April 4, 1933. A. E. PEW, JR 1,903,407

PROCESS FOR PREPARING CRUDE OIL FOR DISTILLATION INTO LUBRICATING OILS

Filed Feb. 28, 1928 2 Sheets-Sheet 1

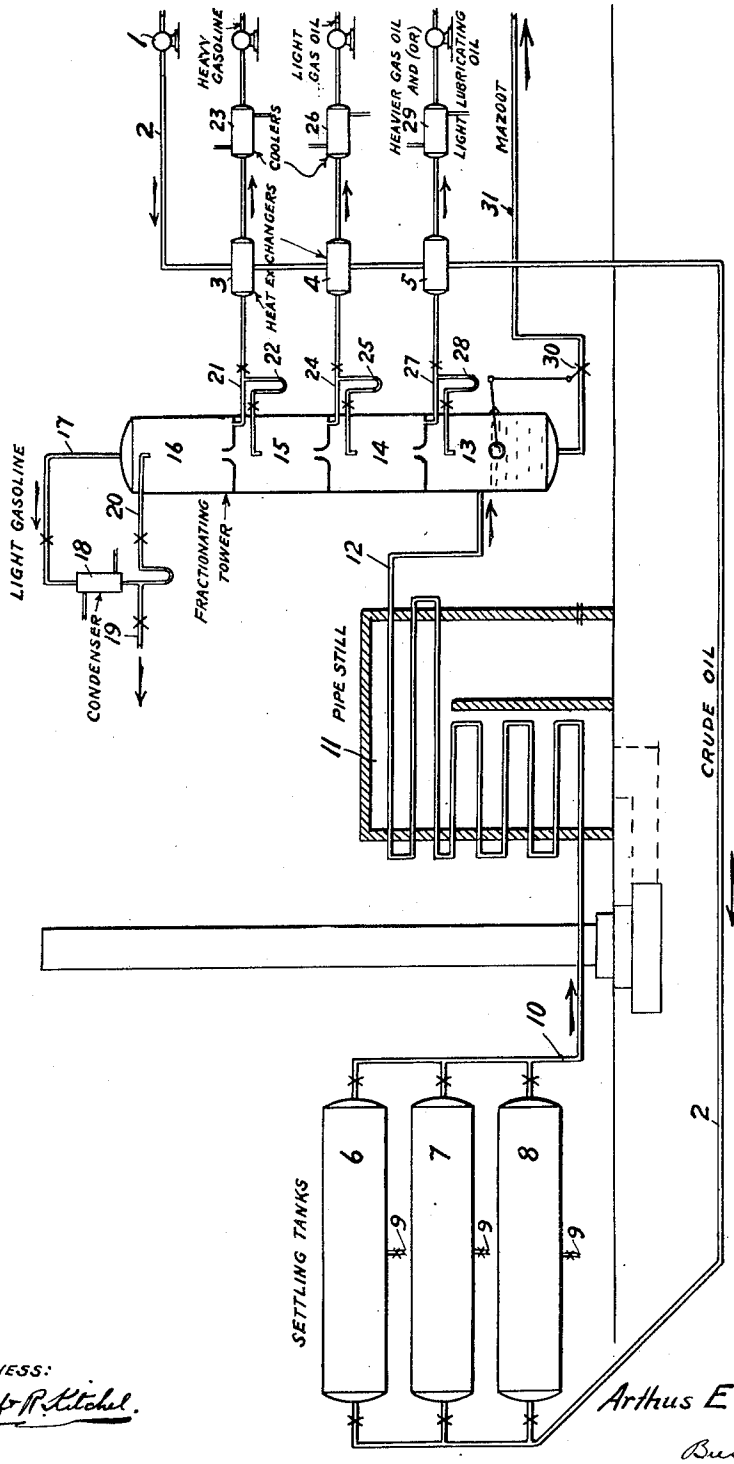


FIG. 1A.

WITNESS:
R. B. R. Kitchel.

INVENTOR
Arthus E. Pew, Jr.
BY
Bussey and Harding
ATTORNEYS.

April 4, 1933. A. E. PEW, JR 1,903,407

PROCESS FOR PREPARING CRUDE OIL FOR DISTILLATION INTO LUBRICATING OILS

Filed Feb. 28, 1928 2 Sheets-Sheet 2

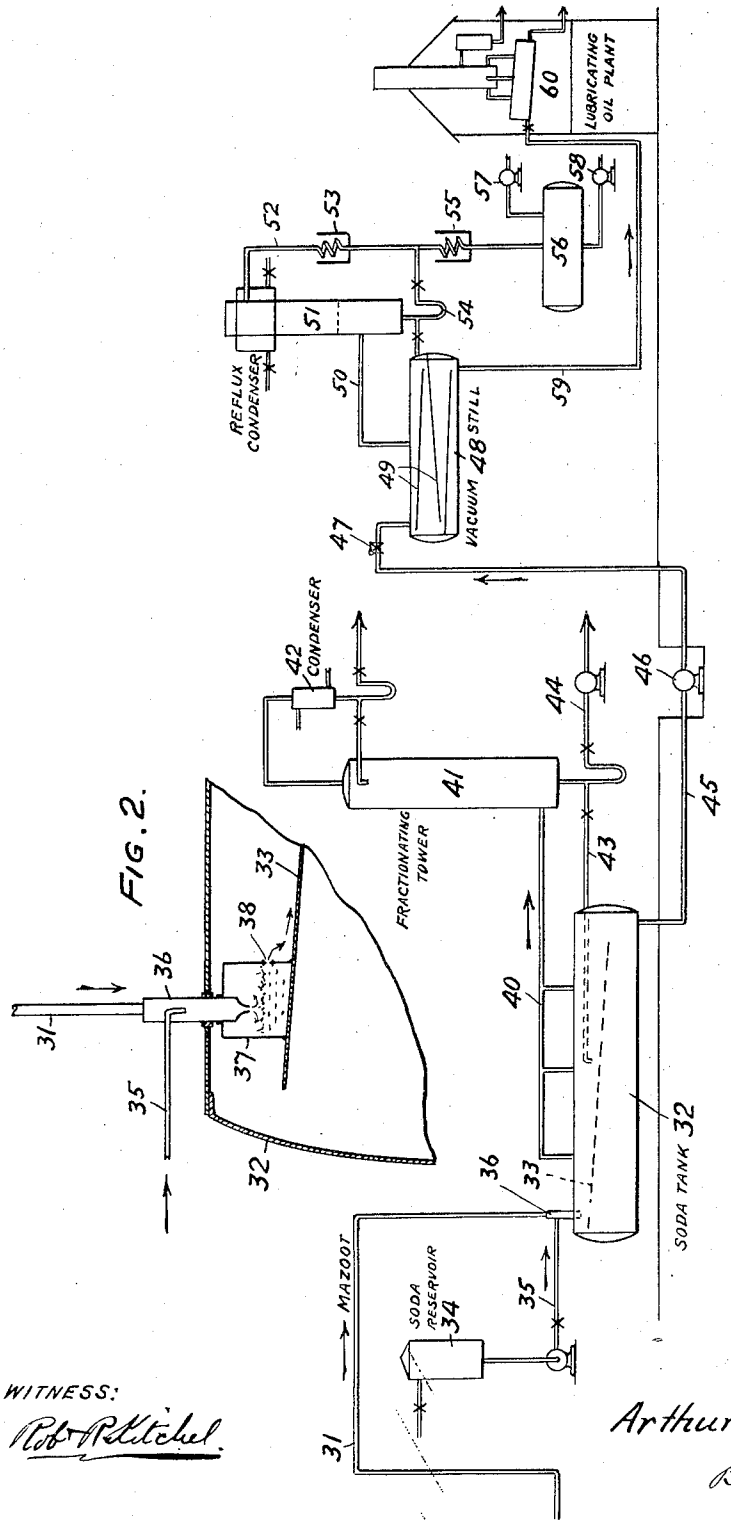


FIG. 2.

FIG. 1B.

WITNESS:
Robt. P. Kitchel.

INVENTOR
Arthur E. Pew, Jr.
BY
Busser and Harding
ATTORNEYS.

Patented Apr. 4, 1933 1,903,407

UNITED STATES PATENT OFFICE

ARTHUR E. PEW, JR., OF BRYN MAWR, PENNSYLVANIA, ASSIGNOR TO SUN OIL COMPANY, OF PHILADELPHIA, PENNSYLVANIA, A CORPORATION OF NEW JERSEY

PROCESS FOR PREPARING CRUDE OIL FOR DISTILLATION INTO LUBRICATING OILS

Application filed February 28, 1928. Serial No. 257,559.

In the manufacture of lubricating oils, I have found that, in order to obtain lubricating distillates free from products of cracking and oxidation, it is necessary not only to distil such oils from lubricating stock under conditions that will not produce such products but also to avoid the production of such products in the operations of "topping the crude" to distil off the lighter fractions, such as gasoline and gas oil.

A process for preparing lubricating stock from crude oil that accomplishes this result is set forth in applications filed by me December 22, 1926, Serial No. 156,287, and January 8, 1927, Serial No. 159,754, said applications having now respectively matured as Patents No. 1,833,691, November 24, 1931, and No. 1,761,153, June 3, 1930.

The object of the present invention is to improve and simplify the process set forth in my said applications.

The process is not restricted to execution in any specific apparatus, but the apparatus illustrated in the accompanying drawings has been found especially well adapted to the practice of the process.

Figs. 1A and 1B comprise a diagrammatic view of this apparatus.

Fig. 2 is a vertical sectional detail of a device for feeding caustic soda to the oil stock.

Crude oil is pumped, by pump 1, through line 2, including heat exchangers 3, 4 and 5, to be hereinafter described, and is thereby heated to a suitable temperature, say from 250°–350° F. The oil thence flows into a settling tank, which is maintained under a pressure not less than that which corresponds to the vapor pressure of the oil and water at the desired temperature. A preferred temperature of 305° F. gives an absolute pressure of 72.7 pounds. In addition to this, it is preferred to carry a pressure of 8 or 10 pounds, or a total pressure of (say) 80 pounds. In this tank water and sediment settle and are drawn off through an outlet 9 at the bottom. It is desirable that there should be a substantially continuous outflow from this separator of dehydrated oil. This is made possible, or facilitated, by the provision of a plurality of tanks 6, 7 and 8, preferably arranged in parallel. If these tanks are arranged in parallel as shown, the oil from line 2 may be delivered to and withdrawn from the tanks in endless rotation, in such manner that while oil is being delivered to one tank, it is settling in a second tank and is being withdrawn from a third tank. It is preferred that the oil, before settling, should be heated in the heat exchangers 3, 4 and 5, or by any other source of heat, to a temperature sufficient to reduce its viscosity to not above 35 seconds Saybolt, say to a temperature approximating 300° F. and that, while settling, it should be subjected to a superatmospheric pressure (say of five atmospheres) sufficient to prevent vaporization of water and light oil fractions. This settling process is more fully set forth in an application filed by me June 14, 1927, Ser. No. 198,763.

The dehydrated oil flows through line 10 into one or more pipe stills 11, wherein the oil is heated and the desired fractions vaporized. Preferably but a single pipe still is used, since, if all the fractions intended to be distilled at this stage are vaporized in a single still, the temperature required will be lower than if successively higher oiling fractions are vaporized in successive stills, with consequent reduced danger of serious cracking. The temperature in still 11 should be below that at which substantial cracking occurs, and need not be as high as 600° F. The temperature may be still further reduced by maintaining a comparatively low vacuum in and beyond the still, although a vacuum is unnecessary at this stage of the process.

A mixture of oil and oil vapor leaves the still 11 through line 12 and enters a tower near its bottom. The tower is divided into four sections 13, 14, 15 and 16. The lower section 13 acts as a vapor separator. The vapors freed in this section flow up through the sections 14, 15 and 16 successively. Vapors that are uncondensed in such passage comprise mainly or wholly gasoline and flow out through a top vapor line 17 into a water-cooled or oil-cooled condenser 18. The condensate from the condenser passes, in reg-

ulable proportions, partly, through line 19, to storage and partly, through line 20, into the top of the tower. The reflux going back through line 20 causes fractional condensation of heavy gasoline in the top section 16. This heavy gasoline outflows through line 21, part of it flowing to storage through heat exchanger 3, where it transfers heat to the incoming crude, and the remainder passing, through line 22, to the upper part of section 15, wherein it effects condensation of light or medium gas oil. This gas oil outflows through line 24, part of it flowing to storage through heat exchanger 4 and further heating the incoming crude, and the remainder passing, through line 25, to the upper part of section 14, wherein it effects condensation of heavier gas oil or light lubricating stock or both. The latter outflows through line 27, part of it flowing to storage through heat exchanger 5 and still further heating the incoming crude and the remainder passing through line 28, to the upper part of the lower section 13. The progressively higher boiling point distillates flowing out through lines 21, 24 and 27 go through coolers, 23, 26, and 29 on their way to storage. It will be understood that it is impossible to distill off gas oil without distilling off a small percentage of light lubricating oil.

The number of fractions into which the vapor entering the tower is condensed is, of course, a matter of selection. It will also be understood that the proportion of each distillate that is led back to the tower is capable of regulation by appropriate valves. My invention is principally concerned with obtaining, by means of any suitable distilling apparatus, a residue, or mazoot, consisting of crude oil from which the light ends have been removed. This mazoot is continuously withdrawn, through line 31, from the bottom of section 13, which is equipped with a float valve 30 that maintains a predetermined depth of oil in the tank. The term 'mazoot' is used in the sense in which it is ordinarily used by oil producers, namely, to define a crude oil which has been topped to have a high flash with respect to its viscosity, thus indicating careful fractionation.

In my operation of the described apparatus, I so control the temperature of distillation and the operation of the tower that all fractions are removed that have a flash point below about 235° F. In other words, the mazoot, upon distillation, yields a first ten per cent. fraction having a flash not below 235° F. It will be understood, however, that this explanation of the character of the mazoot is merely illustrative and that it may be varied within rather wide limits. Thus, if, in the lubricating plant 60 (Fig. 1B) for which I desire to prepare lubricating stock, it is desired to distil off the lightest lubricating oils, the described distillation will be so conducted as to produce a mazoot which, upon distillation, will yield a first ten per cent. fraction having a flash substantially below 235° F.

The mazoot flowing through line 31 enters a tank or still 32, which I shall conveniently refer to as a soda tank. The oil flows down over and along one or more pans 33 in the soda tank. At the same time, a solution of caustic soda, or other neutralizing material, is admitted to the tank and mixes with the entering oil.

In Fig. 2 is shown an efficient device for continuously supplying soda to, and mixing it with, the entering oil. A soda solution from a supply vessel 34 is pumped through a small pipe 35 and discharges into a short pipe 36, of larger diameter extending through, and welded to, the still shell. Pipe 36 is secured to the end of, or constitutes an extension of, the oil pipe line 31. The discharge end of pipe 36 is provided with a swedged nipple and opens into a box 37, which is welded to pan 33 and has a comparatively tight joint around pipe 36. Box 37 is provided, in its front side, with a slit 38 located at some distance above pan 33.

The mixture of oil and soda solutions is forcibly discharged from pipe 36 into the box 37 and the mixture that at any given time is contained in the box is kept in a state of great turbulence. The described arrangement not only insures a thorough mixing of the oil and soda solution but prevents the mixture from splashing around inside the tank and insures a steady delivery of the mixture through slot 38 onto the pan 33.

Inasmuch as the temperature of the oil will usually be above 500° F. and may be as high as 575° F. or even somewhat higher, it is possible to maintain the soda tank at any convenient pressure, including atmospheric pressure, and still insure the quick evaporation of all the water introduced with the soda as the mixture flows down the pan 33.

The water vapor and with it a slight amount of oil vapor flows out through line 40 to a fractionating tower 41. The vapors pass out the top of tower 41 into a conventional condenser 42, part of the condensate going to storage and the remainder being put back into the tower as a reflux. If it should be desired to return the oil fractions, or any part thereof, condensed in tower 41 to the soda tank, this may be done through line 43; or they, or part of them, may be conveyed through line 44 to storage.

The mazoot, with the partially dehydrated soda in solution or suspension therein, passes out through line 45 and is pumped, by pump 46, through a back-pressure valve 47, into a tank 48, where the oil is flowed over vaporizing pans 49. In this tank there are vaporized the lighter fractions that it is desired

to exclude from the stock that is to go to the lubricating plant.

The vaporization of these fractions is customarily effected at a temperature sufficiently high to produce a substantial amount of cracking and thereby produce a residue from which it is impossible to distil the highest grade lubricating oil even though the distillation of the lubricating stock may be conducted under non-cracking conditions. In my process, however, the tank or still 48 is maintained under a vacuum sufficiently high to reduce the boiling point of the fractions to be vaporized to the extent required to avoid cracking. It is not essential that any substantial amount of heat should be applied to effect this vaporization. Indeed, it is preferred to apply no heat. If, for examp'e, the still 48 is maintained under a vacuum of (say) 28 inches, then, due to the reduction in pressure from atmospheric pressure or thereabouts, enough specific heat is converted into latent heat to enable all the fractions which it is desired to eliminate (which may be heavy gas oil or light lubricating fractions or both) to be vaporized.

Due to the great velocities at which vapors form and leave the surface of the oil flowing over the pans 49, some entrainment of heavier oil may occur. The reflux condenser 51, into which the vapors from the still pass through line 50, permits, by temperature control, the lighter products to pass out through line 52 and throws the heavier products down into trap 54, from which, should the entrainment be particularly objectionable, the heavier products flow back into the tank 48; or, should the entrainment be unobjectionable, such heavier products may be led to a cooler 55, from which they may be led off separately; or, as shown in the drawings, they may mix with the overhead distil'ate flowing through line 52 and cooler 53, the mixed condensates flowing thence into a receiving tank 56. A vacuum is maintained in tank 56 by means of a vacuum equipment 57. The oil is pumped from tank 56 by means of a pump 58.

The residual oil leaving tank 48 passes through line 59 to a lubricating oil distillation apparatus 60 which may be of any efficient character. A preferred lubricating oil distillation apparatus is set forth in my said app'ication filed January 28, 1927, Ser. No. 159,754, as well as in an earlier application filed May 29, 1926, Ser. No. 112,485.

It will be noted that at no stage of the described process is the oil raised to a substantial cracking temperature, and that air is excluded throughout the entire process. There can thus be formed neither those decomposition products that are formed, by cracking, when oil is exposed to high temperatures for a substantial length of time, nor those oxidation products that are formed at a lower temperature when distillation occurs under conditions that do not exclude the presence of air. The fractionations are efficient, temperature and pressure conditions are carefully and accurately controlled, and the process may be so conducted that the residual oil passing to the lubricating oil distillation plant 60 will contain no fractions except those higher boiling fractions whose distillation may be most economically effected in such plant.

Variations in the process, some of which do and some of which do not, involve slight modifications in the apparatus or connections, may be practiced.

Thus, soda tank 32 and tower 41 may be placed under vacuum in order to evaporate a substantial amount of lighter oil vapor in addition to the smaller amount that, operating as atmospheric pressure, would come off with the water vapor. Indeed, by subjecting tank 32 to a sufficiently high vacuum, it is possible to dispense with the still 48 and instead drive off, in still 32, all fractions that it is not desired to feed to the lubricating plant 60, or stills 32 and 48 may be both utilized by operating them under progressively higher vacuums.

It is also practicable to operate the pipe still 11 and the tower associated therewith at such temperatures and under such conditions that the oil in line 31 will contain those, or some of those, fractions that, in the preferred mode of operation described, are distilled off in section 13, or in section 13 and one or more other sections of the vaporizer, and to distill off such fractions, or such fractions and still heavier fractions, in still 48, or in still 32 alone by operating it under a sufficiently high vacuum, or in stills 32 and 48 by operating them under progressively higher vacuums.

Also, it is practicable to operate the pipe still 11 and the tower associated therewith at such temperatures and under such conditions that the oil in line 27 will be that fraction of the crude that consists of or contains the lubricating cuts that it is desired to treat in stills 32 and 48. Thus the oil in line 27 may be that fraction of the crude that contains the lubricating oil cuts up to and including 750 @ 100 viscosity. These cuts may, in whole or in part, be distilled off in still 48, or in still 32 alone by operating it under vacuum, or in stills 32 and 48 by operating them under progressively higher vacuums. In this case, the residual oil in line 31 may be utilized as fuel oil.

While the process is primarily intended to produce a residual oil that is adapted to be distilled for the production of those lubricating distillates which may be most economically produced in the special lubricating plant 60, including particularly those high boiling distillates which it is impracticable,

even with the application of heat, to vaporize in stills 32 and 48, it will be understood that the demand for high boiling lubricating distillates may, at a given time, be so limited relatively to the demand for low and medium boiling lubricating distillates, that, if the plant is so operated that none of such low and medium boiling distillates remain in the residual oil going to line 59, such residual oil may be utilized as fuel oil.

Having now fully described my invention, what I claim and desire to protect by Letters Patent is:—

1. The process of preparing stock for distillation of lubricating oils, which comprises evaporating off, from a crude oil, low boiling constituents at a temperature below that required to produce substantial cracking and fractionating the product to remove therefrom lighter ends and separate out heated oil, containing the desired lubricating fractions, at a temperature substantially above the boiling point of water at atmospheric pressure, mixing with said heated oil while in liquid phase an alkali solution and subjecting the mixture of oil and alkali solution while distributed over a larger area to such absolute pressure as to effect, at the temperature of the oil, the evaporation of solvent of the alkali, and subjecting the thus treated liquid oil, while distributed over a large area, without the application of substantial heat, to a high vacuum equivalent to an absolute pressure substantially below that under which said solvent was evaporated and thereby evaporating off lighter fractions of the oil.

2. The process of preparing crude oil for the distillation of relatively high boiling fractions, which comprises forcing crude oil through an elongated confined passage while subjecting it to a temperature not sufficiently high to effect substantial cracking but sufficiently above 500° F. to effect vaporization of a substantial fraction of the crude oil, fractionating the vapors and flowing away the hot unvaporized residual oil, intimately mixing an alkali solution with said residual oil while the latter is in liquid phase and at a temperature above 500° F. and, while the mixture of oil and alkali solution is distributed over a large area, evaporating off solvent of the alkali and, while the liquid oil is subjected to a relatively high vacuum and distributed over a large area, evaporating off lighter fractions thereof, thereby providing a residual lubricating stock containing the desired higher boiling lubricating fractions.

3. The process of preparing stock for distillation of lubricating oils which comprises separating, from the crude oil, by partial evaporation and fractionation, at a temperature below that required to produce substantial cracking, a residual oil containing the lubricating fractions which it is desired to subsequently distil, mixing with such heated residual oil while in liquid phase an alkali solution and subjecting the mixture of oil and alkali solution, while distributed over a large area, to such absolute pressure as to simultaneously effect, at the temperature of the oil, the evaporation of solvent of the alkali and a lighter fraction of the oil, and leave a residual oil stock containing the desired lubricating fractions, and treating the mixed vapors to effect the separation and condensation of the oil.

4. The process of preparing stock for distillation of lubricating oils which comprises separating, from the crude oil, by partial evaporation and fractionation, at a temperature below that required to produce substantial cracking, a residual oil containing the lubricating fractions which it is desired to subsequently distil, mixing with such heated residual oil while in liquid phase an alkali solution and subjecting the mixture of oil and alkali solution, while distributed over a large area, to such absolute pressure as to effect, at the temperature of the oil, the evaporation of solvent of the alkali and a substantial proportion of the lighter oil constituents in addition to any oil that may be entrained with the evaporated solvent, treating the mixed vapors to effect the separation and condensation of the oil, and subjecting the residual oil, while distributed over a large area, to a high vacuum equivalent to an absolute pressure substantially below that under which said solvent was evaporated and thereby evaporating off higher boiling oil fraction, leaving a residual oil containing the lubricating oil fractions which it is desired to subsequently distil.

5. The process of preparing stock for distillation of lubricating oils, which comprises establishing a flowing stream of liquid oil containing the desired higher boiling lubricating fractions and lighter fractions and heated to a temperature in excess of 500° F. and establishing a flow of an alkali solution, bringing said streams together and intimately mixing them at said temperature, and, while the mixture of oil and alkali is distributed over a large area evaporating off solvent of the alkali and a substantial proportion of the lighter oil constituents in addition to any oil that may be entrained with the evaporated solvent and, while the liquid oil is distributed over a large area and subjected to a relatively high vacuum, evaporating off higher boiling fractions thereof leaving a residual oil stock containing the higher boiling lubricating fractions which it is desired to subsequently distil.

6. The process of preparing mineral oil stock for distillation of lubricating oils which comprises establishing a flowing stream of hot liquid oil containing lubricat-

ing oil fractions, adding an alkali solution to the hot liquid oil stream and then, without substantial drop of pressure or temperature, distributing the mixture over a large area and thereby effecting evaporation of solvent introduced with the alkali and leaving a residual oil containing lubricating oil fractions and alkali.

7. The process of preparing mineral oil stock for distillation of lubricating oils which comprises establishing a flowing stream of liquid oil containing the lubricating oil fractions and heated to a temperature in excess of 500° F. and establishing a flow of an alkali solution, bringing said streams together and intimately mixing them, and, while the mixture of oil and alkali is distributed over a large area at a temperature not substantially below that specified and at a pressure not substantially below that under which said mixture was effected, effecting evaporation of solvent introduced with the alkali and leaving a residual oil containing lubricating oil fractions and alkali.

In testimony of which invention, I have hereunto set my hand, at Philadelphia, Pennsylvania, on this 25th day of February, 1928.

ARTHUR E. PEW, JR.