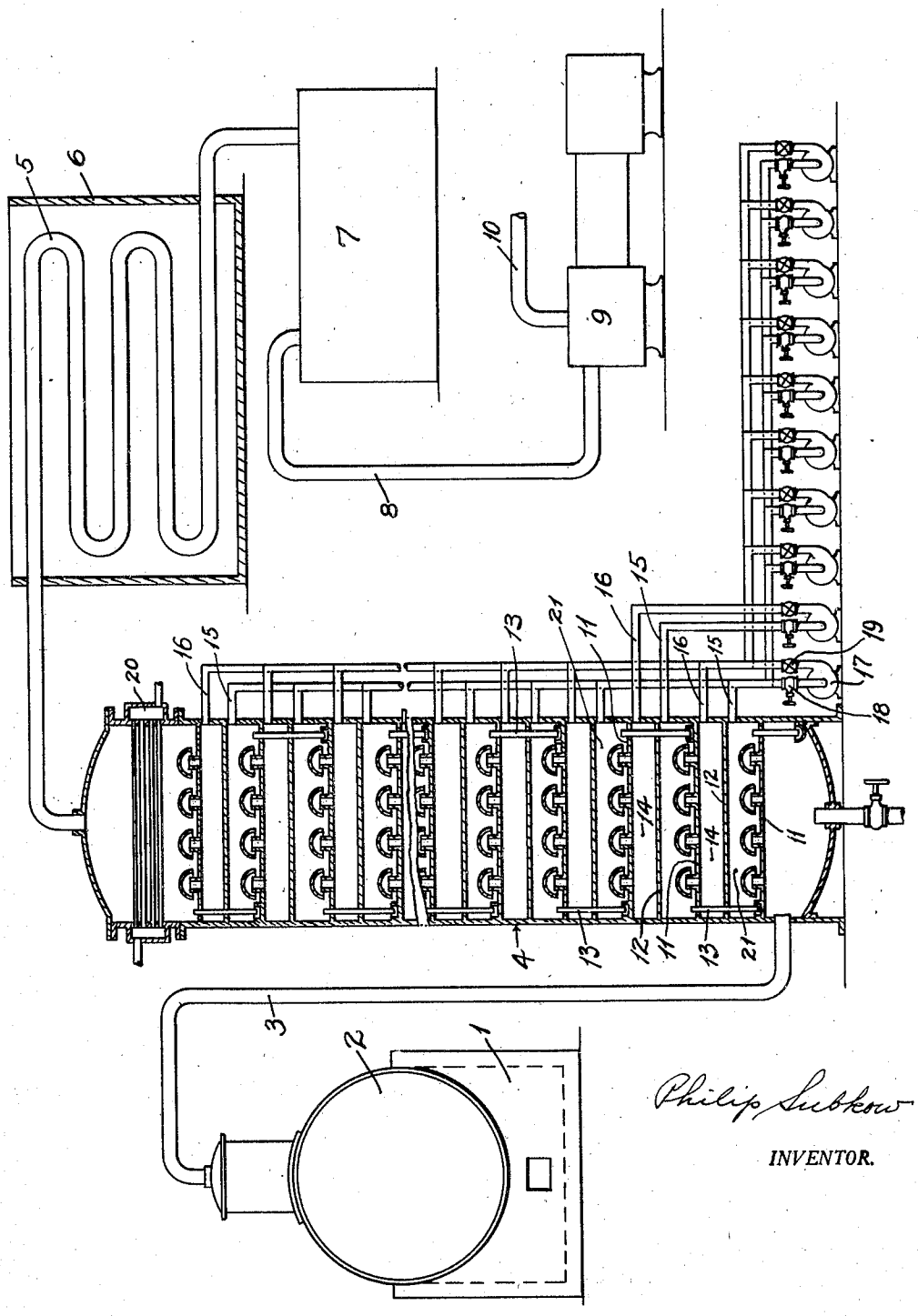
July 22, 1930. P. SUBKOW 1,771,385

PROCESS AND APPARATUS FOR VACUUM DISTILLATION

Filed Sept. 6, 1927



Philip Subkow

INVENTOR.

Patented July 22, 1930 1,771,385

UNITED STATES PATENT OFFICE

PHILIP SUBKOW, OF LOS ANGELES, CALIFORNIA, ASSIGNOR TO UNION OIL COMPANY OF CALIFORNIA, OF LOS ANGELES, CALIFORNIA, A CORPORATION OF CALIFORNIA

PROCESS AND APPARATUS FOR VACUUM DISTILLATION

Application filed September 6, 1927. Serial No. 217,791.

In vacuum distillation, the amount of vacuum which may be imposed on the vaporizing surface is determined in part by the back pressure of the vapor conduits and vapor-treating means interposed between the vaporizing surface and the vacuum generating means. The usual rectification or fractionating tower, wherein vapor is fractionated by repeated counter-current contacts with partial condensates from the vapor, usually imposes a back pressure on a vapor generating means which, in part, nullifies the efficiency of the vacuum distillation. In most fractionating columns of the "bubble" type, the back pressure per plate may be as much as 25 m. m. of mercury. It is not uncommon for a fractionating column to be from 10 to 20 plates high. While "filled" type columns show a smaller back pressure, yet the total back pressure of a column giving desired fractionating efficiencies will often be found to be such that the back pressure thus imposed upon the vaporizing surface limits, to a very material degree, the amount of vacuum maintainable thereon. It is, therefore, well-known that fractionating columns can not be employed in high vacuum distillations. Such high vacuum distillations have become of particular importance in the production of lubricating oils wherein the pressure at times may be less than 25 m. m. or range up to 200 or 300 m. m. Fractionating columns have not been found feasible at such vacuum and the only means of fractionating the vapors, at the present time, is that of fractional condensation.

It is one of the objects of this invention to devise a process and apparatus for vacuum distillation wherein the vapors are rectified by repeated counter-current contacts with partial condensates formed from said vapors; that is rectified under such conditions that this does not limit the degree of vacuum maintained on the vaporizing surface; i. e. under such conditions as would eliminate the back pressure of the rectifying process or apparatus. Generically, this process comprises generating vapors under the desired vacuum and fractionating the vapors at succeedingly higher sub-atmospheric pressure; i. e. at succeedingly lower vacuums by counter-current contact which partial condensates produce from said vapors. It is another object of this invention to rectify vapors under vacuum wherein the rectification column functions both as a fractionating unit and as a vacuum pump whereby the vapors are compressed at successive stages to lower degrees of vacuum and, at each stage of compression, contacted with a partial condensate produced in the following stages.

Further objects will be recognized by those skilled in the art from the following description taken in connection with the drawing which shows a distillation apparatus embodying the novel method of distillation and fractionation.

(1) is a furnace, (2) is a still, (3) a vapor conduit, (4) a fractionating column, (5) a condenser, (6) a condenser box, (7) a run-down tank, (8) a residual vapor line, (9) a vacuum pump, (10) a vapor exhaust pipe.

The fractionating column (4) is composed of a series of bubble plates (11). Between the successive bubble plates are positioned imperforate plates (12).

Downcomer (13) leads the liquid from one bubble plate to the next lower bubble plate, and passes through imperforate plate (12). The space (21) above the bubble plate is connected with the space (14) between the imperforate plate (12) and the next upper plate (11) by means of conduits (15) and (16) which are connected by means of pump (17). Conduit (15) is controlled by a valve (18) and conduit (16) is controlled by a valve (19). As will be seen, there is a pump (17) positioned between each bubble plate and the next upper chamber (14). At the top of the tower (4) is a dephlegmating coil (20).

The operation of the device is as follows:

Vapor is generated in still (2) and passed through line (3) into the tower (4). The various pumps (17) are so operated by the regulation of their speed and/or the regulation of the valves (18) and (19) that successive degrees of vacuum are produced. They would thus function to force the vapors against the back pressure of the next upper plate and also to cause successive stages of exhaust. Thus the various pumps (17), func-

tioning with pump (9), would produce the desired vacuum in (2). These pumps (17) and pump (9) may be conceived of as successive stages of one vacuum producing means, producing successive higher compressions until the vapors are exhausted into the atmosphere or into the vapor collecting line (10) which may be at the desired pressure, as, for instance, when it is connected to a vapor collecting line. While the drawing shows as many pumps as there are plates, it will be understood that one pump may function for two or more plates. Thus vapor may pass through two or three or more plates counter-current to downcoming liquid and then into a stage of vacuum, i. e., a vacuum pump, to be compressed to pass through a succeeding number of plates; then into another stage of vacuum, i. e., a vacuum pump, and so on until it passes into the final condenser. This is a matter of design depending on the capacity of the pumps and the back pressure of each section of the tower. Thus the fractionating column operates at succeedingly higher pressures to produce fractionation and, at the same time, the vapors are exhausted at successive stages to be fractionated between compressions. In this manner, no one pump must bear the brunt of all the exhausting action, and the regulation of fractionation and distillation is made easier. It is obvious that the pumps and tower will be well lagged to prevent heat losses.

As an example of one method of application, suppose distillation is to occur at 25 m. m. pressure and vapors are to be exhausted to atmosphere. Assuming that the back pressure of the condensing unit is equal to 50 m. m., the amount of vacuum, which must be generated by the several pumps of the fractionating column, is equal to approximately 685 m. m. If the fractionating column is composed of ten stages of vacuum, i. e. ten vacuum pumps, each pump must generate approximately 68.5 m. m. vacuum. It is obvious that a different proportioning of the amount of vacuum generated by the various pumps may be obtained. Thus, since the volume of vapor decreases upwardly through the column, the upper pumps may operate to generate greater vacuum than the lower exhaust pumps. It will be observed here that the back pressure of the fractionating column may be and probably will be much less than 685 m. m. and that, therefore, the vacuum generating means associated with the fractionating column may generate much more than the compression necessary to overcome the back pressure of the column. The column functions as a vacuum generating means as well as a fractionating or rectifying column. However, should the back pressure of the column just equal or be greater than 685 m. m., the pumps will function to overcome this back pressure and ensure the maintenance of the high vacuum in the still and cause an exhaust of the vapors at successive stages with a parallel rectification. While the specific embodiment shows the pumps positioned outside the column and connected thereto by relatively long vapor lines, the pumps may be positioned inside the column or positioned adjacent to the column in a manner described in co-pending applications Serial Nos. 217,789 and 217,790, filed September 6, 1927.

The above description is not to be taken as limiting my invention, but merely descriptive of the best manner of carrying it out, my invention being described in the following claims, in which I claim:

1. The process of rectification in vacuum which comprises contacting vapors with partial condensates in successive stages at increasing sub-atmospheric pressure.

2. The process of rectifying vapors in vacuum which comprises passing said vapors into counter-current contact with partial condensates produced from said vapors in a plurality of stages and maintaining the successive stages at increasing sub-atmospheric pressures.

3. The process of rectifying vapors in vacuum which comprises passing said vapors into contact with a partial condensate produced from said vapors, withdrawing said vapors, compressing said vapors to a pressure less than atmospheric, introducing said compressed vapors into counter-current contact with a partial condensate produced from said vapors, withdrawing the vapors resulting from the last mentioned counter-current and finally condensing said vapors.

4. The process of rectifying vapors in vacuum which comprises passing said vapors into counter-current contact with a partial condensate produced from said vapors in a rectification column, compressing said vapors at a plurality of points in said column to produce an increasing pressure in the direction of flow of vapors through said column and maintaining the pressure through said column at less than atmospheric.

5. The process of rectifying vapors which comprises introducing said vapors at sub-atmospheric pressure into a rectification column, withdrawing vapors from a portion of the tower, compressing said vapors to a higher sub-atmospheric pressure, introducing said compressed vapors into said tower at a point adjacent to their point of withdrawal to cause rectification at higher sub-atmospheric pressures.

6. The process of distillation which comprises vaporizing a liquid at sub-atmospheric pressure, fractionating the vapors at succeedingly higher sub-atmospheric pressures but at pressures higher than in the vaporizing means and finally condensing said vapors.

7. The process of distillation, which comprises vaporizing vapors in a vaporizing

means at sub-atmospheric pressure, fractionating the vapors in a fractionating means at succeedingly higher sub-atmospheric pressures but at pressures higher than in the vaporizing means, maintaining the pressure gradient across said fractionating means greater than the frictional resistance of said fractionating means, and finally condensing said vapors.

Signed at Los Angeles, in the county of Los Angeles and State of California, this 31st day of August, A. D. 1927.

PHILIP SUBKOW.

CERTIFICATE OF CORRECTION.

Patent No. 1,771,385. Granted July 22, 1930, to

PHILIP SUBKOW.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 2, line 11, for "if" read it; same page, line 100, claim 3, after "vapors" first occurrence, insert the words "to cause said contact at a pressure higher than during the first mentioned contact"; line 122, claim 5, for "pressures" read pressure; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 9th day of September, A. D. 1930.

M. J. Moore,
Acting Commissioner of Patents.

(Seal)