Oct. 15, 1935. J. E. SCHULZE 2,017,820

MINERAL OIL DISTILLATION

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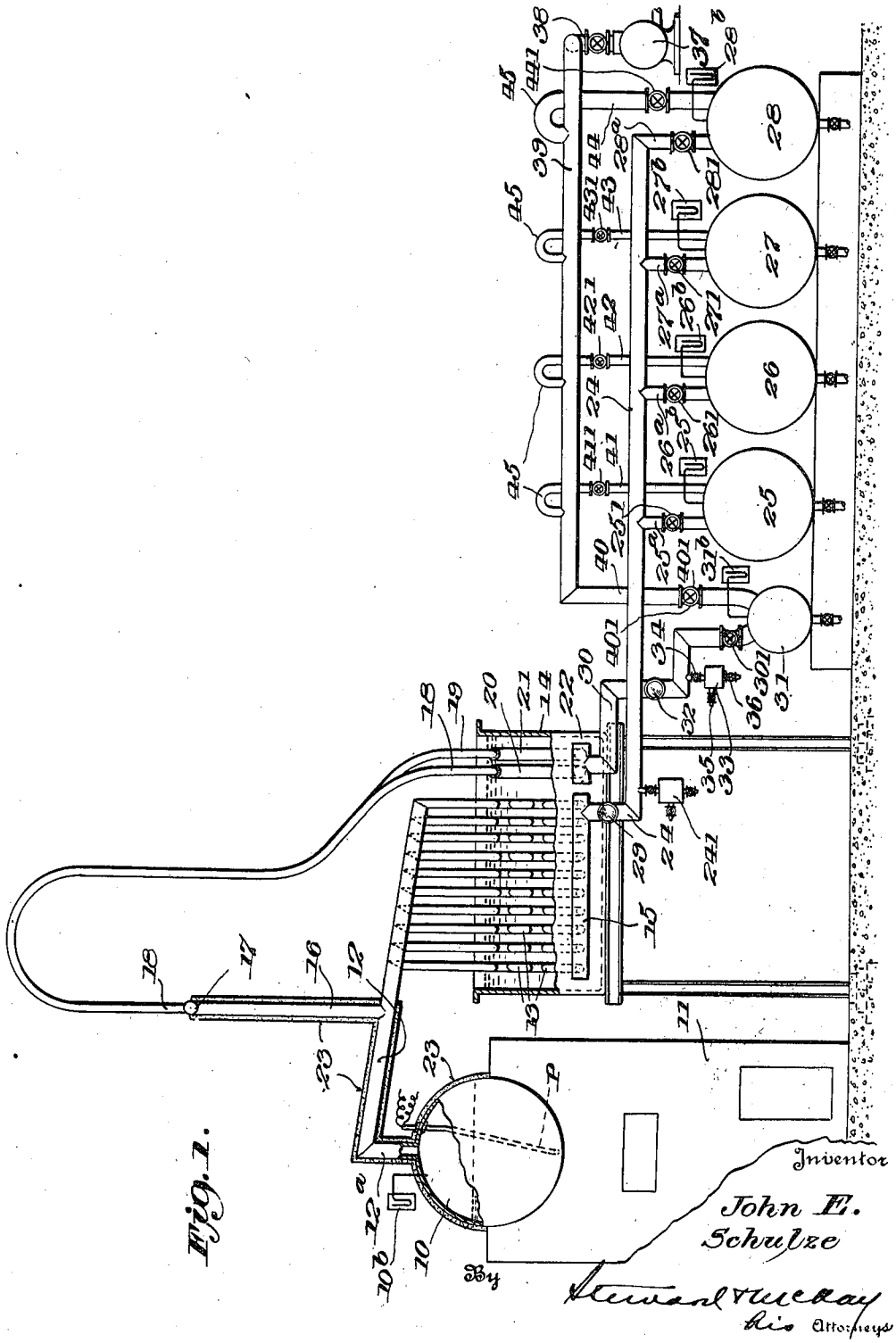


Fig. 1.

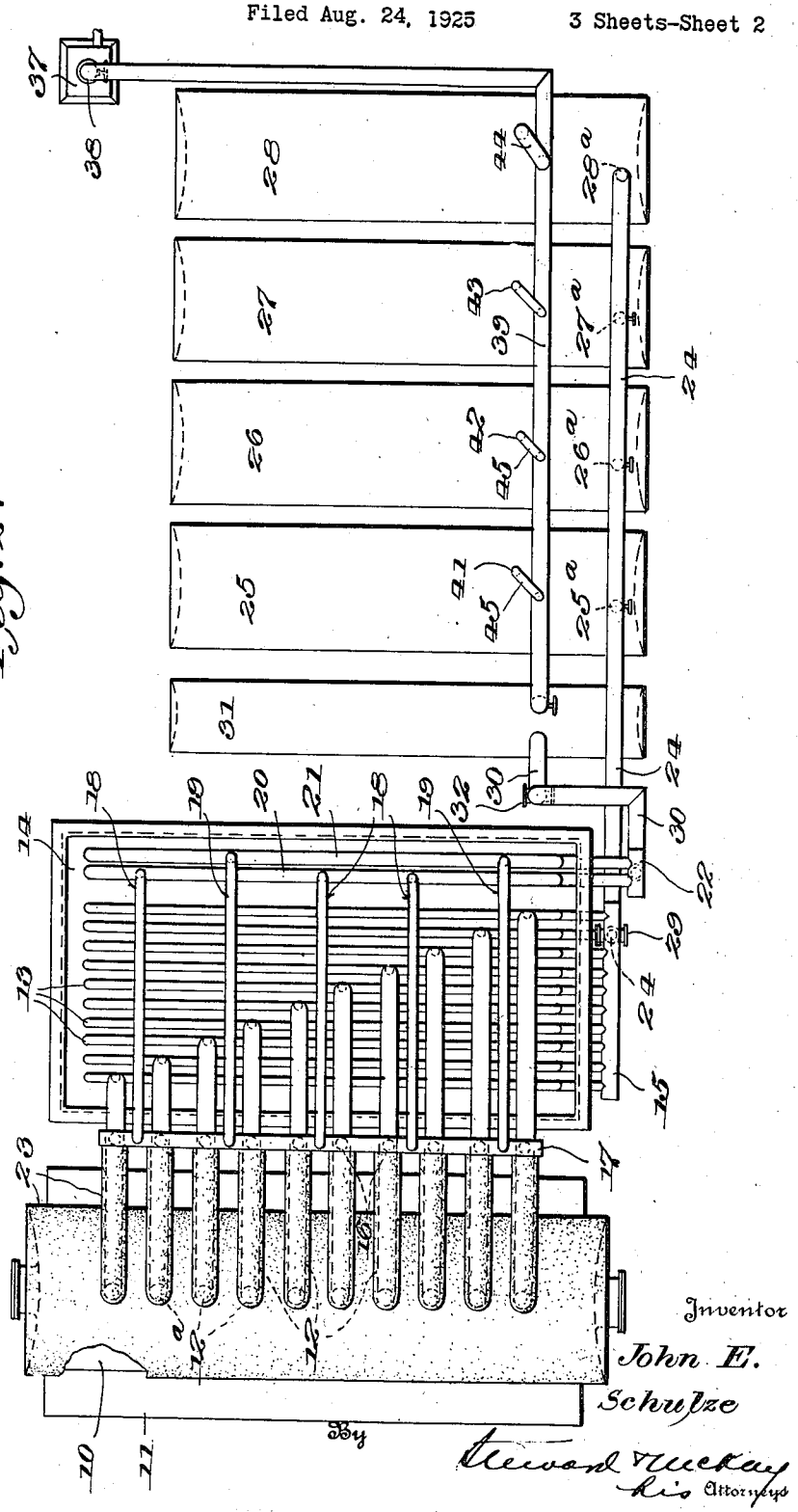
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His Attorneys

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MINERAL OIL DISTILLATION

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Fig. 2.



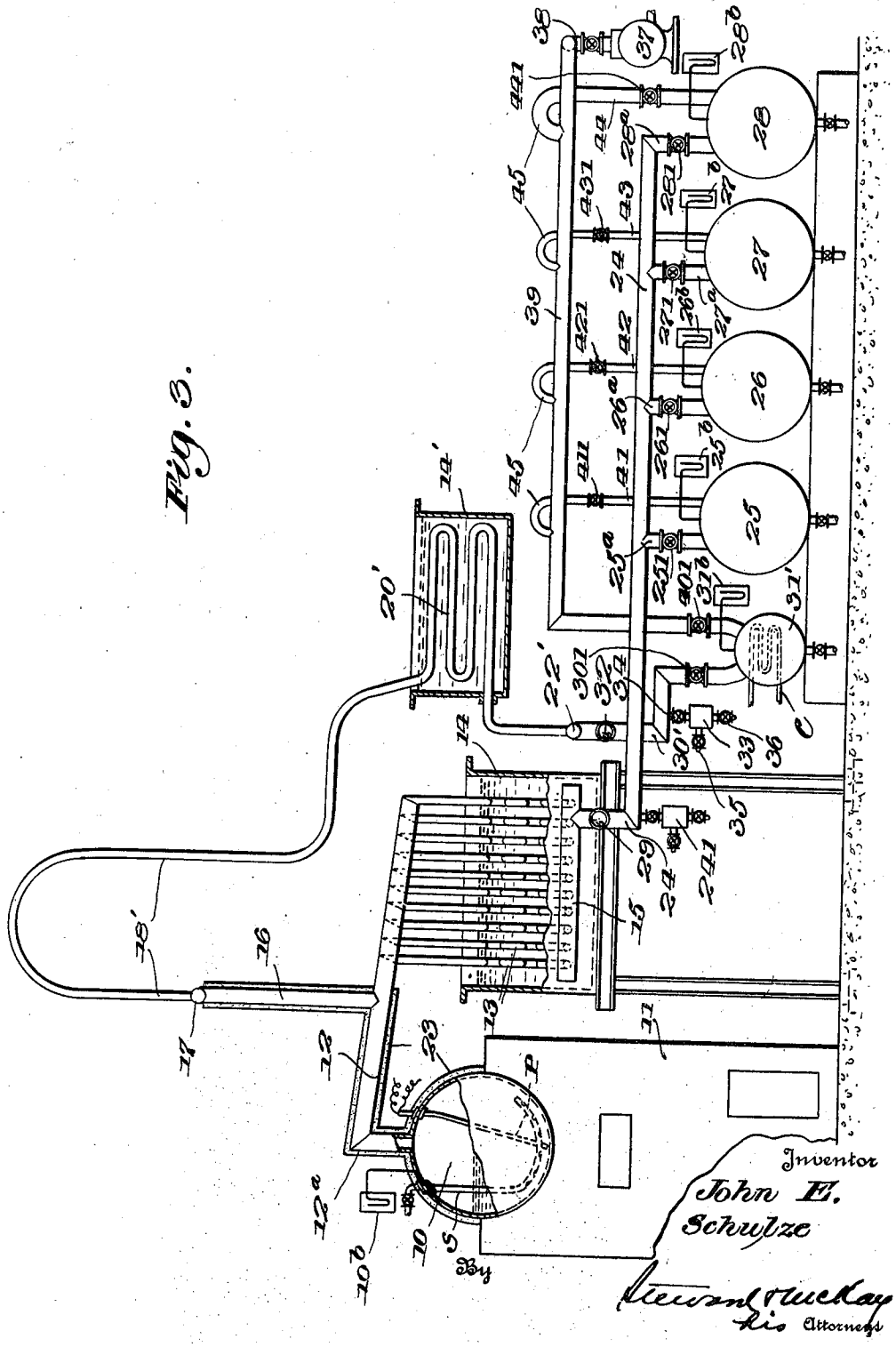
Inventor
John E. Schulze
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His Attorneys

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MINERAL OIL DISTILLATION

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Fig. 3.



Patented Oct. 15, 1935 2,017,820

UNITED STATES PATENT OFFICE

2,017,820

MINERAL OIL DISTILLATION

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

Application August 24, 1925, Serial No. 52,118

26 Claims. (Cl. 196—77)

This invention relates to mineral oil distillation; and in particular it relates to processes of and apparatus for distilling petroleum oils in the manufacture of useful products therefrom, and especially in the manufacture of lubricating oils by distillation under high vacuum. The novel oils resulting from the new processes are also comprised in the invention.

While not restricted in certain of its broader aspects to distillation of lubricating oils under vacuum, the invention is of greatest present utility in that field which will therefore be more particularly referred to hereinafter in explaining the principles upon which the broad invention rests.

In a prior patent, No. 1,448,709, granted March 13, 1923, this applicant has disclosed and claimed a high vacuum distillation process for the manufacture of useful products, notably lubricating oils, from petroleum oils and residues; and the present invention, although broad in scope as above indicated, is concerned more particularly with the manufacture of high grade lubricating oils by methods involving broadly the principles and conditions of operation disclosed in said prior patent.

The patented process aforesaid makes use of distillation pressures which do not exceed 25 millimeters mercury absolute as a maximum and which are most desirably even much lower, commonly 5 millimeters or less. Said process gives excellent results in practice and at the present time is employed commercially on a large scale for the manufacture of high grade lubricating oils. Nevertheless it has been found that with certain types of crudes or residua, especially those high in combined sulfur, nitrogen, organic acids, or complexes including some or all of such constituents, difficulty is sometimes experienced in obtaining by the patented process lubricating oil fractions that are perfectly sweet as to odor and taste and that do not darken in color or assume an undesirable cast after prolonged exposure to light or standing in storage for a considerable time. This instability and other unsatisfactory characteristics of lubricating oil distillates obtained from such starting materials are due to the presence in said distillates of organic acids or compounds of sulfur or nitrogen, or complexes of these, which are tenaciously held by the lubricating distillates notwithstanding the fact that such contaminating impurities are generally of substantially lower boiling point than said distillates.

Among such crude oils above referred to may be mentioned by way of example the following:

Smackover crude	Gravity 17.3° Bé. Sulfur about 3%
Nevada County (Arkansas) crude	Gravity 14° Bé. Sulfur about 3%
Cacalilao (Mexico) crude	Gravity 12° Bé. Sulfur about 4 to 5%.

California and Mexican crudes generally, most of which are high in sulfur and in organic acids especially.

The foregoing are merely typical of starting materials well adapted for manufacture of high grade lubricating oils by the process of the present invention. Heretofore it has been impossible to produce marketable lubricating oils from such starting materials.

Principal objects of the invention are to provide a method or process wherein the difficulties heretofore encountered in utilizing this class of starting materials are overcome and production of valuable and thoroughly satisfactory high grade distillates is made possible, especially lubricating oil distillates, from low grade crudes and residua of the type above discussed; also to provide apparatus for carrying out such method. Other objects are in general to make it possible to obtain from such starting materials sweet and stable mineral oil distillates, whether lubricating oils or not, and also irrespective of whether the distillation be conducted under high vacuum or not. Still further objects and advantages of the invention will become apparent as the description proceeds.

In general, the process of the present invention involves vaporization of the crude petroleum or other mineral oil starting material, or a large part thereof, and fractional condensation of the resultant vapors in such manner that separation of the desired condensate is effected with minimum exposure thereof to contact with the lighter vapors and gases which carry most of the deleterious compounds it is desired to exclude from said condensate, so that little or no opportunity is afforded for said condensate to absorb and become contaminated by said compounds. In the best way of practicing the invention, the desired condensate obtained in the fractional condensation is not allowed to cool materially below the temperature at which it is formed until after there is no further opportunity for contacting with the malodorous vapors and gases before mentioned.

Most desirably, but not necessarily in all cases, the vaporization of the crude oil is accomplished by distillation under high vacuum of the order of magnitude characterizing the process disclosed in said Patent No. 1,448,709 before mentioned. Furthermore, by employing an appropriately arranged apparatus and properly operating the same, it is feasible simultaneously to apply differing degrees of vacuum to the desired liquid condensate on the one hand, and to the uncondensed malodorous vapors and gases on the other, respectively, whereby removal of said vapors and gases from the vicinity of said condensate may be greatly promoted and expedited with advantage.

In processing a given mineral oil starting material in accordance with the invention, it is also highly desirable, where such material is high in impurities such as those hereinabove mentioned, to conduct the distillation in the presence of a small quantity of a neutralizing agent that is alkaline in nature, such as caustic soda, soda ash, lime, or other suitable basic reagent. The employment of such a neutralizing agent may be in connection with an initial distillation of the starting material or in connection with a redistillation of a crude or raw distillate obtained in an initial run. The conjoint use of such a neutralizing agent and the method of separating malodorous volatile products as uncondensed vapors and gases from the desired condensates, in the manner outlined generally above, gives excellent results and constitutes an important feature of the invention, as will more fully hereinafter appear.

In order to afford a full understanding of the principles of the invention, a typical practical embodiment thereof will now be described in detail in connection with the accompanying drawings which illustrate an appropriate apparatus installation for carrying out the process. In these drawings

Fig. 1 is a side elevation of such installation;

Fig. 2 is a plan of same; and

Fig. 3 is a view in side elevation of an installation similar to that shown in Figs. 1 and 2 but modified to permit employment of steam in the distillation. In all the views, parts are shown broken away and in section.

Referring to the drawings, **10** represents a horizontal cylindrical still supported above a heating furnace indicated generally at **11** which may be constructed and fired in any suitable manner to heat the still indirectly rather than by direct contact of flame with the still bottom. In the present instance, the still **10** is especially constructed for operation under extremely high vacuum (i. e. extremely low absolute pressure) of an order of magnitude corresponding to 25 millimeters of mercury (or less) absolute pressure. To this end the still is interiorly braced as, for example, in the manner illustrated in my prior Patent No. 1,448,709, dated March 13, 1923. In order to provide vapor offtake means of ample cross-sectional area, the still is equipped with a plurality of vapor offtakes **12**, in this instance ten in number, of uniform diameter, leaving the still at uniformly spaced points along the length thereof. The vertical portion **12a** of each of the vapor offtakes **12** is relatively very short in order to minimize refluxing of distillate to the still. To the same end, the remainder of each of the vapor offtakes is inclined downwardly from the vertical portion **12a** toward the cooling coil **13** into which each offtake **12** leads. These cooling coils **13** are submerged in cooling water contained in a box or tank **14**. Said cooling coils, one of which is provided for each offtake **12**, discharge into a large-diameter collecting header **15**.

At **16** is a riser, one for each offtake **12**, the risers **16** all entering a horizontal header **17**. From the header **17** two sets of vapor lines **18** and **19** extend upwardly a considerable distance above the header **17** and then bend downwardly to connect with cooling coils **20** and **21**, respectively, disposed in cooling tank **14**. In the present instance, there are three vapor lines **18** in one set and two vapor lines **19** in the other set. The coils **20** and **21**, of which there are two in this case and which are of larger diameter than the lines **18** and **19**, discharge into a collecting header **22** of still larger diameter.

The described arrangement of the vapor pipes **18** and **19** in sets discharging, respectively, into separate cooling coils **20** and **21** is in harmony with the general purpose of ensuring ample cross-sectional area at all points in the piping system leading from the still. This general purpose may be accomplished in various ways, of course, but the arrangement here shown by way of example is effective and satisfactory in practice.

The piping system just described, beginning with risers **16** and extending to header **22** constitute parts of what may be conveniently termed the "header distillate" line or "deodorizer" system, further reference to which will be made hereinafter.

Provision is made for guarding against substantial condensation of vapors at the point where they leave the still and for a short distance therefrom. This is for the double purpose of avoiding refluxing to the still and also of minimizing opportunity for contact of the distillation vapors with liquid condensate formed therefrom. In other words, the object is to get the distillation vapors out of and at least a short distance away from the still before material condensation occurs, and then to effect condensation of the desired liquid products at a point where no refluxing of condensate to the still takes place, and under conditions such that the condensate and residual uncondensed vapors and gases are rapidly separated from each other so that absorption or solution of such vapors and gases by the condensate is practically prevented.

This object may be attained in various specific ways, but the arrangement here illustrated is a typical practical way of effectively accomplishing the result desired. In the installation here shown, the upper part of the still is covered with heavy heat insulation or lagging **23**. Furthermore, each of the vapor offtakes **12**, is similarly insulated not only over portion **12a**, but also over its downwardly inclined portion down to the point where the riser **16** branches off. Thus there is substantially no condensation in any of the offtakes **12** until that point is reached. In the present instance, the riser **16** is also shown insulated and the header **17** may also be insulated. But the advisability in any given instance of insulating said risers and header is determined largely by the prevailing atmospheric temperature. Similarly the vapor lines **18**, **19**, may or may not be insulated from where they leave header **17** up to the point where they bend downward again, but in the present example they are uncovered and therefore air-cooled. The extent to which these parts, **16**—**19**, of the piping system are insulated may of course be adjusted as re-

quired by seasonal temperature conditions. By such adjustment the vapors and gases in the ascending lines are on the one hand cooled sufficiently to cause nearly all vapors of desired liquid products to condense, the proportion of such vapors escaping condensation and passing over through the deodorizer system being thus minimized; while on the other hand the cooling in said ascending lines is not carried so far that the deleterious vaporous or gaseous impurities are condensed or their absorption by the useful condensate favored.

The remainder of each offtake 12, beyond the junction of riser 16 therewith, is freely exposed to air-cooling, because in this part of the system collects the desired liquid condensate and proper cooling thereof is advantageous.

Returning now to the header 15, liquid collecting in this header is discharged through run-down line 24 to one of the run-down or receiving tanks indicated at 25, 26, 27 and 28, there being four such receiving tanks in the battery here illustrated although of course the number may be greater or less as practical operating conditions require. Said receiving tanks are connected, respectively, by valved branches 25^a, 26^a, 27^a and 28^a, with the aforesaid line 24. A look-box 29 is placed in a vertical portion of said line.

The header 22 into which the header-distillate lines 18 and 19 discharge is connected by line 30 with header-distillate receiving tank 31 which, for reasons that will appear hereinafter, may be relatively small as compared to the main receiving tanks. A look-box 32 is provided in a vertical portion of line 30, and it is also convenient to provide a sampling device 33 which is connected by valved pipe 34 with line 30, and is also provided with a valved bleeder inlet 35 and a valved draw-off pipe 36.

The entire distilling system is maintained under the desired degree or degrees of vacuum by means of a vacuum pump equipment indicated conventionally at 37, this equipment being either a single vacuum pump of suitable size or a double or triple stage pumping equipment, as may be found most desirable in any given case. The intake of the vacuum pump system has a valved connection 38 to the main vacuum line 39—40 in which header-distillate receiving tank 31 is placed. The four main receiving tanks are connected to said line 39 by valved pipes 41, 42, 43 and 44, for a purpose that will appear hereinafter. Said pipes extend somewhat above vacuum line 39, as indicated at 45, before entering the same, thus providing against accidental entry from said vacuum line into any of the main receiving tanks, when the valves may be open, of liquid impurities that may collect in said vacuum line.

All seams and joints throughout the apparatus system are made absolutely tight, most desirably by arc-welding, thus making it possible to operate at extremely low absolute pressures and to maintain non-oxidizing conditions during the distillation. Mercury manometers for observing the prevailing absolute pressure are provided at different points in the system as indicated, for example at 10^b for the still, at 25^b, 26^b, 27^b, 28^b, for the main receivers, and at 31^b for the header-distillate receiver 31.

In order to illustrate further the principles of the invention, a detailed description will now be given of one practical procedure that may be used in the manufacture of lubricating oils in accordance with the new process from Nevada County (Arkansas) crude, employing the above described apparatus system in conducting the distillation. A typical Nevada County crude has a gravity of about 14° Baumé as a rule, and ordinarily contains 3% of sulfur or thereabouts. This crude contains practically no gasoline, naphtha or kerosene, the first runnings being gas oil of around 29° Baumé gravity.

If the crude contains more than one-half of one per cent moisture, as it sometimes does, it should first be dehydrated to reduce the moisture content to that proportion or less. This dehydration may be accomplished in various ways known in the art and therefore the operation need not be described here. The crude, sufficiently free from moisture, is charged into the still 10 up to about 80% of the cubic capacity thereof and the still is gradually heated up to within 10° F. or so of the temperature at which the crude has been found by preliminary test to boil initially under 5 mm. mercury absolute pressure. This temperature is usually about 350° F. with the usual run of Nevada County crude. This temperature having been attained, the distilling system is then placed under vacuum by operation of the vacuum pump system indicated at 37 which, as already explained, may comprise as many single or multiple stage dry vacuum pumps as are found desirable to use in practice. This vacuum is applied to the still, main receivers and other parts of the system through the line 39 and, by way of header-distillate receiver 31, through the header-distillate or deodorizer lines 18, 19. At the outset, the valves connecting the main receiving tanks with run-down line 24, that is, valves 251, 261, 271, and 281, are all open. So also are valves 301 and 401. Valve 441 in pipe 44, and also valves 411, 421 and 431, are closed. When the absolute pressure in the still has been reduced to about 10 mm. absolute, as measured by the manometer 10^b, the initial distillate of gas oil begins to come over. At this stage, valves 261, 271, and 281 may be closed, if desired, assuming that the gas oil is to be collected in receiver 25. By reference to the indicating manometers, it will be found that the pressure in the still 10 and the header-distillate receiving tank 31 is substantially the same, while the pressure in the receiving tank 25 is slightly higher. In practice this difference in pressure should usually amount to some four or five millimeters during the first part of the gas oil cut in order that a relatively large quantity of hot oil vapors may be immediately pulled over into the deodorizer system to warm the latter up promptly and get it to functioning properly. Later on, the pressure difference becomes less until by the end of the gas oil cut it may have decreased to only about one millimeter. It will be found that a considerable quantity of liquid header-distillate collects in the receiving tank 31, this distillate being characterized by very foul odor and a strong bluish phosphorescent cast. It is characteristic of this header-distillate that if only an extremely minute quantity thereof is added to a good lubricating oil it will adversely affect the "cast" of the oil and render the oil unstable. This distillate collecting in receiver 31 while the gas oil cut is being taken off consists itself largely of light gas oil fractions containing a very large proportion of the total quantity of the objectionable malodorous and unstabilizing constituents contained originally in the crude, the removal and separation of which is one of the principal objects of the invention. The liquid caught in receiver 31, therefore, comprises rela-

tively low-boiling constituents which escape condensation in the risers 16, header 17 and the ascending portions of header-distillate lines 18, 19, and are rapidly drawn off or sucked away from the heavier constituents which are condensed in that portion of the system and which flow thence through cooling coils 13, header 15 and run-down line 24 to the receiver tank 25. This separation of the lighter contaminating impurities from the desired liquid condensate is materially favored by the before-mentioned pressure differential which tends to cause flow through the deodorizer lines 18, 19, in preference to flow directly to the receiving tank 25 through the cooling coils and run-down line. This tendency of course affects not only the uncondensed vapors and gases in said lines 18, 19, but also to some extent the liquid condensate formed therein and in the risers 16. But in the case of said condensate this tendency is almost entirely overcome by the action of gravity so that notwithstanding the slightly higher suction or vacuum in the header-distillate lines, comparatively little liquid condensate is carried over as such through said lines provided the height of the overhead piping system 16, 17, 18, is properly adjusted and the lagging or heat insulation is also properly adjusted. This of course is governed to a large extent by the prevailing atmospheric temperature and varies with different seasons of the year. In practice, it is only necessary to have the piping system 16, 17, 18, extend high enough from main vapor line 12, to ensure adequate condensation at the maximum seasonal temperatures to be encountered, and then to increase the amount of lagging as may be necessary to compensate for decrease of temperature in winter. When the system is operating, the risers 16, headers 17, and ascending portions of lines 18, 19, should be quite hot, so that during the first part of the gas oil cut sonsiderable gas oil passes over into the header-distillate receiver 31; and at all times these ascending lines should be so warm that the condensate formed therein is not cooled far below its condensing temperature. In this way the tendency for malodorous vapors and gases to be re-absorbed or dissolved by condensate flowing down the inner walls of the headers to the line 12 is minimized. It will therefore be seen that in the arrangement and mode of operation described, the hot still vapors and gases pass upwardly in countercurrent contact with downwardly flowing lubricating oil condensate, the hot condensate leaving the base of risers 16 relatively free from volatile contaminants.

It may be noted here that when the apparatus is functioning properly or to best advantage, a fog is distinctly discernible in the look-box 32 in line 30 leading to the receiver 31; while no fog is discernible in look-box 29 in the run-down line 24. This condition exists when the absolute pressure in the still 10, and deodorizer system, including header-distillate receiver 31, is somewhat lower than the pressure in receiver 25, 26, or 27, whichever one of these receivers may be in use at the time, and such pressure differential can readily be maintained by properly setting the control valves in the connections to the vacuum pump so that the pump pull is wholly or mainly through the deodorizer system.

Thus far the discussion has dealt only with the initial or gas oil cut. The distillation of gas oil continues until a test sample of condensate taken from run-down line 24 through sampling device 241 (similar to sampling device 33) shows a flash point which has been selected as proper to determine the conclusion of the gas oil cut. For example, this flash point may be selected as 290° F. in a typical instance. At this point the still temperature is ordinarily about 450° F. as indicated by a pyrometer P having its thermocouple located about one inch above the bottom of the still. This marks the beginning of the lubricating distillate cut. In the meantime the operator has "cracked" or slightly opened valve 421 controlling line 42 which connects receiver 26 with vacuum line 39, this being done before valve 261 is opened. In this way the pressure in receiver 26 can be reduced to that existing in receiver 25 at the end of the gas oil cut, say 6 millimeters for example. This having been done, the operator now closes valve 421, opens valve 261, and closes valve 251, whereupon the stream of distillate flows into receiver 26. By this time it is no longer necessary to maintain as large a pressure differential as 4 or 5 millimeters because by far the greater part of the objectionable impurities have now been pulled over through the deodorizer system during the initial distillation which is intentionally forced somewhat, as by rapid firing, in order to bring about an immediate heavy rush of vapors through the deodorizer system, as described. Therefore after this initial rapid rush of vapors has been continued until a couple of hundred gallons of header distillate has collected in tank 31, the system may then be operated with a much smaller pressure differential, say one millimeter or thereabouts. To this end the proper control valve 411, 421, or 431, as the case may be, may be opened very slightly, if necessary.

The cut collected in receiving tank 26 may, if desired, be the entire quantity of lubricants that can be distilled from the particular crude undergoing treatment up to the temperature where substantial decomposition or cracking begins to take place in the still. However, it is usually more convenient to divide this lubricant yield into two or more portions or cuts, and in the present instance it will be assumed that the lighter portion, corresponding to light and medium motor oil, is collected in receiving tank 26 and a heavier portion, corresponding to heavy motor oil, is collected in receiver 27. Thus, when the gas has been taken off as described and the succeeding lubricating distillate has been cut into tank 26, the distillation may be continued at an absolute pressure which has by this time been reduced usually to around 4 to 6 millimeters of mercury in the still and which may decrease gradually to as low as 1.5 to 3 millimeters toward the end of the distillation. After the gas oil has all been distilled off, it is found that the proportion of distillate that continues to come over through the deodorizer lines 18, 19 and collect in receiver 31 is relatively very small indeed, quite commonly amounting to as little as one-half of one per cent by volume of the crude initially charged into the still for the entire remainder of the distillation, and being substantially less foul than the header distillate obtained during the gas-oil cut. This later part of the header-distillate may therefore be collected separately and re-run if desired. On the other hand, the quantity of header-distillate obtained during the gas oil cut amounts frequently to as much as 5 per cent of the initial charge of crude. In this connection it must of course be borne in mind that this 5 per cent is large'y gas oil which, in association

with the small percentage of deleterious impurities to be eliminated, is carried over through the deodorizer lines because of the rapid rate of distillation and heavy pump pull on said lines at the beginning of the distillation, as above described. Until the still temperature as indicated by pyrometer P reaches about 630° F., the vacuum control valves are adjusted as may be necessary to maintain the before mentioned foggy appearance in the header-distillate line look-box 32, thus ensuring always a slightly lower pressure in said line and in the still than in the particular receiver into which the distillate stream may be flowing.

In a typical instance, the lubricating distillate is run into tank 26 until the flash point of a test sample is about 380° F., at which stage the still temperature approximates 550° F.; but it is to be understood that the selection of the point at which to cut the distillate into tank 27 may be varied as plant operating conditions require.

The lubricating oil distillate having been cut into receiver 27, the distillation continues as described with usually some further lowering of the absolute pressure, down to say 1.5 to 3 millimeters as observed in the still, which means a slightly higher pressure (e. g. 2.5 to 4 mm.) in receiver 27 as indicated by the manometer 27b.

The distillation is continued, running the lubricating cut into receiver 27 until tests of the distillate indicate that the point has been reached where decomposition or cracking is taking place or is about to take place in the still. The approach of this point is indicated when the flash point of successive test samples becomes stationary instead of gradually increasing as was the case during the earlier stages of the distillation. After remaining practically stationary for some time, the flash point of test samples will be found to be gradually lowering instead of increasing, and at this point it is best practice to cut the distillate into receiving tank 28. The still temperature is now approximately 630° F., and since cracking has now begun, it is desirable to complete the distillation as rapidly as possible. Accordingly valve 441 is now opened wide and the full pull of the pump is exerted through the large pipe connection 44 and receiver 28 directly upon the run down line 24, a procedure which immediately causes a substantial increase in the size of the stream coming through line 24. Hardly any distillate other than normally fixed gases comes over through the deodorizer line from now on.

The distillation can be continued up to whatever maximum distillation temperature is found in practice to be commercially advantageous, notwithstanding the fact that the lubricating fraction thus collected in receiver 28 contains cracked products and is therefore not as high in grade as the oil collected in receivers 26 and 27.

The residue remaining in the still 10 after the distillation has been run as described, is asphaltic in character and may be either a hard or soft asphalt, or a flux oil, depending upon the extent to which the distillation is carried. All of these products are valuable commercially, and the particular form in which it is desirable to obtain the residue in any given instance may to a considerable extent be determined by market conditions existing at the time.

The gas oil fraction collected in receiver 25 may be considered to be a finished product and disposed of as such.

The lubricating oil cuts in receivers 26 and 27 are good lubricating oil products but still contain a small proportion of organic acids which should be removed in order to render the products perfectly sweet, clean, and stable. Accordingly, these cuts are most desirably re-run in a manner to be described presently.

The heavy lubricating distillate contained in receiver 28 also contains organic acids but in addition is further characterized by an appreciable content of cracked products resulting from decomposition of the oil in the still at temperatures above about 630° F. This heavy lubricating cut is therefore also re-run to obtain finished products.

The re-running of the several cuts contained in receivers 26, 27 and 28 may be conducted in the same manner in each instance if these cuts are re-run separately. It is not essential, however, that they be separately re-run, but on the contrary the entire yield of lubricants obtained in the first distillation may be either collected or mixed together and re-run as a whole. There are certain marked advantages, however, in re-running the several cuts separately, and this method is therefore to be recommended in practice. A description of how any one of these cuts is re-run will suffice for all.

In order to eliminate organic acids and traces of other impurities that may be contained in the cut to be re-run, the oil is now treated with a neutralizing agent having the power to react with the organic acids and with such other impurities as may be present in such manner as to enable substantially complete elimination and separation thereof from the oil in the re-run distillation. Various neutralizing agents may be employed for this purpose, but a caustic alkali, such as caustic soda, is effective and convenient to use and is therefore preferred. Examples of other basic reagents that may be employed, however, are soda ash, lime, and the like. In using caustic soda as a neutralizing agent, this may be employed in a water solution of say 30° to 40° Baumé strength. Assuming a 40° Baumé solution, it is found sufficient to employ about a pint of the solution for each barrel of lubricating oil distillate to be re-run. The exact amount may vary somewhat depending upon the nature of the starting material. Such a proportion of the solution having been added to the charge of oil, the charge is then thoroughly agitated, as by blowing in air, or otherwise, until the neutralizing agent has been uniformly distributed through the charge. The charge is then re-run in apparatus which may be of exactly the same character as that shown in Figs. 1 and 2 and hereinabove described in detail. The re-run distillation may be carried out in precisely the same manner as the first distillation except that the particular cuts of lubricating oils made may be different from or more numerous than those of the first distillation, it being convenient ordinarily to so determine the cuts as to obtain a range of finished oils corresponding generally to commercial types of oils demanded by the trade.

In thus re-running the lubricating oil distillate initially collected in receiver 26, a certain further quantity of non-viscous distillate is obtained of which the viscosity ranges from 60 to 90 seconds at 100° F., but the proportion is relatively very small. A somewhat larger amount, approximating say 10 per cent, is also obtained in re-running the heavy cracked lubricating distillate initially collected in receiver 28. The residues remaining after re-running the distil-

lates initially collected in receivers 26, 27 and 28 are accumulated, and when the accumulation is sufficient it is also re-run in the manner already described and approximately 90 per cent of the accumulation is recovered as valuable lubricants. The final residue from such re-run of accumulated residues may be disposed of as fuel oil or otherwise as may be desired.

The following table shows in summarized form the results, in a typical instance, of distilling a Nevada County, Arkansas, crude of 14° Baumé gravity and 3 per cent sulfur content in accordance with the principles of the present invention. The test methods used in obtaining the data given in the table are those set forth in U. S. Government Standard Specification 2C (Technical Paper 323A, Department of the Interior, 1924).

Product	Gas oil	100 pale	200 pale	300 pale	500 pale	750 pale	1,000 pale	1,300 pale
Percent of crude	24.23	9.70	8.38	5.65	4.19	5.65	5.41	2.16
Gravity (Bé.)	29.0	22.0	20.7	20.2	19.9	19.3	18.9	18.7
Flash (° F.)	------	335	375	385	415	435	460	480
Fire (° F.)	------	390	445	450	500	530	565	590
Viscosity at 100° F.	------	105	221	310	508	740	1076	1356
Color N.P.A.	------	1.75	2.0	2.25	2.5	3.0	3.5 to 4.0	4.0 to 4.5
Coldtest (° F.)	------	−35	−20	−10	−5	0	5	5
Conradson carbon residue value	------	.01	.02	.03	.04	.07	.09	.10
Demulsibility at 130° F.	------	1620	1620	1620	1580	1500	1500	1200
R. E. test [1]	------	1	1	2	2+	3	6	7

[1] Resistance of an oil to emulsification, as specified by A. S. T. M

Recapitulation—Total yield

	Per cent
Gas oil	24.23
Finished lubricating oil	41.14
Fuel oil	0.64
Asphalt	33.68
	99.69

The oils of which identifying data are given in the foregoing table are typical of novel lubricating oils that can be produced by the process of the invention. They are all narrow-cut lubricating oils, that is, oils characterized by narrow viscosity range and narrow boiling point range, and obtained by overhead distillation, all as set forth in this applicant's prior Patent No. 1,448,709 aforesaid, but they are sharply distinguishable from the oils disclosed in said prior patent by the fact that, for any given viscosity, the flash and fire points are, respectively, at least about 10° F. higher and, in the case of the higher viscosities, as much as 40° and 50° F. higher, than the flash and fire points of correspondingly viscous oils of said prior patent. Furthermore, the new oils are either odorless or clean-smelling, and are stable, as already pointed out.

In the foregoing table, the captions "100 pale", "200 pale", etc. are trade designations based upon the approximate viscosity and general color classification of the particular product so designated. Thus, "200 pale" identifies a lubricating oil having a viscosity of approximately 200 seconds at 100° F. as measured on the Saybolt universal viscosimeter. While the general color classification of this 200 viscosity product is given in the table as "pale", an oil having an N. P. A. color of 2.0 would actually be designated in the present custom of the trade as a "Filtered pale" oil, and in fact this trade designation would apply to all oils having an N. P. A. color not greater than 4.5. However, as it is not necessary to filter oils produced by the present process to attain such high grade color, they are referred to in the above table merely as "pale" in order not to convey the impression that they have been filtered.

The series of lubricating oils whose identifying characteristics and properties are set out in the above table are new products in the art and are clearly distinguishable from lubricating oils heretofore known. Moreover, the particular series of oils above given is merely typical of novel lubricating oils that can be manufactured in accordance with the invention. The N. P. A. color, the cold test, and the Conradson carbon residue percentage, characterizing the new oils, are in each instance closely similar to the corresponding identifying characteristics of the lubricating oils produced in accordance with the process disclosed in the Schulze Patent 1,448,709 before mentioned. However, the present oils are distinguishable from the oils disclosed in the aforesaid Schulze patent by the fact that the flash and fire points are invariably considerably higher, always at least 5° F. higher, and ordinarily from 10° to 40° higher. This superiority in flash and fire points is the more marked the higher is the viscosity of the oils compared.

While the dimensions of the various parts of the apparatus system herein disclosed may of course be varied widely in practice within the scope of the invention, the dimensions and capacities of the more important parts of one form of apparatus installation that has proved effective and satisfactory in practice will be given by way of illustration but not at all by way of limitation. In this typical installation, horizontal cylindrical still 10 is nine feet in diameter and thirty feet long, giving an approximate charging capacity of about 250 barrels of 42 gallons each. Each of the ten offtake lines 12 has an internal diameter of ten inches, and the vertical portion 12a thereof rises to a height of about eighteen inches above the still, it being desirable to have this height as small as conveniently possible. Each of the cooling coils 13, into which the offtakes 12 discharge liquid, is a three-inch pipe; and the header 15 into which the ten cooling coils 13 discharge is ten inches in diameter, as is also the run-down line 24 leading to the horizontal cylindrical receivers 25, 26, 27 and 28, each of which latter is in this instance seven feet in diameter by sixteen feet long.

Each of the ten risers 16 is six inches in diameter, and extends upward ten feet to enter the header 17, which is eight inches in diameter. The five deodorizer lines 18, 19, are of four-inch pipe and extend ten feet higher. The cooling coils 20, 21, into which the lines 18, 19, discharge, are six-inch pipes; and the header 22 is ten inches in diameter as is also the line 30 leading therefrom to the small receiver 31 which is four feet in diameter and sixteen feet long. The vacuum line 39, 40, is ten inches in diameter as is also the pipe 44 leading therefrom to run-down tank 28. The pipes 41, 42 and 43 connecting tanks 25, 26 and 27 to vacuum line 39 are four inches in diameter; while the pipes 25a, 26a, 27a and 28a, which connect said tanks with run-down line 24 are six inches in diameter.

In general, it is important that the piping connections, both vapor and liquid lines, between the still and the vacuum-producing means, shall

be of such ample capacity as to favor free and substantially unobstructed pull of the vacuum-producing means upon the still at all stages and under all conditions of normal operation. It is also important that the vacuum-producing means be able at all times to exert a direct and free pull through the cooling coils and associated piping upon the receiving tanks; but it is also desirable that the arrangement shall be such that the pull of the vacuum-producing means upon the still can be maintained slightly greater than the pull of said means upon the receivers, thus creating a pronounced tendency for the distillate from the still to flow toward the vacuum pump by way of risers and overhead lines 16—18 as already described and for the purposes set forth. This object can be achieved in various different ways, but in the specific arrangement and design of the apparatus herein disclosed it is achieved at least in part by reason of the fact that in pulling upon the receiving tanks 25—28, the action of the vacuum-producing means is to some extent affected by the liquid in the cooling coils 13 which, although by no means filling said coils in normal operation, probably exerts a slight throttling action.

The process of the invention may also be carried out with the aid of steam, and in some classes of work the employment of steam as an auxiliary distilling agent is of advantage. Fig. 3 illustrates how the apparatus system may be arranged where the distillation is to be carried on with the aid of steam, most desirably superheated, introduced into the charge through valved pipe S. In this arrangement, the overhead vapor lines 18' lead to a cold condenser indicated generally at 14', there being most desirably a separate condenser coil 20' for each of the lines 18'. The water in condenser tank 14' should be maintained substantially cooler than that in tank 14. In the coils 20', both steam and oil vapors condense and are then discharged into header 22', from which the liquid products are conveyed by line 30' to a receiver 31'. While only one receiver 31' is here shown, it is sometimes desirable in practice to employ two such receivers into which the liquid products may be directed alternately in order that the condensate may be more readily removed from time to time without disturbing operation of the system as a whole. It is also sometimes desirable to operate with two or more such receivers 31' in series. Whether one or more of these tanks 31' be employed, it or they should be kept cold, but not below the freezing point of water, by suitable cooling means such, for example, as cooling coils C through which a cooling medium of any suitable character may be continuously passed.

From the receiver or receivers 31', the vacuum line 39, 40, extends to the vacuum pump 37 as before described, the system as a whole being similar, except in the particulars above noted, to that illustrated in Figs. 1 and 2.

In the specific example hereinabove given, illustrating how the method of the invention may be practiced with the aid of the apparatus shown in Fig. 1, the manufacturing procedure involves two distillations, namely, a first distillation of a crude without the addition thereto of a neutralizing agent but with separation of a large proportion of the malodorous and other impurities as described; and a second or re-run distillation of the lubricating distillate or distillates in the same manner but with the addition of a neutralizing agent, such as caustic alkali, to the oil that is to be so re-run. However, it is not to be inferred that the invention is restricted to a procedure involving two or more successive distillations. On the contrary it is feasible and in some cases advantageous to add a neutralizing agent directly in the first instance to the crude or residuum used as a starting material and to distill over finished lubricating oil products directly without the necessity of re-running. However, where the crude or residuum employed as starting material is an asphaltic base material, it is ordinarily desirable not to add a neutralizing agent directly thereto because this would decrease the value for many purposes of the asphalt residue left in the still at the end of the distillation. By using a two-stage procedure of the character hereinabove described, this difficulty is avoided, an asphaltic residue free from alkali being obtained from the first distillation, while the presence of alkali in the final residuum resulting from the last of the re-run distillations is not objectionable since this residuum is not asphaltic and is useful only as a fuel oil. Where the crude or residuum employed as starting material is non-asphaltic in character and yields a distillation residue in which the presence of alkali is unobjectionable, or where it is feasible commercially to dispose of an asphaltic residuum containing alkaline matter, the foregoing considerations are not so important, and it is therefore practicable in some cases to manufacture finished products direct by a single distillation from the crude starting material.

Nor is it to be inferred from the specific description hereinabove given that the process of the invention is restricted to batch or intermittent distillation. A method involving batch distillation has been described hereinabove merely for purposes of illustration and it is entirely feasible to practice the new process in a continuous manner, employing a battery of stills through which the oil passes successively, each still yielding a distillate having characteristics determined by the conditions under which that still is being operated.

Where the starting material is a crude oil containing a rather large proportion of gas oil, say 25 to 30 per cent, such as Smackover crude for example, or where the crude contains a considerable proportion of other relatively light fractions such as gasoline, naphtha and kerosene, it is feasible to top the crude in an ordinary pipe still or steam still down to a point where the residue has an initial boiling point of around 300° to 350° F. at 5 to 10 millimeters absolute pressure. This of course involves the use of temperatures in the topping plant of 575° to 600° F. or thereabouts, operating under atmospheric pressure.

The process of the invention is also applicable to the treatment of paraffine base crudes and residua, but in treating such starting materials it is necessary to guard against clogging of the vapor lines, cooling coils, etc. with congealed wax. This can be readily done as by steam-jacketing the apparatus parts in question and also maintaining the receiving tanks warm so that the condensates collecting therein can be removed and suitably treated for separation of contained wax by centrifuging or other appropriate method.

In practicing the process, conducting the distillation, whether in one stage or more than one stage, under strictly non-oxidizing conditions is a matter of vital importance for attainment of satisfactory results. Every precaution

should be taken to ensure absolute tightness of the apparatus by welding all joints and seams as already stated. Continual leakage of even a relatively very minute quantity of air into the system can seriously affect the proper operation of the process and result in production of inferior products. It is also important to avoid as far as possible refluxing of distillate to the still since this results in discoloration and other damage to the lubricating products by cracking or otherwise prior to attaining still temperatures around 630° F.

It is feasible to use other types of oil-vaporizers than the horizontal cylindrical still type herein disclosed by way of illustration. Thus, for example, pipe stills operating in conjunction with appropriate dephlegmators or separating drums of construction suitable to withstand the use of extremely low absolute pressures, can be used to advantage in some cases.

It is found in practice that in the described rerunning or redistillation of the lubricants obtained in the first distillation after the gas oil has been taken off and before cracking temperatures have been reached (i. e. the lubricants initially collected in receivers **26** and **27**, in the specific illustrative example above given), those of the resultant finished lubricating oils that have a viscosity of 300 seconds Saybolt (at 100° F.) and higher are tasteless as well as odorless. Notwithstanding the fact that they are direct distillates, not treated with acid or filtered, they are as tasteless and odorless as the best grades of so-called white medicinal oils now on the market. Nevertheless they show color as indicated in the table hereinabove given. Heretofore no tasteless mineral oils have been known that were not water-white in color, and invariably they have been acid-treated and upon redistillation, whether at ordinary pressures or very low absolute pressures, they yield decomposition products. Tasteless mineral oils having appreciable color are novel products in the art.

The process of the invention may also be employed for production of finished lubricating oils from partially finished lubricating oils made by other processes. Furthermore even finished lubricating oils resulting from such other processes may be re-distilled in accordance with the principles of this invention to produce lubricating oils of much higher grade. The use as starting material in the present process of such finished or partially finished oils produced by other processes is an important practical phase of the present invention.

It is to be especially noted that the present invention makes possible the manufacture of good finished lubricating oils as direct overhead distillates from any crude, residuum, or other heavy mineral oil material, no matter how high may be its content of sulfur, organic acids or other contaminating impurities, and that no acid treatment, filtration, or other additional finishing treatment is required to produce high grade lubricants of the best color, greatest stability and other desirable characteristics.

What I claim is:

1. The process of producing lubricating oil distillates which comprises mixing a basic neutralizing agent with a body of mineral oil containing lubricating oil components and also lighter and lower-boiling odor-producing components, heating said body of mineral oil to vaporizing temperature and thereby obtaining lubricating oil vapors in mixture with malodorous gases and relatively light condensable vapors, conducting the resultant hot vapors and gases into a zone wherein they are caused to pass upwardly in countercurrent contact with downwardly flowing lubricating oil condensate formed at a higher point in said zone, so regulating the temperature conditions in said zone as to ensure sufficient cooling of the vapor to condense therefrom substantially all the desired lubricating components thereof but, at the same time, to avoid further cooling to a temperature so much below the vaporizing point of said condensate as to permit condensation of said relatively light malodorous vapors or substantial absorption of said malodorous vapors and gases by said condensate, rapidly conducting the uncondensed vapors and gases away from said zone, separately conducting the hot lubricating oil condensate away from said zone and out of contact with said uncondensed vapors and gases, and then, after the condensate has left said zone, permitting it to cool further, all the stated operations being conducted under non-oxidizing conditions and under low absolute pressure.

2. In the manufacture of useful mineral lubricating oil products from mineral oil containing difficultly separable but relatively low-boiling impurities, the process which comprises distilling a mineral oil in the presence of a basic neutralizing agent under an absolute pressure not exceeding 25 millimeters of mercury and under non-oxidizing conditions, conducting the distillation vapors upward and cooling the same under such pressure to a temperature not substantially below the vaporizing point of relatively heavy lubricating components thereof at such pressure while rapidly withdrawing uncondensed lighter vapors, and still further cooling the resultant condensate out of contact with said uncondensed vapors.

3. In the manufacture of lubricating oils, the process which comprises mixing a small proportion of a basic reagent with mineral oil comprising lubricants associated with relatively light impurities, distilling said oil under an absolute pressure not exceeding about 25 millimeters of mercury and under non-oxidizing conditions to obtain mixed vapors therefrom while avoiding substantial refluxing to the body of mineral oil being distilled, conducting the mixed vapors rapidly into a cooling zone also maintained at an absolute pressure not exceeding about 25 millimeters of mercury and at a temperature sufficiently low to produce a lubricating oil condensate but not much lower than the condensing temperature, whereby to prevent extensive condensation of said relatively light impurities or absorption thereof by said condensate and separately conducting uncondensed impurities and said lubricating oil condensate away from said zone under suction, the suction being maintained higher upon said impurities than upon said condensate.

4. The process as set forth in claim 3, further characterized by the fact that the pressure at which the lubricating fractions are distilled and condensed does not exceed 15 millimeters absolute.

5. The process as set forth in claim 3, further characterized by the fact that the pressure at which the heaviest lubricating fractions are distilled and condensed does not substantially exceed 5 millimeters.

6. In the manufacture of lubricating oils from mineral oil material containing lubricating constituents together with associated impurities of lower vaporizing point that are largely inseparable therefrom by usual distillation methods and that undesirably affect the odor, color and sta-

bility of finished lubricating oils containing the same, the process which comprises vaporizing lubricating fractions from such mineral oil material in a still under strictly non-oxidizing conditions and at an absolute pressure not exceeding 25 millimeters of mercury, in the presence of an added basic neutralizing agent, conducting the resultant distillation vapors and gases into a zone, maintained under absolute pressure not exceeding 25 millimeters of mercury, wherein they are caused to pass upwardly in countercurrent contact with downwardly flowing lubricating oil condensate formed at a higher point in said zone, so regulating the temperature conditions in said zone as to ensure sufficient cooling of the vapor mixture to condense therefrom substantially all the desired lubricating oil components thereof but, at the same time, to avoid further cooling to a temperature so much below the vaporizing point of said condensate as to permit condensation of vaporous impurities or substantial absorption of either vaporous or gaseous impurities by said condensate, sucking away uncondensed vapors and gases out of contact with the hot lubricating condensate under an absolute pressure at least as low as that at which vaporization was effected in the distillation, and separately conducting away from said zone and cooling said lubricating condensate.

7. In the manufacture of lubricating oils, the process set forth in claim 6, further characterized by the fact that the pressure under which vaporization and condensation of the relatively heavy lubricating fractions is effected at no time exceeds 10 millimeters of mercury absolute and does not substantially exceed 5 millimeters absolute during a substantial part of the distillation.

8. In the manufacture of lubricating oils, the process set forth in claim 6, further characterized by the fact that the pressure under which vaporization and condensation of the relatively heavy lubricating fractions is effected at no time exceeds 10 millimeters of mercury absolute and does not substantially exceed 5 millimeters absolute during a substantial part of the distillation, and by the fact that the difference between the absolute pressures under which the uncondensed vapors and the condensate are respectively conducted away is of the order of magnitude of a few millimeters of mercury.

9. In the manufacture of lubricating oils from mineral oil material containing lubricating constituents together with associated impurities of lower vaporizing point that are largely inseparable therefrom by usual distillation methods and that undesirably affect the odor, color and stability of finished lubricating oils containing the same, the process which comprises adding a basic neutralizing agent to such mineral oil material and vaporizing lubricating fractions therefrom in a still under strictly non-oxidizing conditions and under absolute pressure not exceeding 15 millimeters of mercury, while avoiding substantial refluxing of condensate to the still, rapidly conducting away resultant distillation vapors and gases, without substantial cooling, to a point beyond where refluxing to the still can occur, then cooling said vapors sufficiently to condense the greater part of the lubricating constituents thereof while avoiding cooling substantially further than is necessary to effect this result, and sucking away uncondensed vapors and gases out of contact with the hot lubricating condensate under an absolute pressure at least as low as that at which vaporization was effected in the distillation, and separately conducting away and cooling said lubricating condensate under a somewhat higher absolute pressure.

10. In the manufacture of lubricating oils from mineral oil material containing lubricating constituents together with associated impurities of lower vaporizing point that are largely inseparable therefrom by usual distillation methods and that undesirably affect the odor, color and stability of finished lubricating oil containing the same, the process which comprises vaporizing lubricating fractions from such mineral oil material in a still under strictly non-oxidizing conditions and at an absolute pressure not exceeding 25 millimeters of mercury while avoiding substantial refluxing of condensate to the still, rapidly conducting away resultant distillation vapors and gases, without substantial cooling, to a point beyond where refluxing to the still can occur, then cooling said vapors sufficiently to condense the greater part of the lubricating constituents thereof while avoiding cooling substantially further than is necessary to effect this result, sucking condensate and uncondensed vapors and gases away from each other through separate conduit systems, the suction applied to said uncondensed vapors and gases being greater than that applied to said condensate, and further cooling said uncondensed vapors and gases to obtain therefrom a lower boiling condensate, the process further including the addition of a basic neutralizing agent to the mineral oil material from which the lubricating constituents are to be vaporized.

11. In the manufacture of lubricating oils, the process set forth in claim 6, further characterized by the fact that the pressure under which vaporization of the lubricating fractions is effected at no time exceeds 10 millimeters of mercury absolute and does not substantially exceed 5 millimeters absolute during a substantial part of the distillation, and by the fact that the difference between the absolute pressures under which the uncondensed vapors and the condensate are respectively conducted away is of the order of magnitude of a few millimeters of mercury.

12. In the manufacture of lubricating oils from mineral oil material containing lubricating constituents together with associated impurities of lower vaporizing point that are largely inseparable therefrom by usual distillation methods and that undesirably affect the odor, color and stability of finished lubricating oils containing the same, the process which comprises adding a basic neutralizing agent to such mineral oil material and vaporizing lubricating fractions therefrom in a still under strictly non-oxidizing conditions and at an absolute pressure not exceeding 10 millimeters of mercury and not substantially exceeding 5 millimeters during a substantial part of the distillation, while avoiding substantial refluxing of condensate to the still, rapidly conducting away resultant distillation vapors and gases, without substantial cooling, to a point beyond where refluxing to the still can occur, then cooling said vapors sufficiently to condense the greater part of the lubricating constituents thereof while avoiding cooling substantially further than is necessary to effect this result, sucking condensate and uncondensed vapors and gases away from each other through separate conduit systems, the suction applied to said uncondensed vapors and gases being greater than that applied to said condensate, and further cooling said uncondensed vapors and gases to obtain therefrom a lower boiling condensate.

13. In the manufacture of lubricating oils, the process as set forth in claim 12, further characterized by the fact that the difference in pressure under which the uncondensed vapors and the condensate are respectively conducted away is of the order of magnitude of a few millimeters of mercury.

14. In the manufacture of lubricating oils, the process which comprises distilling lubricating fractions from a crude oil or residuum at absolute pressures not exceeding 25 millimeters of mercury, and below 10 millimeters for the major part of the distillation, and, redistilling the resultant lubricating oil in the presence of a basic neutralizing agent at absolute pressures of similar magnitude.

15. In the manufacture of lubricating oils, the process set forth in claim 14, further characterized by the fact that the crude oil or residuum employed has an asphaltic base and contains difficultly separable relatively low boiling contaminants injurious to finished lubricating oil.

16. In the manufacture of lubricating oils, the process of improving previously manufactured lubricating oils which comprises subjecting such a manufactured lubricating oil to distillation in the presence of a basic neutralizing agent at absolute pressures not exceeding 25 millimeters of mercury and most desirably substantially below 10 millimeters, and collecting directly a lubricating oil distillate of better stability than such previously manufactured oil.

17. The process of manufacturing high-grade finished lubricating oils without acid treatment or filtration which comprises vaporizing, under absolute pressure not exceeding 25 millimeters of mercury and under non-oxidizing conditions, a lubricant-containing mineral oil crude or residuum high in combined sulphur, nitrogen, organic acids, or complexes including some or all of such constituents, thereby producing lubricating oil vapors mixed with vaporous and gaseous malodorous impurities which are lower-boiling than the lubricating oil components, lowering the temperature of the mixture sufficiently to condense most of the lubricating oil components therefrom, then separately and rapidly conducting away out of contact with each other the resultant lubricating oil condensate and the uncondensed vapors and gases, said lubricating condensate being maintained under an absolute pressure not substantially exceeding 10 millimeters of mercury while flowing out of contact with the aforesaid uncondensed vapors and gases, and further cooling and collecting said condensate, under like low absolute pressure as a high grade finished lubricating oil requiring no acid treatment or filtration to render it fit for immediate use.

18. In the manufacture of lubricating oils from mineral oil materials containing, or yielding upon distillation, relatively volatile malodorous constituents difficultly separable from a desired lubricating oil product, the process which comprises heating such mineral oil material and obtaining therefrom lubricating oil vapors in association with relatively volatile malodorous constituents, leading such mixture of vapors and gases away through a plurality of large-diameter conduits arranged in multiple, cooling the mixture sufficiently to obtain therefrom a lubricating oil condensate while rapidly sucking away gases and uncondensed vapors, causing the condensate to flow, still warm but out of contact with said gases and uncondensed vapors, in a plurality of streams through large-diameter conduits arranged in multiple, and applying to said condensate while so flowing a high vacuum corresponding to an absolute pressure of between 1.5 and 25 millimeters of mercury.

19. The process of manufacturing lubricating oils as overhead distillates which comprises distilling mineral oil which contains lubricating oil components in association with odor-producing components in the presence of a basic neutralizing agent under absolute pressure not exceeding 25 millimeters of mercury and under non-oxidizing conditions, thereby producing a mixture of lubricating oil vapors with difficultly separable vaporous and gaseous lower-boiling malodorous contaminants, conducting the mixed distillation vapors upwardly while cooling the same under such pressure sufficiently to condense desired lubricating oil components therefrom while avoiding cooling to the condensing point of the associated lower-boiling malodorous contaminants and rapidly withdrawing uncondensed vapors and gases out of contact with the resultant lubricating oil condensate, then still further cooling said condensate under such pressure out of contact with said uncondensed vapors and gases.

20. The process as claimed in claim 19, wherein the stated operations are all effected under an absolute pressure not exceeding 15 millimeters.

21. The process as claimed in claim 19, wherein the stated operations are all effected under an absolute pressure which does not exceed 15 millimeters and is for the most part below 10 millimeters.

22. The process of manufacturing high-grade finished lubricating oils without acid treatment or filtration, which comprises vaporizing, under absolute pressure not exceeding 25 millimeters of mercury and under non-oxidizing conditions, a lubricant-containing mineral oil crude or residuum high in combined sulphur, nitrogen, organic acids, or complexes including some or all of such constituents, thereby producing lubricating oil vapors mixed with vaporous and gaseous malodorous impurities which are lower-boiling than the lubricating oil components, subjecting such mixed vapors in a traveling stream to reflux condensation under absolute pressure not exceeding 25 millimeters and at a temperature above the condensing temperature of said impurities but sufficiently low to condense lubricating oil components, rapidly sucking away uncondensed vapors and gases, and flowing the resultant lubricating oil condensate out of contact with said traveling stream of mixed vapors while cooling said condensate still under absolute pressure not exceeding 25 millimeters, and collecting the same as a high grade finished lubricating oil requiring no acid treatment or filtration to render it fit for immediate use.

23. The process as claimed in claim 22, wherein the stated operations are all effected under an absolute pressure not exceeding 15 millimeters.

24. The process as claimed in claim 22, wherein the stated operations are all effected under an absolute pressure which does not exceed 15 millimeters and is for the most part below 10 millimeters.

25. The process of manufacturing lubricating oils as overhead distillates which comprises distilling mineral oil which contains lubricating oil components in association with odor-producing components in the presence of a basic neutralizing agent under absolute pressure not exceeding 25 millimeters of mercury and under non-oxi-

dizing conditions, thereby producing a mixture of lubricating oil vapors with difficultly separable vaporous and gaseous lower-boiling malodorous contaminants, conducting said mixture into a condensing zone and cooling the mixture under such pressure sufficiently to condense desired lubricating components therefrom while rapidly withdrawing uncondensed vapors and gases from said zone, separately conducting the resultant lubricating condensate away from said condensing zone and further cooling the same under such pressure and out of contact with said uncondensed vapors and gases.

26. The process as claimed in claim 25, wherein a small proportion of caustic soda is employed as the neutralizing agent, and the stated operations are all effected under an absolute pressure which does not substantially exceed 15 millimeters and is for the most part below 10 millimeters.

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