtained as lubricating oil distillates requiring no acid treatment or filtration to render them finished marketable lubricating oils of good color and stability.

By proceeding in the manner above described, a number of highly important advantages are realized. Separation of the heavier and more heat-sensitive constituents of the crude charging stock from the lubricating oil constituents being accomplished quickly, the objectionable long continued heating of the heavier constituents necessarily involved in batch distillation of the crude charging stock, referred to hereinabove, is avoided. It is true that such quick separation requires heating the crude charging stock to temperatures as high as, say, 725° F., or higher than can be employed under any conditions without some breaking down or cracking of the heavier oil components, including some of the heavy lubricating oil components; but the time factor is so greatly reduced that the loss of lubricants from cracking is relatively slight and indeed negligible in comparison with the great advantages accruing from the combination of this preliminary quick separating step with the succeeding batch redistillation in the absence of the disturbing tarry and other heavy components previously gotten rid of at the outset. Thus, although continuous distillation is not capable of producing directly overhead lubricating oil distillates that require no acid treatment or filtration to render them stable, it becomes possible in accordance with the present invention to utilize the greater rapidity and economy of continuous distillation in eliminating anywhere from 40 to 60 per cent of the initial crude charging stock before resorting to distillation by the batch or discontinuous method which experience shows to be essential for production, by direct overhead distillation and without acid-treatment or filtration, of finished lubricating oils of good color and stability.

The relatively quick preliminary separation

constituting the initial stage of the present process may be carried out continuously in various ways. Thus, as will be hereinafter more particularly illustrated and referred to by way of concrete example, the crude charging material may be heated to the necessary temperature and passed through a series of flash vaporizers to distill off whatever gas oil or other non-viscous fractions the material may contain, and also to distill off the desired lubricating oil fractions from the lightest and least viscous to the heaviest and most viscous; leaving unvaporized, as flux oil (e. g.), a tarry residue of heavier components of the crude charging stock. The vapors produced in each vaporizer may be passed upwardly through a fractionating tower or column adapted to minimize "vacuum drop" through the tower (increase in absolute pressure from the top toward the bottom) as much as possible. Whatever liquid residual material remains unvaporized in any given flash vaporizer is passed into the next succeeding vaporizer for re-flashing, ordinarily after being heated to a higher temperature, to vaporize such further lubricating components as can be thus obtained therefrom.

In another type of continuous distillation procedure which may be employed within the scope of the present invention, but which at present is deemed less desirable from a practical standpoint, the mineral oil material to be distilled may flow, by gravity or otherwise, through a series of stills of the ordinary shell type, for example, connected in series, each still being maintained at a predetermined vaporizing temperature which increases with each succeeding still in the series. A low absolute pressure may be maintained on the surface of the oil in each of these stills, and the vapors evolved in each, which are progressively heavier and higher boiling fractions from the first still to the last of the series, may be drawn off through suitable fractionating towers, as before, yielding various lubricating oil cuts as straight overhead distillates which are to be further treated in the second stage of the new process.

By employing in the second or re-distillation stage of the process what is essentially batch or discontinuous distillation, the unfinished lubricating distillates obtained from the preceding continuous distillation stage, which are inevitably somewhat unstable and sometimes relatively dark in color, can be substantially wholly freed of these contaminating components and rendered substantially odorless or free of objectionable odor (i. e. "sweet"), of exceptionally good color, and stable. This is due to the fundamentally different nature of batch or discontinuous distillation as compared with continuous distillation. In batch or discontinuous distillation, the body of oil to be distilled is not replenished during the distillation, but the body of oil as a whole is gradually raised to higher and higher temperatures, smoothly and substantially uniformly, instead of step-wise as in continuous distillation. In other words, the distilling temperature increases by increments each of which is extremely small, that is, on the order of only a fraction of a degree Fahrenheit, for example, over a substantial time unit. Because of this fact, there is at no time any superheating of the liquid oil or the evolved vapors through exposure to temperatures higher than the boiling point of the fraction being evolved at the moment, and consequently cracking or decomposition from that source is eliminated. Moreover, because of the uniform composition of the vapors going through the fractionating column at any given time during the distillation, such small quantity of unstabilizing and color-darkening contaminants as may be carried by that particular very narrow cut fraction can be separated with comparatively extreme sharpness from the lubricating oil distillate by the use of fractionating means which interpose minimum resistance to vapor flow and which, under other conditions, would prove relatively inefficient. The lubricating oil distillate is thus so completely freed of such contaminants that it constitutes a finished lubricating oil just as it is condensed and led away from the distilling system, without having to be acid-treated or filtered to render it a high grade merchantable product.

While the process of the invention, in its broader aspects, is not limited to the employment in either stage of any particular type of fractionating tower or column, or indeed to the employment of any fractionating means properly so called, it is desirable to employ in both stages, and especially in the second stage, fractionating means functioning with the least resistance to passage of the vapors which, under the conditions characterizing the high vacuum distillation here involved, travel at very high velocity. In particular, it is desirable to give the oil vapors a whirling movement in contact with counterflowing reflux in a manner to be hereinafter described. Such a method of fractionation is believed to be broadly novel in the manufacture of overhead lubricating oil distillates.

In order to explain further the underlying principles of the invention, a detailed description will now be given of typical embodiments of the new process and apparatus which have been found particularly advantageous in practice. It is to be understood, however, that the details hereinafter given are merely for the purpose of illustrating one practical way in which the invention may be satisfactorily practiced, and that the invention is not restricted to the particular details disclosed but may be embodied in various other specific forms so long as the broad underlying principles of operation are adhered to, all as set out in the appended claims.

The accompanying drawings illustrate more or less diagrammatically, and to some extent in the form of a flow sheet, a typical distillation plant wherein the process of the invention can be effectively carried out. In these drawings,

Fig. 1 illustrates the primary distillation unit of the system, showing the principal apparatus or equipment parts in side elevation, parts being broken away and in section to disclose certain structural details;

Fig. 2 is a similar view of the secondary or re-run distillation unit; and

Figs. 3 to 5 show certain parts in greater detail.

The primary distillation unit (Fig. 1), in the specific form here illustrated, is adapted and arranged for continuous distillation of the initial mineral oil charging stock by a succession of flash vaporizations whereby all the lubricating oil components of the charging stock, from very light to extra heavy, together with any lighter fractions, such as gas oil, which the charging stock may contain, are continuously vaporized at low absolute pressures, the resultant vapors being fractionated to separate the lubricating values in several cuts or fractions, as may be desired, and also to obtain the gas oil mainly as another cut or

cuts separate from the lube cuts, while rapidly withdrawing from the various condensed lube distillates uncondensed vapors and gases including a substantial proportion of whatever malodorous and other contaminating matters may be present. Some of these contaminants may be condensed with one or more gas oil cuts.

As here illustrated, the primary distillation unit comprises towers 10, 11, and 12, together with pipe still 13 cooperating with tower 11, and pipe still 14 cooperating with tower 12. In the present example, the crude charging stock may be assumed to be a topped crude petroleum containing little or no material more volatile than gas oil. Also, in this specific example, the distillation and quick separation of lubricating constituents therefrom, properly speaking, is preceded by an initial flashing-off in tower 10 of some of the gas oil and a small amount of such lighter constituents as may be present, the heat for which is obtained through recovery of heat from a later stage of the flash-vaporizing procedure. It is to be understood therefore that this preliminary flashing step, while desirable from the standpoint of economy and a valuable feature of the process in its best practical embodiment, is not essential in the broader aspects of the invention. The crude charging stock is drawn through supply line 15 by pump 16 and, after being sent through line 17 to certain heat exchange means (67, 73, 98) and being heated to around 375°–400° F., as will hereinafter appear, it is returned through line 18 and is discharged by the pump into tower 10 as indicated at 19. Where the crude charging stock contains salt water, as is often the case, this should be removed by passing the stock through a dehydrator 20 interposed at a point in line 17 where the stock has attained a temperature of, say, 300° F. In return line 18 is interposed a mixing coil 21 where a suitable quantity of caustic soda solution is thoroughly mixed with the flowing stream of preheated charging stock, the caustic soda solution being introduced into the line through pipe 22 which enters the line in advance of the mixing coil 21. The strength and quantity of the caustic solution used depends upon the nature of the crude charging stock to be processed. Ordinarily a 40° Baumé caustic soda solution is employed and the quantity required practically never exceeds 0.3 per cent by volume of the crude charging stock even where its content of sulphur compounds and other contaminants is relatively very high. The slight amount of water introduced into the oil in this way is unobjectionable. It is desirable to have the soda, or other suitable alkaline neutralizing agent, present in the liquid stock throughout the succeeding operations. The preheated charging stock is delivered tangentially into tower 10 at a point slightly above the stripping plates 23 contained in the lower part of the tower. Just above the point of charging stock delivery or inlet is an entrainment separator 24 so designed as to impart considerable velocity and centrifugal action to the vapor ascending the tower, with a minimum drop in vacuum, to throw out any liquid particles entrained with the vapor, thus permitting a clean separation of such particles from the vapor prior to entering the fractionating zone located above the entrainment separator. The construction of the entrainment separator is shown in greater detail in Fig. 3. The vapors rising to plate 241 are deflected laterally by curved vanes 242 which direct the vapors against screen 243 while the vapors are also given a whirling movement. Entrained liquid particles arrested by the screen drain downwardly along the walls of the column.

In the fractionating zone are provided a plurality of suitable fractionating plates 25 cooperating with downwardly sloping side rings 26. The lowest of these plates has a cooperating side ring 27 which slopes upwardly, the combination constituting a collecting plate. Curved guide vanes 271 (Fig. 4) serve to impart whirling movement to the ascending vapors. The higher boiling portions of the ascending vapors, which consist mainly of gas oil, are condensed in reflux condenser 28, the liquid reflux condensate being discharged through distributor head 281 at the top of the column and flowing down over the fractionating plates, of which there are eight in the present example, in intimate contact with the ascending vapor. While the plates are so designed as to give intimate contact of counter-flowing reflux and vapor they are of such character as to cause minimum vacuum drop in the column. This type of plate or tray has been found to be particularly suitable for use in connection with the present process and to be far superior to bubble plates, the use of which has heretofore been attempted in lubricating oil distillation but has been far from satisfactory. No novelty is claimed for the illustrated plate construction per se, but the use of such plates, or of plates having generally similar characteristics, in high vacuum distillation of lubricating oils is believed to be novel with the present applicant. Other specific designs of plate can of course be employed within the broad scope of the invention.

Gases, lighter vapors and water vapor escaping condensation in the reflux condenser 28 pass to a final condenser 29 where a still lighter gas oil is condensed together with some malodorous and unstabilizing constituents. The remaining uncondensed vapors and gases are swiftly sucked away through large-diameter offtake 30 by suitable exhausting means, such as a 2-stage vacuum pump (not shown). The exhausting means is so operated as to maintain the entire interior of tower 10 under low absolute pressure. This absolute pressure should not exceed 50 millimeters of mercury as a maximum at any point in the column, and it is desirable in practice that this maximum should be substantially less than 50 millimeters, say 30 millimeters. Due to the resistance offered to the upward flow of vapors by the entrainment separator and fractionating plates, the absolute pressure at the point of initial vaporization, that is, at the point where the charging stock is delivered into the column, will be somewhat higher than the pressure at the top of the column. Where plates of the general character here illustrated by way of example and recommended for use are employed, however, this vacuum drop or pressure increase is reduced to a minimum and considerably below what is possible with a bubble-tray system of comparable fractionating effect. Thus, assuming that the pressure at the point of vaporization is to be maintained at substantially 30 millimeters, this can be accomplished by operating the vacuum pump to maintain an absolute pressure of 25 millimeters at the top of tower 10 just before the vapors enter the reflux condenser 28.

The light gas oil and malodorous fraction or cut condensed in the final condenser 29 is led away through line 31 to constant level seal tank 32, from which it is withdrawn by pump 33 and delivered through line 34 to storage. The seal

tank has a pressure balancing connection 321 to vacuum line 30. Pump 33 is located a sufficient distance below the seal tank to give the necessary suction head, and the rate of operation of the pump is automatically controlled in a well known manner by appropriate connections to float 341 in the seal tank to maintain the liquid level in the seal tank constant.

The higher boiling gas oil fraction caught by collecting plate 27 is taken from the tower through line 35 to seal tank 36, from which it is withdrawn by pump 37 and pumped through cooler 38 and thence through line 39 to storage, the operation of the pump 37 being automatically controlled, by means similar to that already described for pump 33, to maintain a constant level of the liquid distillate collecting in seal tank 36. The seal tank is provided with pressure balancing connection 361 to a point in the column at least one plate higher than where line 35 leaves it or, of course, this balancing connection may go to the vacuum line (e. g. 30).

That portion of the charging stock which does not vaporize upon delivery into tower 10 passes down over stripping plates 23 and accumulates in the base of the tower to the level indicated at 40, this level being maintained constant by float control mechanism 41 which controls the rate of operation of charging stock feed pump 16 through regulation of its steam supply, the arrangement being the same as described for pump 33 and seal tank 32. This unvaporized residue of liquid oil containing substantially all the original lubricating oil constituents of the raw charging stock, together with most of the gas oil, is next heated to a much higher temperature in order that a large part of the lubricating constituents may be flash-vaporized therefrom in tower 11 at still lower absolute pressures than those prevailing in tower 10. To this end, the residual oil is drawn from the base of the tower 10 through line 42 by pump 43 and forced through the convection and radiant tube banks 44, 45, of pipe still 13, where it is heated to the desired higher temperature and is discharged into tower 11 at 46, at which point a vaporizing pressure of, say, 12 millimeters absolute, is maintained. The pipe still heater is so designed that the oil passes through the heating tubes at high velocity (e. g. 6 to 8 feet per second) with extreme turbulence, whereby the danger of local overheating and cracking of the oil is eliminated. A slight pressure is maintained at the discharge end of the outlet of the pipe still into tower 11, by means of a valve 461, in order to minimize vaporization of oil in the tubes of the pipe still heater.

The lighter portions of the highly heated oil thus discharged into tower 11, including both gas oil and lubricating oil fractions, flash into vapor which ascends the column. Such portion as is not vaporized passes downwardly as a liquid over stripping plates 47 and collects in the constricted base 48 to a level 49 automatically controlled through the regulating action of float control 50 on the operation of a re-circulating supply pump 100, to be referred to again hereinafter, which augments and maintains constant the supply of hot liquid residue available for withdrawal through line 52 by pump 53.

In ascending tower 11, the oil vapors flashed off therein undergo fractionation by means generally similar to that already described for tower 10. In addition to an entrainment separator 54, there are provided two sets of fractionating plates 55, 56, with cooperating collector plates 57 and 58, respectively, and reflux condensers 59 and 60, respectively. This enables the lubricating oil components of the ascending vapors to be fractionated, condensed and withdrawn from the tower in two cuts or fractions, one comprising relatively light components corresponding generally to light lubricating oil, and the other comprising medium and heavy lubricating oil. Division of the lubricating oil condensate into this or any other specific number of fractions is a matter of choice and depends largely upon considerations of a practical nature unnecessary to discuss. Combining the two fractionating sections in a single tower, as here illustrated, is economical as to cost of construction and operation, but is not essential to the practice of the invention.

The vapors evolved at 46 pass upwardly through tower 11 with a whirling movement, as previously explained in connection with tower 10, in intimate contact with a moderately high-boiling lubricating oil reflux provided by condensation in reflux condenser 59 of the heavier lubricating oil fractions of the vapors, this reflux being directed to the topmost of the plates 55 largely by side ring 591 and filming down over the remaining plates of this lower series. Lower boiling lubricating oil vapors pass beyond condenser 59, whirling upwardly in contact with descending films of liquid lubricating oil reflux provided by condensation in condenser 60 of those lower-boiling lubricating constituents which escape condensation therebelow. This reflux is delivered to the topmost of plates 56 by circular distributor 601.

Vapors of gas oil and any other lighter products escape condensation in reflux condenser 60 and pass on into water-cooled final condenser 61, where the principal gas oil fraction is condensed, together with some malodorous and unstabilizing impurities. Residual uncondensed vapor and gases are drawn away at high rate of speed through large diameter offtake 62 by suitable exhausting means (not shown) which, for this tower, may advantageously be a 3-stage vacuum pump.

In a typical instance, the exhausting means just mentioned is operated at such rate as to maintain an absolute pressure of about 4 millimeters of mercury at the top of the column just below the reflux condenser 60. Under such conditions, employing the special low resistance type of fractionating plates hereinabove recommended, there is a vacuum drop or pressure increase of only about 8 millimeters down through both fractionating sections and the entrainment separator 54, the absolute pressure at the discharge inlet 46 being thus only about 12 millimeters, while the pressure immediately below the collecting plates 57 and 58 is approximately 11 millimeters and 7 millimeters, respectively. These figures are of course not to be understood as in any sense restrictive, although they are characteristic of good practice within the scope of the invention.

Condensed light lubricating oil distillate is led away from collecting plate 58 through line 63 to constant level seal tank 64, from which it is withdrawn by pump 65 and pumped through line 66, heat exchanger 67, water-cooled cooler 68, and line 69 to storage.

Similarly, medium-heavy lubricating distillate condensed in the lower fractionating section and collecting on plate 57 passes to storage by way

of line 70, seal tank 71, pump 72, heat exchanger 73, water-cooled cooler 74 and line 75.

Pumps 65 and 72, like all other pumps in the system drawing liquid oil from a container maintained under high vacuum, are located a sufficient distance below their respective seal tanks 64 and 71 to ensure the necessary head of oil on their intakes for proper operation.

In a typical instance, the vapor temperatures in tower 11 may be approximately as follows: 560° F., just below collecting plate 57; 500° F. just below reflux condenser 59; 465° F. just below collecting plate 58; and 350° F. just below reflux condenser 60.

The reflux condensers 59 and 60 utilize as their cooling medium the charging stock which passes through the cooling spaces thereof prior to being delivered into tower 10. For this purpose, the crude charging stock line 17 is extended as shown on the drawings, the charging stock flowing therethrough and through the cooling spaces of the said reflux condensers in the directions indicated by the arrows. The cooling of the lubricating distillates in heat exchangers 67 and 73 is similarly effected by means of the initial charging stock to be preheated, branch piping 76 and 77, connected with charging stock line 17, being provided for said heat exchangers 67 and 73, respectively, to attain this end. Suitable valves (not shown) may be provided in the piping system to regulate and vary the distribution of the crude charging stock to the cooling spaces of the several heat exchangers and reflux condensers referred to.

The gas oil fraction condensed in condenser 61, which amounts in a typical instance to around 12 to 14 per cent of the crude charging stock, is led away to storage by way of line 78, seal tank 79, pump 80 and line 81, the arrangement and automatic operation of pump 80 being similar to that already described for the other similarly functioning pumps. This gas oil fraction also includes some malodorous and unstabilizing contaminants, as already stated.

The several seal tanks assocated with tower 11 are provided with suitable pressure balancing connections 641, 711, 791, as shown.

Unvaporized liquid oil which may be at a temperature of around 600° F. in a typical instance, is drawn from the base of tower 11 by pump 53 and forced at high velocity and with turbulent flow through tube banks 82, 83, of pipe still 14, being thereby heated to a still higher temperature, which is the maximum for the process and which may be from 700° to 725° F. At this temperature, under slight pressure regulable by valve 831 it is discharged into tower 12 at point 84, and flash vaporization of the heaviest lubricating oil fractions obtainable as overhead distillates there takes place. The absolute pressure maintained at the flash vaporization point 84 in tower 12 should be the minimum for this location in any of the towers. A pressure of 6 millimeters absolute and vapor temperature of about 670° F. at this point in tower 12 is good practice, with a pressure of 3 millimeters and vapor temperature of 490° F. at the top of the column above the fractionating section comprising fractionating plates 85, collecting ring plate 86 and entrainment separator 861. The heavy lubricating oil vapors flashed off at 84, together with a very small percentage of lower boiling vapors which are practically all non-lubricants, pass upwardly through the system of fractionator plates 85 (eight in number in this instance) in intimate contact with down-flowing films of condensed heavy lubricating oil vapors in reflux condenser 87 which may be water-cooled as here shown. Distributor 871 delivers this liquid reflux to the topmost of plates 85.

Reflux condenser 87 is so operated, through appropriate regulation of its cooling water, that a very small proportion of lubricating oil vapors escape condensation therein, and pass therefrom, together with residual uncondensed lighter vapors and gases, into final water-cooled condenser 88, where a small amount of condensate is formed and passes off through line 89 and thence by way of seal tank 90, automatically controlled pump 91 and line 92, to storage. Residual uncondensed vapors and gases are drawn away through large-diameter offtake 93 by suitable exhausting or vacuum-producing means (not shown). The condensate formed in condenser 88, which commonly amounts to around 0.5 per cent of the crude charging stock, comprises about equal proportions of lubricating oil and contaminants of malodorous and unstabilizing character. It is good practice to allow this small quantity of lubricating constituents to escape condensation in condenser 87 in order to ensure better separation of said contaminants from the heavy lubricating distillate drawn off from plate 86. This heavy lube distillate passes to storage by way of seal tank 841, pump 842, water-cooled cooler 843, and line 844; operation of the pump being controlled by constant-level control float device 845. Seal tanks 841 and 90 are provided, respectively, with pressure-balancing connections 846 and 901, as shown.

The unvaporized liquid residue of the oil charged into tower 12 constitutes the final residuum obtained in the present process from the primary distillation after all the extractable lubricating values have been vaporized from the initial charging material. This final residuum, which may be in the nature of a flux oil, passes down over stripping plates 94 and accumulates in the base of tower 12 to a level maintained constant through suitable connections of a float control device 95 to flux oil pump 96 which draws flux oil, at a temperature of, say, 650° F., from the base of tower 12 through line 951 at an automatically regulated rate and delivers it to storage by way of line 97, heat exchanger 98, and line 99. In heat exchanger 98, the extremely hot flux oil gives up a large part of its heat units to the initial crude charging stock which has already been preheated to a considerable extent by passage through reflux condensers and heat exchangers operated in conjunction with tower 11, as already described.

Associated with pipe still charging pumps 43 and 53, which may conveniently be electrically driven, are shown steam stand-by pumps 431 and 521, respectively, for alternative operation in cases of current supply failure.

In order to increase the efficiency of vaporization in tower 12 in effecting as complete removal as possible of lubricating values from material being processed, without at the same time increasing the temperature of the pipe still discharge into tower 12, a portion of the very hot flux oil collecting in the base of tower 12 may be re-circulated through pipe still 14. This may be effected in various ways, but a good practical method is that shown on the drawings wherein the portion of hot flux oil (e. g. at 650° F.) to be thus re-circulated is picked up by pump 100 and delivered through line 101 to sump 48 at the base

of tower 11, the operation of the pump being automatically regulated as to rate by liquid level control device 50 suitably connected to the steam supply thereof, as shown. This very hot flux oil is thus combined with the somewhat cooler (e. g. 600° F.) unvaporized liquid residue resulting from the flash vaporization occurring in tower 11. The volume of the material pumped through pipe still 14 by pump 53, and discharged at 84 into tower 12, is thus augmented materially and its total available heat content much increased without the necessity of increasing the predetermined desirable pipe still discharge temperature into tower 12. This re-circulation of flux oil from tower 12 therefore accomplishes the desired purpose of effecting the maximum vaporization of heavy lubes in tower 12 at a lower pipe still discharge temperature than would otherwise be feasible. An additional advantage gained by re-circulating the residual unvaporized material from the base of tower 12 is that it permits maintenance of a constant thruput through the tubes of pipe still 14 and facilitates the operation at a constant discharge temperature into tower 12, irrespective of possible variations in operating conditions to a greater or less extent in the preceding parts of the primary distillation system.

Referring now to Fig. 2 of the drawings, the second stage of the complete process of the present invention, in a typical practical embodiment thereof, will now be described in detail. This second stage consists, as already stated, in a secondary or re-run distillation, by discontinuous or batch distillation under even lower absolute pressures than were used in the first stage, of the unfinished lubricating oil distillates obtained in the primary continuous distillation hereinabove described.

In utilizing the apparatus system illustrated in Fig. 2, which is especially desirable in practicing the invention, the re-run distillation, although essentially batch or discontinuous in character so far as the nature of the distillation itself is concerned, is nevertheless semi-continuous from the standpoint of plant manipulation. That is to say, although the lubricating components are vaporized in the order of their respective boiling points which is characteristic of batch distillation, the arrangement of the apparatus is such that, after running a given charge down to a residue or bottoms of, say, 5 or 6 per cent, it is not necessary to shut the re-run unit down, or release the vacuum, or clean out the still, before introducing another charge to be re-run. This is a point of great practical advantage as can readily be seen.

Generally described, this secondary or re-run distillation unit comprises a relatively large capacity vaporizer 110 adapted to contain a suitably large volume of the oil to be processed, in a body having an extensive free upper surface to facilitate rapid vaporization. The vaporizer, which in this instance takes the form of a shell still, is surmounted by a reflux fractionating tower 111 containing fractionating elements 112. These may be similar to those used in the primary distilling system, but they should most desirably be of such character that resistance to vapor flow is reduced even further and, indeed, to the utmost possible minimum consistent with sufficiently intimate and prolonged contact between the counter-flowing vapor and liquid to ensure adequate fractionation and clean separation of impurities from the desired lube distillate under the conditions of operation. Since the quick separation accomplished in the primary distillation stage by continuous flash vaporization has removed to such a great extent the constituents causing the most trouble in batch distillation, and also because of the far more homogeneous character of the vapors that will now be evolved during any given period of the batch distillation, a much smaller load is thrown on a fractionator in such batch distillation than would otherwise be the case. This permits using at this stage fractionating means which offers extremely little resistance to vapor flow and which, although functioning efficiently under these conditions, would not serve well under usual conditions.

The form and arrangement of plates 112 in the fractionating system here illustrated by way of typical example is shown in greater detail in Fig. 5. Liquid reflux is delivered to the top of the series of annular plates 112 by distributor 123a, the liquid following the course indicated by the solid arrows 112a in filming downwardly over the plate surfaces. By means of curved vanes 112b, the ascending vapors, whose course is indicated by the dotted arrows 112c, are given a whirling movement and are also alternately divided into three passes or streams and reunited into a single stream, as they ascend. This fractionator construction is not in itself novel, but it has not been used, prior to this applicant, in connection with high vacuum distillation of lubricating oils.

In order to reduce to a minimum any possibility of locally overheating and thus cracking the oil to be re-run, the vaporizer 110 is not itself heated by contact with flame or hot furnace gases, but the oil to be processed is heated by continuous passage through a pipe still 113, oil being continuously discharged into the vaporizer through line 114 and withdrawn therefrom through line 115 by a centrifugal circulating pump 116 and returned through line 117 to the pipe still for further heating to progressively higher temperatures. The oil passes through the tubes of the pipe still heater at high velocity (e. g. 6 to 8 feet per second) and in a state of extreme turbulence, thereby practically eliminating any chance of locally overheating and cracking portions of the oil being processed. In order to permit very rapid circulation of a large volume of oil per minute through the pipe still heater and vaporizer the pipe still may desirably be of what is known as multi-pass construction, four passes being indicated at 113a for the convection bank of tubes and at 113b for the radiant bank, the several passes being connected in parallel by suitable headers in a known manner. Such an arrangement, together with the ample size of the circulating connections 114, 115, 117, and high operating capacity of pump 116, permits circulation of the oil through the system at the rate of approximately 650 gallons per minute where, in a typical instance the total initial charge to the system is 21,000 gallons. Generally speaking, circulation of the charge through the system, throughout the distillation, at a per minute rate of about 2 to 4 per cent of the volume of the inital charge of material to the rerun unit, is desirable in practice. Since the volume of the charge in the re-run unit decreases to a residue of say, 5 to 6 per cent, the circulating rate, in terms of the proportion of the charge at later stages, increases greatly as the distillation proceeds.

In conducting the secondary or re-run distilla-

tion with the aid of the system illustrated in Fig. 2, feed stock derived from the primary distillation already described, and comprising any of the unfinished lube distillates thus obtained, either singly or in any desired combination, is charged from the re-run charging tank 118 into the system through line 120 in any suitable manner, but most desirably in a manner to be described in greater detail presently. The normal charging level in vaporizer 110 is indicated at 119. With the centrifugal circulating pump in operation, the oil is circulated rapidly through the system in closed circuit, as previously described. The rate of circulation may be observed by means of a suitable flow meter 122 interposed at any convenient point in the circulating lines connecting the heater and the shell still vaporizer. The pipe still heater, in the present example, may be of sufficient capacity (i. e. heating surface area) to raise the temperature of the oil in the shell still approximately 70° F. per hour, and during the initial heating up of the charge, the pipe still heater may be fired at this rate; so that within a period of from 3 to 5 hours, depending on the type of charge being re-run and other factors, the oil is heated to its initial boiling point. It may be stated that prior to running the charging stock into the re-run vaporizer, it is desirable to treat it with approximately 0.2 per cent by volume of a neutralizing agent such as caustic soda of about 40° Baumé strength, this neutralizing solution being thoroughly mixed with the charging stock.

The system comprising the re-run shell still, the fractionating tower, the reflux condenser 123, and final condenser 124, is held under high vacuum throughout the entire distillation of the lubricating fractions by powerful exhausting means which pull all fixed gas and uncondensed vapor out of the system at high velocity through large-diameter offtake 125. In a typical instance, the absolute presure in line 125 at the outlet of condenser 124, may be 3 millimeters of mercury, while in the vapor space above the charge in shell still 110 it may be 5 to 6 millimeters, a loss of only 2 to 3 millimeters in vacuum. Any suitable exhausting means of sufficient capacity may be used for this purpose, but in practice it is found practical in this instance to use a 3-stage ejector equipment, such as that indicated generally at 126, or a 3-stage reciprocating Worthington pump especially developed for this purpose. The tail water from operation of the ejector equipment, together with that which has been used for cooling the condensers 123, 124, may be carried off through water line 127 and further utilized to cool the finished lubricating oil distillate, as will presently appear. By-pass 128 is provided in the water-cooling system of condensers 123, 124, together with controlling valves 129, 130, whereby the operation of reflux condenser 123 can be regulated and controlled to best advantage. It may be here stated that the operation of the several reflux condensers comprised in the primary distillation unit shown in Fig. 1 may be and preferably is controlled in a manner similar to that for reflux condenser 123 of the re-run unit.

As soon as the temperature of initial vaporization is reached in the re-run shell still, the rate of firing of the pipe still heater is controlled to conform with the rate of distillation desired. In a typical instance, where the initial charge consists of the combined lubricating distillates obtained from tower 11 of the primary system, vaporization of relatively light products begins at around 350° to 360° F., and the rate of firing is thereafter so regulated as greatly to reduce the rate at which the temperature of the oil increases, especially during the distillation of the lubricating oil fractions proper, which may be taken to begin when the stream coming over shows a viscosity of 50 to 60 seconds Saybolt (at 100° F.). The temperature of the charge at this point is ordinarily about 375° to 400° F. From then on until the heaviest lubricating oil fractions have been distilled over, which usually requires around 12 to 14 hours in accordance with the present process, the temperature of the charge is raised very slowly and at a substantially uniform rate to a final distillation temperature of about 560° to 575° F., and not exceeding 600° F. as a maximum. The residue of 5 or 6 per cent, to which the charge is run down, is a very heavy high grade lubricating stock which may be accumulated and re-run by batch distillation, yielding, directly, overhead distillates as finished high grade lubricating oils of good color and stability, with viscosities as high as 110 seconds Saybolt (at 212° F.). In the redistillation of the heavier lube distillate obtained from primary tower 12, the final temperature, upon batch redistillation, may be around 650° F. The residue is a good grease stock.

Distillation in the manner just described means a temperature rise approximating 150° to 200° F. during 12 to 14 hours of distillation, which is an average rise of less than 0.25° F. per minute or 15° F. per hour. It may be stated that, in practicing the present process the rate of temperature increase during distillation of the lubricating oil fractions in the re-run or secondary distillation should not average more than 0.5° per minute at the most. This is typical of the kind of distillation characterizing the second stage of the complete process of the invention and well illustrates how materially it differs from the rapid preliminary distillation effected in the primary stage of the process.

The discharge from the pipe still heater to the re-run shell still is controlled by a valve 114a in such manner that a slight positive back pressure (e. g. 5 lbs.) is maintained on the oil to minimize vaporization in the tubes of the pipe still heater. After entering the shell still, the line 114 bends downwardly as shown and terminates in a discharge header 130a extending longitudinally of the still and close to the bottom thereof. This discharge header is provided throughout its length with numerous small diameter discharge openings 131 whose total area may desirably be less than the cross sectional area of the discharge header. This restriction of discharge opening area permits the discharge of the hot oil from the pipe still heater at very high velocity into the lower part of the shell still in many small jets, thus constantly maintaining the body of oil in the shell still in highly turbulent condition and ensuring substantial uniformity of temperature throughout said body The velocity of the oil at the point of discharge through the small openings 131 is on the order of about 30 feet per second. This provision is of great importance in increasing the efficiency and uniformity of vaporization at the extremely low rate of temperature increase above referred to, thus further favoring uniformity in composition (narrow cut) of the vapors evolved during a given period of time, say several minutes, a fact which, in spite of the very low pressure drop

through the fractionator and condenser, as already pointed out, makes for relatively high efficiency in the subsequent fractionation of said vapors and clean separation from the lubricating distillate of the small quantity of relatively volatile malodorous and unstabilizing contaminants included therewith.

The vapors rising from the oil in the shell still pass through the large diameter vapor offtake 132 into the fractionating tower and through the entrainment separator 133 located in the base thereof. This entrainment separator may be the same type as those employed in the primary distillation unit (Fig. 1). The vapors then pass upwardly through the system of fractionating plates or trays 112, previously referred to.

The vapor temperature at the top or outlet of reflux condenser 123 is carefully regulated and controlled throughout the distillation in such manner that no condensation of malodorous or unstablizing products, which are relatively low boiling as compared to the lubricating constituents of the distillation vapors, can occur in the reflux condenser. That is to say, care is taken throughout the distillation to so adjust the water cooling of reflux condenser 123 that the temperature of the uncondensed vapors and gases leaving the same is always high enough to ensure that, at any given time, said vapors include not only those of the aforesaid malodorous and unstabilizing constituents, but also a small proportion of vapors of the considerably higher boiling lubricating constituents then coming over. The precaution, observed also in the operation of tower 12 of the primary distillation system as already pointed out, renders still more certain substantially complete and clean the separation of objectionable contaminants from the desired lubricating oil distillates.

Vapors escaping condensation in reflux condenser 123 pass into condenser 124 where they are cooled to a much lower temperature sufficiently low to compel most of the residual vapors to condense. This condensate, which is of unpleasant odor and unstable, is led away through line 134 and look box 135 to tank 136. It generally amounts to around 0.5 per cent of the quantity of unfinished lubricating distillate originally charged into the re-run or redistillation unit, and it is practically the same in character and composition as the malodorous condensate derived from the final condenser 88 of tower 12 of the primary distillation; and in the practice of the process, both condensates are commonly termed "header" or "stink oil."

The lubricating oil distillate condensed in the tower 111 is led away in side stream from collecting plate 137 through line 138 to constant level seal tank 139. The operation of the fractionating system is so regulated and controlled that the lubricating distillate leaving the tower at any given time is very hot, being practically at its point of initial vaporization. During the greater part of the distillation, the temperature at which it leaves the column or tower is not more than 50 to 60 degrees Fahrenheit below that of the vapors entering the base of the column, and only near the commencement of the distillation is it sometimes as much as 100 degrees below that of the vapors entering the column. This aids greatly in preventing absorption of malodorous constituents from the distillation vapors by the condensate while it remains in the tower; and the possibility of absorption is still further minimized by the maintenance of the extremely high vacuum on the hot condensate even after it leaves the column, thus ensuring withdrawal therefrom of even minute traces of such volatile contaminants. The condensate is withdrawn through line 140 and cooler 141 by pump 142, which then pumps it through line 143 to storage. Line 143 may be provided with sight glass 144 and meter 145, as shown. The lube cooler 141 may utilize as cooling medium the water discharged through line 127 from the ejector system and condensers at the top of the tower. The operation of lube pump 142 is automatically regulated by float control device 146, as indicated.

The lubricating oil distillate going to storage through line 143 is the desired final product of the complete process. It is sweet, stable finished lubricating oil, of excellent color, resulting directly from straight overhead distillation and requiring no further refining or purifying as by acid treatment and/or filtration. Its particular specifications as to viscosity, boiling range, etc. depend, of course, upon which of the several distillates obtained from the continuous primary distillation, or what combination of such distillates, was used as the initial charge in the re-run distillation.

Referring again to Fig. 2 of the drawings, a standby pump 147, which may desirably be steam operated, serves in the capacity of a residue pump when a re-run distillation or cycle is completed, and also serves to some extent as a charging pump in such manner that the operation of the re-run unit may be semi-continuous in the sense already explained hereinabove. When a given re-run still charge has been distilled off until a residue of about 6% remains in the re-run shell still, firing of the pipe still heater is discontinued, centrifugal circulating pump 116 is stopped, valve 152 is closed, and circulation of the aforesaid residue is continued at a reduced rate by means of the steam stand-by pump 147, which is arranged in parallel with pump 116 by valved suction and discharge connections 147a and 147b, respectively, the valves in these connections having been opened for the purpose. While the described re-run distillation has been going on, a fresh supply of charging stock has been prepared for re-running and has been placed in the re-run charging tank 118. When the steam stand-by pump has been operating for some time as just described and the stream of lubricating oil condensate flowing from tower 111 through line 138 has ceased, valve 114a in discharge or delivery line 114 from the re-run pipe still heater is closed, and valves 148 and 149 in residue discharge line 150 are opened, thus causing the hot residue to be pumped from the system through residue cooler 151 to storage. Knowing the actual quantity of residue remaining in the system at the end of the distillation, and knowing the capacity of the steam stand-by pump 147 or observing the rate of flow of the residue as indicated by flow meter 122, an operator can determine the exact time when all of the residue has been removed from the shell still. At this time, the suction valve 152 in line 115 leading from the shell still remaining closed, the valve 153 in line 120 leading from the re-run still charging tank 118 is opened and the valve in line 147a is closed, so that the steam stand-by pump then draws fresh charge from the tank through branch line 154 in which valve 155 has been opened for the purpose. The stand-by pump continues to operate until the remaining portion of the residue, which is thus forced through the pipe still ahead of the fresh charge

from tank 118, is entirely removed from the system, whereupon valve 114a in the heater discharge line 114 is opened, and valve 149 in the residue discharge line 150 is closed. The proper time for effecting this operation is readily determined from a knowledge of the capacity of the tubes in the pipe still heater and the transfer lines, and the rate at which the steam stand-by pump is operated. A further guide is afforded by noting the temperature drop which suddenly occurs at the outlet in the pipe still heater as the last of the hot residue leaves it. Shortly after the first portion of the fresh charge reaches the re-run shell still, the steam stand-by pump 147 is shut down, the valves in lines 147a and 147b are closed, and the centrifugal circulating pump is started to complete the charging of the system with fresh lube distillate stock to be re-run; valve 121 in the suction connection of the pump to the charging tank 118 being opened and remaining open until the charging operation is finished, whereupon it is closed and valve 152 is opened again. In the meantime firing of the pipe still has been resumed.

In the particular installation here illustrated, the discharge side of stand-by pump 147 is also connected by valved line 147b to discharge line 150, whereby the pump may draw oil from the shell still through line 147a and discharge it directly into line 150, which may be necessary or desirable upon occasion.

The manipulation of the re-run unit to render its operation virtually semi-continuous may of course be accomplished in other ways than that described in specific detail hereinabove, such description being given merely by way of concrete example for purposes of explanation. This general method of procedure is very advantageous because it renders operation of the re-run distillation materially more economical and efficient. Since the shell still, fractionating tower, pipe still heater and all lines connecting the heater with the shell still are insulated, the whole system, which is at a temperature well above 500° F. at the end of the distillation, retains a large amount of heat which is thus conserved and utilized to impart a rise in temperature to the fresh charging stock amounting to 70°–80° F. before firing of the pipe still heater is begun. Also, the interval during which the fresh charge is being heated up to initial boiling point for the next distillation is reduced by about one hour as compared to the time required to heat a fresh charge up to the same temperature in a cold unit.

Seal tank 139 has a pressure balancing connection 139a to tower 111, as shown. Seal tank 136 requires a similar pressure balancing connection unless, as in the illustrated installation, it is located far enough below condenser 126 to provide a balancing leg of oil in line 134.

The temperatures and other detailed operating conditions hereinabove given are to be understood merely as indicative of good practice in handling typical topped crudes such, for example, as Urania (Louisiana) and Colombia (South America) crudes. Obviously such operating conditions will vary to some extent with the type of crude charging material, and hence the operating data is to be regarded as given for purposes of illustration and not as rigidly restrictive.

It is very important, both in the primary and re-run distillations or stages of the complete process, and especially in the re-run distillation, to rigidly exclude access of air into the distilling system, since the occurrence of even very slight oxidation makes it impossible to produce directly, as overhead distillates, finished lubricating oil products of the character here in question. All seams and joints exposed to temperature changes should be welded, and the entire system should be made absolutely tight.

Where the crude mineral oil charging stock is of paraffine base, either wholly or to a large extent, the complete process of the invention includes a de-waxing treatment of the lube distillates obtained in the primary distillation, before re-running them by batch distillation. Such de-waxing may be effected in any of various ways now known to the art or suitable for the purpose.

By a stable lubricating oil, as the expression is herein employed, is meant one that does not undergo changes in storage and use which are in the nature of breakdown or decomposition and which are recognized by the industry as characterizing inferior lubricating oils. A lubricating oil which, upon being heated to 300° F. and being held at this temperature for 10 minutes, does not darken in color more than one-half point on the standard Saybolt color scale, does not acquire a bluish cast in place of the desired greenish bloom, and does not have or develop an unpleasant odor, is a stable oil.

What I claim is:

1. The process of manufacturing lubricating oils which comprises first distilling suitable mineral oil charging stock to effect a relatively quick preliminary separation of lubricating oil components therefrom by rapidly heating said charging stock in admixture with a basic neutralizing agent to the necessary maximum vaporizing temperature not substantially exceeding 725° F. and continuously flash-vaporizing lubricating oil components therefrom in at least two successive steps under diminished pressure not substantially exceeding 50 millimeters mercury absolute, and condensing from the resultant vapors, under such diminished pressure, lubricating oil distillate relatively free from non-lubricants but not color stable; then subjecting the unstable lubricating oil distillate so separated to redistillation in the presence of an admixed basic neutralizing agent at pressure not substantially exceeding 25 millimeters absolute and at vaporizing temperatures which increase slowly to the maximum required for substantially complete vaporization of the lubricants at the distillation pressure employed, and subjecting the resultant distillation vapors to column fractionation under such diminished pressure, and leading away in side stream lubricating oil fractions as they are evolved and condensed in the order of their vaporizing points, while separately and rapidly withdrawing uncondensed vapors comprising unstabilizing contaminants, said fractions being, without acid treatment or filtration, finished lubricating oils; all the stated operations being conducted under non-oxidizing conditions.

2. The process of manufacturing lubricating oils which comprises first distilling suitable mineral oil charging stock to effect a relatively quick preliminary separation of lubricating oil components therefrom by rapidly heating said charging stock in the presence of an admixed basic neutralizing agent to the necessary maximum vaporizing temperature not substantially exceeding 725° F. and vaporizing lubricating oil components therefrom under diminished pressure not substantially exceeding 50 millimeters mercury absolute, subjecting the resultant vapors to column fractionation and condensing therefrom,

under such diminished pressure, lubricating oil distillate relatively free from non-lubricants but not stable; then subjecting the lubricating oil distillate so separated, in the presence of an admixed basic neutralizing agent, to redistillation at pressure not substantially exceeding 25 millimeters absolute and at vaporizing temperatures which increase slowly to the maximum required for substantially complete vaporization of the lubricants at the distillation pressure employed, subjecting the resultant distillation vapors to column fractionation under such diminished pressure, and leading away in side stream lubricating oil fractions as they are evolved and condensed in the order of their vaporizing points, while separately and rapidly withdrawing uncondensed vapors comprising unstabilizing contaminants, said fractions being, without acid treatment or filtration, finished lubricating oils; all the stated operations being conducted under non-oxidizing conditions.

3. The process defined in claim 2 further characterized by the fact that the temperature of any given condensed lubricating oil distillate obtained in the re-run or redistillation, at the moment said fraction is being led away out of contact with the distillation vapors coming from the locus of vaporization, is not substantially more than 100 degrees F. below that of the vapors then being evolved at such vaporization locus and, during the greater part of such redistillation, is not substantially more than 50 to 60 degrees F. below such vapor temperature.

4. The process as defined in claim 2, further characterized by the fact that in the first distillation the lubricating oil components are vaporized at not to exceed 20 millimeters absolute pressure, and less than 10 millimeters for the heaviest portion of said components; and that in the redistillation, the maximum distilling temperature attained is from 525° to 625° F., and the lubricating oil components are vaporized at not to exceed 10 millimeters absolute pressure; said process being further characterized by the fact that the temperature of any given condensed lubricating oil distillate obtained in the re-run or redistillation, at the moment said fraction is being led away out of contact with the distillation vapors coming from the locus of vaporization, is not substantially more than 100 degrees F. below that of the vapors then being evolved at such vaporization locus and, during the greater part of such redistillation, is not substantially more than 50 to 60 degrees F. below such vapor temperature.

5. The process defined in claim 2, further characterized by the fact that the continuous flash vaporization of the lubricating constituents of the stock is in two successive stages, in the first of which the vaporizing pressure does not exceed 20 millimeters and the temperature is sufficiently high to cause vaporization of the light and medium lubricating constituents, and in the second of which the vaporizing pressure is less than 10 millimeters and the temperature is sufficiently higher to cause vaporization of the remaining heavier lubricating constituents; while in the redistillation the lubricating constituents are all vaporized at not to exceed 10 millimeters absolute pressure and at temperatures lower in all cases than that employed in the first flash vaporizing stage aforesaid; said process being further characterized by the fact that the temperature of any given condensed lubricating oil distillate obtained in the re-run or redistillation, at the moment said fraction is being led away out of contact with the distillation vapors coming from the locus of vaporization, is not substantially more than 100 degrees F. below that of the vapors then being evolved at such vaporization locus and, during the greater part of such redistillation, is not substantially more than 50 to 60 degrees F. below such vapor temperature.

6. In the manufacture of useful mineral lubricating oil products from mineral oil containing constituents which give rise upon distillation of the oil to difficultly separable but relatively low-boiling malodorous and unstabilizing contaminants, the process which comprises mixing a basic neutralizing agent with such a mineral oil charging stock, effecting a relatively quick preliminary separation of lubricating oil components from said charging stock by rapidly heating the same to the necessary maximum vaporizing temperature not substantially exceeding 725° F. and continuously flash-vaporizing lubricating oil components therefrom under diminished pressure not substantially exceeding 25 millimeters mercury absolute, condensing from the resultant vapors, under such diminished pressure, unstable lubricating oil distillate, mixing a basic neutralizing agent with said unstable distillate and subjecting the mixture to batch or discontinuous distillation and column fractionation under pressure not substantially exceeding 25 millimeters absolute, and leading away in side stream lubricating oil fractions as they are evolved and condensed in the order of their vaporizing points, while separately and rapidly withdrawing uncondensed vapors comprising unstabilizing contaminants, said fractions being, without acid treatment or filtration, finished lubricating oil; all the stated operations being conducted under non-oxidizing conditions.

7. In the manufacture of useful mineral lubricating oil products from mineral oil containing constituents which give rise upon distillation of the oil to difficultly separable but relatively low-boiling malodorous and unstabilizing contaminants, the process which comprises mixing a basic neutralizing agent with such a mineral oil charging stock, distilling lubricating oil fractions therefrom by continuous flash-vaporization at temperature not substantially exceeding 725° F. and under absolute pressure not exceeding 25 millimeters of mercury, and below 10 millimeters for the major part of the distillation, thereby obtaining lubricating oil distillate which, although relatively free from non-lubricants, is not stable; then mixing a basic neutralizing agent with such unstable lubricating oil distillate and subjecting the mixture to batch or discontinuous distillation and column fractionation under pressure less than 10 millimeters absolute for the major part of the distillation, and leading away in side stream lubricating oil fractions as they are evolved and condensed in the order of their vaporizing points, while separately and rapidly withdrawing uncondensed vapors comprising unstabilizing contaminants, said fractions being, without acid treatment or filtration, finished lubricating oil; all the stated operations being conducted under non-oxidizing conditions.

8. The process of manufacturing lubricating oils as overhead distillates which comprises subjecting to continuous flash-vaporization at temperature not substantially exceeding 725° F., under absolute pressure not exceeding 50 millimeters of mercury and in the presence of an admixed basic neutralizing agent, a mineral oil which contains lubricating oil components in

association with odor-producing components, thereby producing a mixture of lubricating oil vapors with difficulty separable vapors and gaseous lower-boiling malodorous contaminants, condensing from such mixture a lubricating oil distillate which, although relatively free from non-lubricants, is not stable, subjecting such unstable lubricating oil distillate to batch or discontinuous vaporization and column fractionation in the presence of a basic neutralizing agent under absolute pressure not exceeding 25 millimeters of mercury and under non-oxidizing conditions, and leading away in side stream lubricating oil fractions as they are evolved and condensed in the order of their vaporizing points, while separately and rapidly withdrawing uncondensed vapors comprising unstabilizing contaminants, said fractions being, without acid treatment or filtration, finished lubricating oil; all the stated operations being conducted under non-oxidizing conditions.

9. The process set forth in claim 8, further characterized by the fact that in both the continuous flash-vaporization and the subsequent batch or discontinuous vaporization, the lubricating components are vaporized under an absolute pressure not exceeding 20 millimeters, and that during the greater part of the batch vaporization said components are vaporized under an absolute pressure below 10 millimeters.

10. The process of manufacturing lubricating oils which comprises heating mineral oil starting material in the presence of an admixed basic neutralizing agent and flash-vaporizing lubricating components thereof at temperatures not substantially exceeding 725° F., subjecting the resultant vapors to column fractionation and condensing lubricating oil distillate therefrom, all at low absolute pressure, then subjecting such condensed oil to redistillation by circulating it in a closed path, in one portion of which path the oil passes through heating means whereby it is gradually heated to progressively higher vaporizing temperatures, while in another portion of said path the oil, in a body having a large free vaporizing surface, is subjected to absolute pressure not substantially exceeding 25 millimeters of mercury and the lubricating components thereof are vaporized progressively in the order of their boiling points and, still under such low absolute pressure, are subjected to column fractionation and condensed as lubricating oil fractions which are led away in side stream, while uncondensed vapors comprising unstabilizing contaminants are separately and rapidly withdrawn, said fractions being, without acid treatment or filtration, finished lubricating oils; all the stated operations being conducted under non-oxidizing conditions.

11. The process defined in claim 10, further characterized by the fact that the flash vaporization of the lubricating components occurs under absolute pressure not exceeding 20 millimeters and ranging downward to less than 10 millimeters for the heaviest fractions, while in the subsequent redistillation the lubricating components are vaporized under absolute pressure not exceeding 10 millimeters.

12. The process defined in claim 10, further characterized by the fact that said body of oil at the locus of vaporization is maintained in a state of substantial turbulence, whereby fresh portions of the oil are constantly being presented at said free vaporizing surface.

13. The process defined in claim 10, further characterized by the fact that the circulation of the oil during the redistillation is so conducted that oil is discharged from the heating means into said body of oil at the vaporization locus, and oil is withdrawn from said body of oil for return to the heating means, at a rate per minute equal to at least 2 per cent of the total amount of oil initially charged into the redistillation cycle.

14. The process defined in claim 10, further characterized by the fact that in said redistillation, the heating of the oil is so regulated as to increase its temperature at a rate not substantially exceeding 0.5° F. per minute.

15. The process defined in claim 10, further characterized by the fact that in said redistillation, the heating of the oil is so regulated as to increase its temperature at a rate on the order of approximately 0.25° F. per minute.

16. The process of manufacturing lubricating oils which comprises circulating a charge of suitable mineral oil material in a closed path including a heating zone, a vaporizing zone, and the necessary connecting conduits, the greater part of the charge being maintained in the vaporizing zone as a substantial body of oil presenting a large free vaporizing surface, heating said mineral oil material, by repeated passage thereof through said heating zone, over a range of increasing temperatures required to vaporize the desired lubricating oil constituents thereof in the order of their boiling points while maintaining upon the surface of the oil in said vaporizing zone an absolute pressure not exceeding 25 millimeters of mercury, the heating of the charge being so regulated that the average rate of temperature increase does not exceed about 0.5° F. per minute, withdrawing resultant vapors from said vaporizing zone, and condensing lubricating oil distillate therefrom at such low absolute pressure.

17. The process defined in claim 16, further characterized by the fact that the heating of the charge is so regulated that the average rate of temperature increase is on the order of 0.25° F. per minute.

18. The process defined in claim 16, further characterized by the fact that the oil passes through said heating zone in a stream or streams of restricted cross-section and at relatively high velocity.

19. The process defined in claim 16, further characterized by the fact that the body of oil in said vaporizing zone is maintained in a condition of substantial turbulence during the distillation, whereby fresh portions of the oil are constantly being presented at said free vaporizing surface.

20. The process defined in claim 16, further characterized by the fact that the oil passes through said heating zone in a stream or streams of restricted cross-section and at relatively high velocity, and is maintained under superatmospheric pressure until discharged into the vaporizing zone in order to minimize vaporization prior thereto.

21. The process defined in claim 16, further characterized by the fact that the oil passes through said heating zone in a stream or streams of restricted cross-section and at relatively high velocity, and is discharged below the surface of the body of oil in the vaporizing zone in a plurality of jets.

22. The process defined in claim 16, further characterized by the fact that the oil passes through said heating zone in a stream or streams of restricted cross-section and at relatively high

velocity, and is discharged below the surface of the body of oil in the vaporizing zone in a plurality of jets in which the oil has a discharge velocity on the order of 30 feet per second.

23. The process defined in claim 16, further characterized by the fact that the circulation of the oil during the stated procedure is so conducted that oil is discharged from the heating zone into the vaporizing zone, and oil is withdrawn from the vaporizing zone for return to the heating zone at a rate per minute equal to at least about 2 per cent of the total amount of oil initially charged into the cycle.

JOHN E. SCHULZE.