

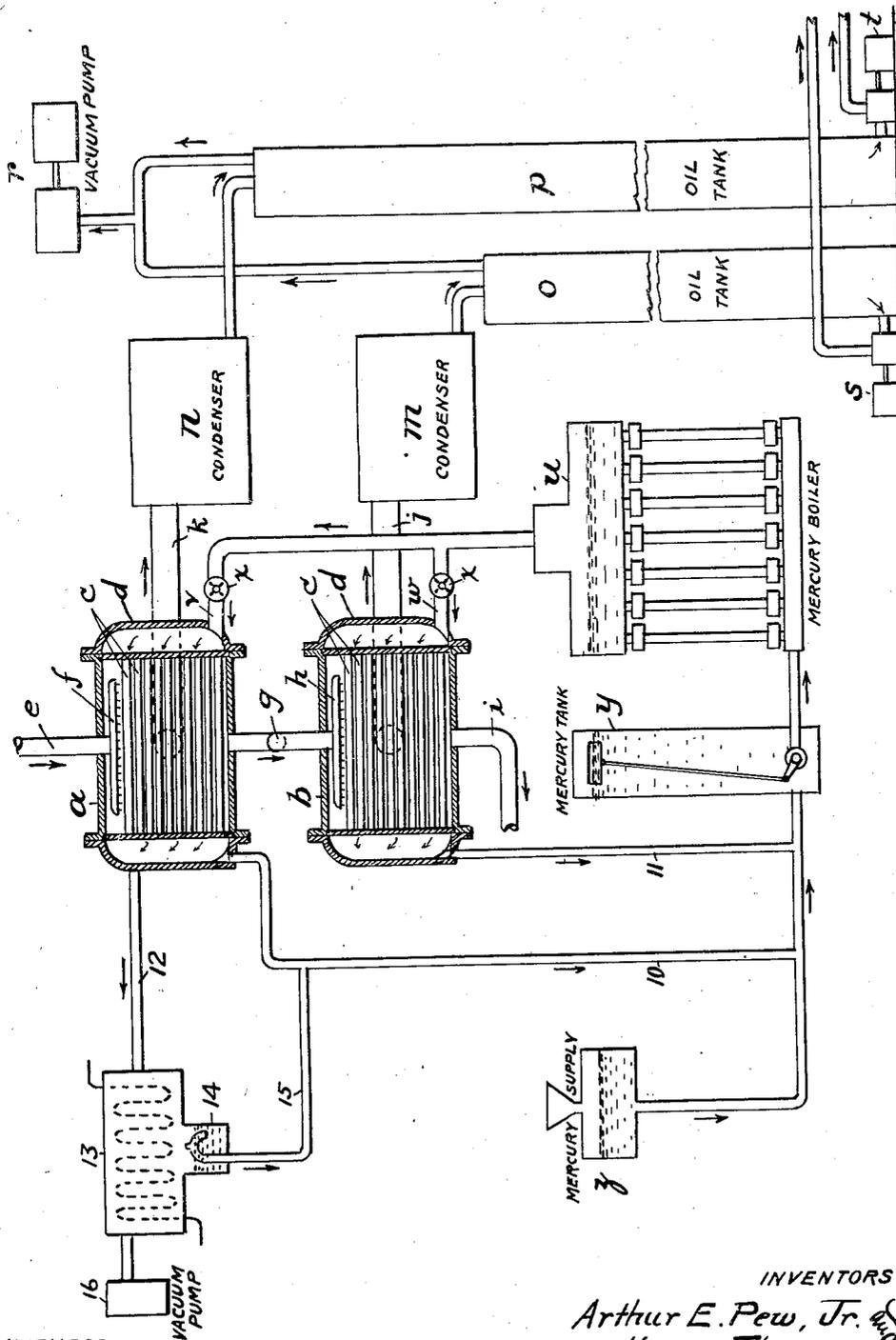
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A. E. PEW, JR., ET AL

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PROCESS OF MINERAL OIL DISTILLATION

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WITNESS:
R. H. Mitchell

INVENTORS
Arthur E. Pew, Jr. &
Henry Thomas
BY
Bussor and Harding
ATTORNEYS.

UNITED STATES PATENT OFFICE

ARTHUR E. PEW, JR., OF BRYN MAWR, AND HENRY THOMAS, OF RIDLEY PARK, PENNSYLVANIA, ASSIGNORS TO SUN OIL COMPANY, OF PHILADELPHIA, PENNSYLVANIA, A CORPORATION OF NEW JERSEY

PROCESS OF MINERAL-OIL DISTILLATION

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The object of the invention is to distill mineral oils or any distillate or residuum thereof by a process and apparatus adapted to produce the highest grades of refined products. Furthermore specific objects of the invention are to prevent or minimize decomposition and cracking, to dispense with the usual refining step of treatment with sulfuric acid and caustic soda and filtration, to effect distillation in a continuous manner, and to substantially reduce the refining cost.

In the process of distilling oil most commonly practiced, the hot gases formed by the combustion of fuel are brought into contact with a steel tube or pipe containing the oil. The heat is transferred from the hot gases to the oil and then distillation proceeds. In all such processes, there is necessarily a great difference in temperature between the hot gases and the oil, and substantial decomposition and cracking inevitably occur. In these processes, also, it is found impracticable to provide sufficient heating surfaces to absorb the heat from the flue gases, and as a result all the processes are either inefficient or unduly costly.

In accordance with the present invention, the oil to be distilled, preferably a reduced crude oil, from which it is desired to extract lubricating oils, is caused to flow down through a vaporizing chamber over heated tubes contained in the chamber; the oil spreading over the surface of the tubes in a thin layer so as to expose, to the greatest possible degree, every particle of the oil to the influence of heat and thereby effect a relatively rapid rise in its temperature; the oil flowing by gravity from tubes at a higher level to tubes at a lower level, the lighter fractions of the oil, in the course of its downward progress, being vaporized, the vapors being conducted from the vaporizing chamber and condensed. To heat the tubes, a hot fluid is caused to circulate therethrough, heat being absorbed from the circulating fluid by the downwardly flowing oil. The heating fluid is thus reduced in temperature in the course of its flow through the tubes.

In order to separately remove and condense oil vapors of substantially different

boiling points, it is preferred to employ a series of vaporizers, one above another (say three). The tubes of one vaporizer may be connected with the tubes of an adjacent vaporizer and the heating fluid may be caused to circulate first through the tubes of the lowest vaporizer and then successively through higher vaporizers, so that the heat conditions in the several vaporizers will be substantially different; the uppermost vaporizer being maintained at the lowest temperature, the lowermost vaporizer at the highest temperature and the intermediate vaporizer at an intermediate temperature. Therefore, the lightest fractions of oil will distill off in the uppermost vaporizer, and successively heavier fractions in the succeeding lower vaporizers. The number of vaporizers and the temperatures at which they are maintained are controlled by the character of the oil to be distilled and the products desired to be obtained therefrom. The several nests of tubes in the several vaporizers may, however, be arranged in multiple, instead of in series; that is, the heating fluid may be caused to flow from the boiler independently and directly to the several vaporizer chambers, any suitable means being provided to regulate the temperature of the heating fluid entering each vaporizer. It is preferred to regulate the temperature of the heating fluid by maintaining it under different absolute pressures, varying, as desired, from a high degree of vacuum to at or near atmospheric pressure or even super-atmospheric pressure. In carrying out our process, we prefer to maintain the heating fluid under a relatively high vacuum in the uppermost vaporizer and under progressively increasing absolute pressures in the succeeding vaporizers, thereby affording a convenient means for producing progressively higher temperatures in the successive vaporizers.

A serious problem arising in connection with the practice of the above distillation process, so far as it has been described, is the provision of a suitable heating fluid for circulation through the pipes. An operative heating fluid is an oil of suitable high boiling point, but a fluid of this character deposits

carbon on the inner walls of the tubes, necessitating their frequent cleaning. Any heating fluid, to be efficient and economical to the degree desired, should be one that will permit of the continuous operation of the plant. The use of mercury vapor presents distinct advantages. It has high conductivity and will not oxidize or disintegrate when heated or brought into contact with water, air or steel. Its range of temperature, when distilled under atmospheric pressure and down to a vacuum of twenty-eight inches, is approximately the same temperature range as is desirable for the distillation of the lubricating oils contained in petroleum. Its first cost is high, but bearing in mind that the cooled and condensed mercury vapor is reheated and used indefinitely, and that the loss by leakage is so small as to be an almost negligible factor, the cost of the mercury required to fill the system is essentially a part of the plant investment.

Another feature of the process is that that part of the system through which the oil and oil vapor circulate and in which the oil condenses and accumulates is maintained, preferably, under vacuum, which may be more or less partial but which, to secure the best results, should be an almost perfect vacuum, say from one to twenty-five millimeters mercury absolute pressure. That part of the system containing mercury may be maintained at different absolute pressures, as above explained, varying from over twenty-eight inches of vacuum to atmospheric pressure. We prefer that the mercury vapor in the uppermost vaporizer shall be maintained under a vacuum of about twenty-eight inches, the vacuum being progressively lower in progressively lower vaporizers. In fact, the mercury in the lowermost vaporizer may be at or near atmospheric pressure. The vaporizers carrying the higher vacuum should be located at a sufficiently higher level than those containing a lower vacuum to enable condensed mercury to flow freely to the control tank.

The construction of the apparatus and the operation of the process will be readily comprehended, with the aid of the above description, by reference to the drawing, which is a diagram of a complete distillation plant. For convenience, there are shown but two vaporizers, although it will be understood, from the foregoing description, that the number of vaporizers that will be preferably employed depends on the character of the reduced crude or other product to be distilled and on the character and variety of the products desired.

Each vaporizer *a* and *b* comprises a nest of tubes *c* and connecting headers *d*, *d*. An oil-inlet pipe *e* communicates with a distributor *f* in the top of the upper vaporizing chamber, from which the oil flows down over

the tubes *c* therein. The residue from the upper vaporizer chamber passes down through an oil seal *g* into a distributor *h* in the top of the lower vaporizer chamber, from which oil flows down over the tubes *c* therein, the residue flowing out through outlet-pipe *i*.

From the two vaporizer chambers extend vapor-outlet pipes *j* and *k* respectively to condensers *m* and *n* respectively, which communicate respectively with oil tanks *o* and *p*. By means of a vacuum pump *r*, any desired degree of vacuum may be maintained in the oil-containing part of the system. *s* and *t* are pumps for removing the distillate, from time to time, from the tanks *o* and *p*.

A mercury boiler *u* communicates with pipes *v* and *w* opening into headers of the respective vaporizers. A throttle valve, or automatic control valve, *x*, may be positioned in each of pipes *v* and *w*. A control tank *y* regulates the level of mercury in the boiler. *z* is a mercury supply tank.

10, 11 are pipes extending from headers of the respective vaporizers and through which condensed mercury is conveyed to the mercury line extending to the boiler. From a header of the upper vaporizer extends a mercury vapor outflow pipe 12 to a tank 13 containing a coil through which cold water circulates and which is provided with a catch basin 14 at the bottom to collect the condensed mercury, which flows out through a pipe 15 to pipe 10. A dry vacuum pump 16, is connected with the tank 13, thereby maintaining the desired degree of vacuum in the mercury vapor containing part of the vaporizer *a*.

It heretofore has been stated that that part of the system through which oil and oil vapors circulate should be maintained under a more or less partial vacuum, preferably equivalent to from one to twenty-five millimeters mercury absolute pressure. In ordinary processes for distilling lubricating oil, it is necessary to heat the oil to be distilled to a temperature so high that it is partly decomposed, thereby producing distillates containing decomposition products that require expensive purification treatment to remove; and their complete removal can never be effected. It is, of course, well understood that by distilling oil under a partial vacuum, the distillation temperatures of the fractions to be distilled are materially reduced, thereby enabling the distillation to be effected at lower temperatures and hence with less decomposition. It is also well understood that as an absolute vacuum is approached the boiling points drop at a rate that rapidly and progressively increases, so that, if it were possible to reasonably closely approach an absolute vacuum, the boiling points of all except the heaviest fractions could be reduced to below the temperature at which any substantial amount of decomposition occurs. In distilling the high

boiling point fractions, the highest vacuum that it is practicable, with modern engineering methods, to secure, is desirable. With a maximum practicable vacuum, it would be possible to distil crude oil practically down to coke at such relatively low temperatures as to produce abnormally heavy and viscous lubricants that would contain so small a proportion of cracked products that they could be said to be almost free therefrom and would require no subsequent expensive purification treatment.

Known high vacuum distillation processes (so-called) do not, however, afford the conditions required to avoid the formation of decomposition products, and in some, if not all, cases, do not even succeed in securing the high vacuum that they are supposed to secure. Thus, in batch distillation, the absolute pressure above the body of oil might be an absolute vacuum, and yet the oil would actually vaporize at an absolute pressure substantially in excess of the minimum degree of vacuum which it is sought to obtain; for the reason that the hydrostatic pressure toward the bottom of the column, where vapor globules begin to form, is such that the vapor as it forms is in fact subject to an absolute pressure substantially above the desired, intended and necessary absolute pressure. Not only does vaporization occur at a much higher temperature than that calculated to be necessary on the basis of the degree of vacuum above the oil body, but the vapor after it forms is subjected to such higher temperature, accompanied by the formation of such decomposition products as are produced at that temperature by cracking or otherwise.

It has also been proposed (although applicants are not advised whether the conception antedates the present invention) to run oil through a long tube in a liquid stream whose volume is much less than that of the tube, to heat the tube in the usual way by hot furnace gases, and to convey, through the tube, the vapors, formed during the passage of the oil through the tube, to a fractional condensing apparatus, whereby distillates of different cuts may be obtained. In this process, the vapors are not only forced to travel through the tube for a considerable distance in contact with the flowing oil, but such vapors, while they are in the tube, are subject to the heat of the heating medium (which, owing to the low heat conductivity of hot gases, must be several hundred degrees higher than is necessary to produce the vapors), thus subjecting the oil vapors, as well as the liquid oil, to local overheating. In fact, the conditions required to avoid local burning of the oil and cracking of oil vapors are not avoided at all, regardless of the degree of vacuum that may be obtained within the tubes. Moreover, in such process, the vapor is compelled to travel for a long distance through the tube

to the outlet thereof. It is impossible, in such an elongated stream of vapor, to avoid a substantial pressure drop from a point distant from the vapor exit to the vapor exit; and even though a condition of high vacuum may exist at the outlet end of the tube, the absolute pressure at points more or less distant from such outlet is substantially above that which it is sought to obtain and which it is necessary to obtain in order to satisfactorily distil the highest boiling point products.

By means of the present process we provide all the conditions required to successively practice a high vacuum process and have succeeded in converting the entire body of oil, except a small percentage of residual coke, into lubricants that require no subsequent expensive purification, and some of which possess characteristics heretofore wholly unknown.

The principal conditions whereby we secure the results sought may be enumerated as follows:

(1) The maintenance above the oil, while it is being heated (except in the distillation of relatively low boiling fractions) of a vacuum of not lower than substantially twenty-nine inches, that is, not lower than that corresponding to an absolute pressure of twenty-five millimeters mercury. It is preferred, however, to reduce the absolute pressure to as nearly an absolute vacuum as is practicable, particularly in distilling the higher boiling fractions. It is practicable to secure a substantially higher vacuum than that specified.

(2) The avoidance of a column of oil of any substantial height, thereby making the factor of hydrostatic pressure negligible.

(3) The maintenance of the smallest practicable temperature difference between the oil and the heating medium. This is accomplished in our process by the use, as a heating medium, of a substance having the characteristics of mercury vapor, which, because of its high heat conductivity, need be at a temperature not very greatly above the temperature of the oil to be vaporized.

(4) The application of heat, principally if not wholly, to only the liquid oil, and the avoidance of conditions whereby the vapors when formed continue to be subject to the temperature conditions required to form them; or, in other words, the maintenance of conditions under which the vapors tend to cool from the moment they leave the surface of the oil body. In the present process, the films of oil surrounding the tubes effectively prevent the establishment of direct heat exchange relations between the oil vapor and the heating medium.

(5) The avoidance of prolonged contact between the liquid oil being heated and the vapors that have escaped therefrom, which is accomplished by immediately withdrawing

them from the locus of vaporization to a locus of condensation. None of the vapors need travel for a substantial distance in contact with the film of oil on the tubes and there is no substantial pressure drop between the locus of formation of the vapors and the outlet k or j .

It should be understood, however, that the maintenance of the very high vacuum specified, while preferential in all the vaporizers of a series, is essential only in that vaporizer or those vaporizers wherein the higher or highest boiling point fractions are evaporated.

It is probable that even if the temperature of condensation of the mercury vapor in any given vaporizer be well within the cracking range, there is no cracking whatever of the oil because of the lack of direct heat exchange between the heating medium and the oil vapors and because of the extreme speed and short distance of travel of the oil vapors from the surface of the oil to the vapor outlet—a speed which is proportionate to the intensity of the vacuum, and a distance which is so short that no substantial pressure drop can be established between the vapor exit and the surface of the liquid oil. By reason of the last mentioned conditions, the factor of time (which is one of the factors of cracking) is virtually absent.

We have not herein claimed the feature of the process whereby, due to the balancing columns of mercury in the tank y and boiler u , on the one hand, and in the columns 10 and 11 on the other hand, and to the manipulation of the valves x , different pressures may be established in the boiler and in the mercury vapor chambers of the vaporizer, the same being claimed in connection with the disclosure of a much superior arrangement, proved to be commercially operative, set forth in applications filed by us May 9, 1926, which are continuations in part of this application, patents on said applications having issued May 29, 1929, No. 1,714,811 and 1,714,812.

Nor have we herein claimed broadly important features of the process of distillation of lubricating oil by means of a heating medium, such as mercury vapor; the same being claimed broadly in an application filed by us March 5, 1925, Serial No. 13,040, which is a continuation in part of the present application.

Having now fully described our invention, what we claim and desire to protect by Letters Patent is:

1. The process of distilling lubricating oil which comprises flowing a stream of oil continuously into, through and out of a confined space and while the oil is progressively traveling through said space distributing the oil over a surface adapted to be heated, generating mercury vapor from a body of liquid mer-

cury and flowing such vapor into heat exchange relation, but out of contact, with the oil that is so distributed and flowing over said surface, effecting, by heat exchange and condensation of such vapor, the vaporization of the desired oil fraction, returning condensed mercury to said body of liquid mercury and regenerating it into vapor, removing the oil vapors and condensing them, and minimizing cracking during the vaporization of the oil by maintaining in said confined space an absolute pressure on the oil of less than 25 millimeters mercury, by maintaining so thin a layer of oil on the heated surface as to substantially eliminate hydrostatic pressure and thereby cause the entire body of oil to be under the specified high vacuum, and by limiting the pressure and temperature of condensation of the mercury vapor at the locus of heat exchange.

2. The process of distilling lubricating oil which comprises flowing a stream of oil continuously into, through and out of a confined space and while the oil is progressively traveling through said space distributing the oil over a surface adapted to be heated, generating mercury vapor from a body of liquid mercury and flowing such vapor into heat exchange relation, but out of contact, with the oil that is so distributed and flowing over said surface, effecting, by heat exchange and condensation of such vapor, the vaporization of the desired oil fraction, returning condensed mercury to said body of liquid mercury and regenerating it into vapor, removing the oil vapors and condensing them, and minimizing cracking during the vaporization of the oil by maintaining in said confined space an absolute pressure on the oil of less than 25 millimeters mercury, by maintaining so thin a layer of oil on the heated surface as to substantially eliminate hydrostatic pressure and thereby cause the entire body of oil to be under the specified high vacuum, by limiting the pressure and temperature of condensation of the mercury vapor at the locus of heat exchange, and by maintaining the part of said confined space occupied by vapor out of heat exchange relation with the surface heated by the heating medium.

3. The process of distilling oil which comprises flowing a stream of oil continuously into, through and out of a series of confined spaces and while the oil is traveling through said spaces distributing the oil over surfaces adapted to be heated, generating mercury vapor from a body of liquid mercury and flowing such vapor in paths arranged in multiple into heat exchange relation, but out of contact, with the oil that is so distributed and flowing over said surface, effecting, by heat exchange and condensation of such vapor, the vaporization, in each confined space, of the desired oil fraction, returning condensed mercury to said body of liquid mercury and

regenerating in into vapor, removing the oil vapors from each confined space and condensing them, and minimizing cracking during the vaporization of the oil by maintaining in one or more of said confined spaces an absolute pressure on the oil of less than 25 millimeters mercury, by maintaining so thin a layer of oil on the heated surface as to substantially eliminate hydrostatic pressure and thereby cause the entire body of oil to be under substantially the same absolute pressure, and by regulating the pressure and temperature of condensation of the mercury vapor at each locus of heat exchange independently.

4. The process of distilling oil which comprises flowing a stream of oil continuously into, through and out of a series of confined spaces and while the oil is traveling through said spaces distributing the oil over surfaces adapted to be heated, generating vapor from a body of a liquid heating medium which is adapted to condense, at a given pressure, at the desired temperature of the oil distillation and is stable against decomposition at such temperature, flowing such vapor in paths arranged in multiple into heat exchange relation, but out of contact, with the oil that is so distributed and flowing over said surface while maintaining the vapor at the locus of heat exchange at such pressure, effecting, by heat exchange and condensation of said vapor, the vaporization, in each confined space, of the desired oil fraction, returning the condensed heating medium to said liquid body and regenerating it into vapor, removing the oil vapors from each confined space and condensing them, maintaining in one or more of said confined spaces an absolute pressure on the oil of less than 25 millimeters mercury, maintaining so thin a layer of oil on the heated surface as to substantially eliminate hydrostatic pressure and thereby cause the entire body of oil to be under substantially the same absolute pressure, and in each confined space independently regulating the pressure and temperature of condensation of the vaporous heating medium.

In testimony of which invention, we have hereunto set our hands, at Philadelphia, Penna., on this 7th day of March, 1924.

ARTHUR E. PEW, Jr.
HENRY THOMAS.