Sept. 8, 1931. A. E. HARNSBERGER 1,822,733

VACUUM DISTILLATION OF OILS

Filed Oct. 20, 1927

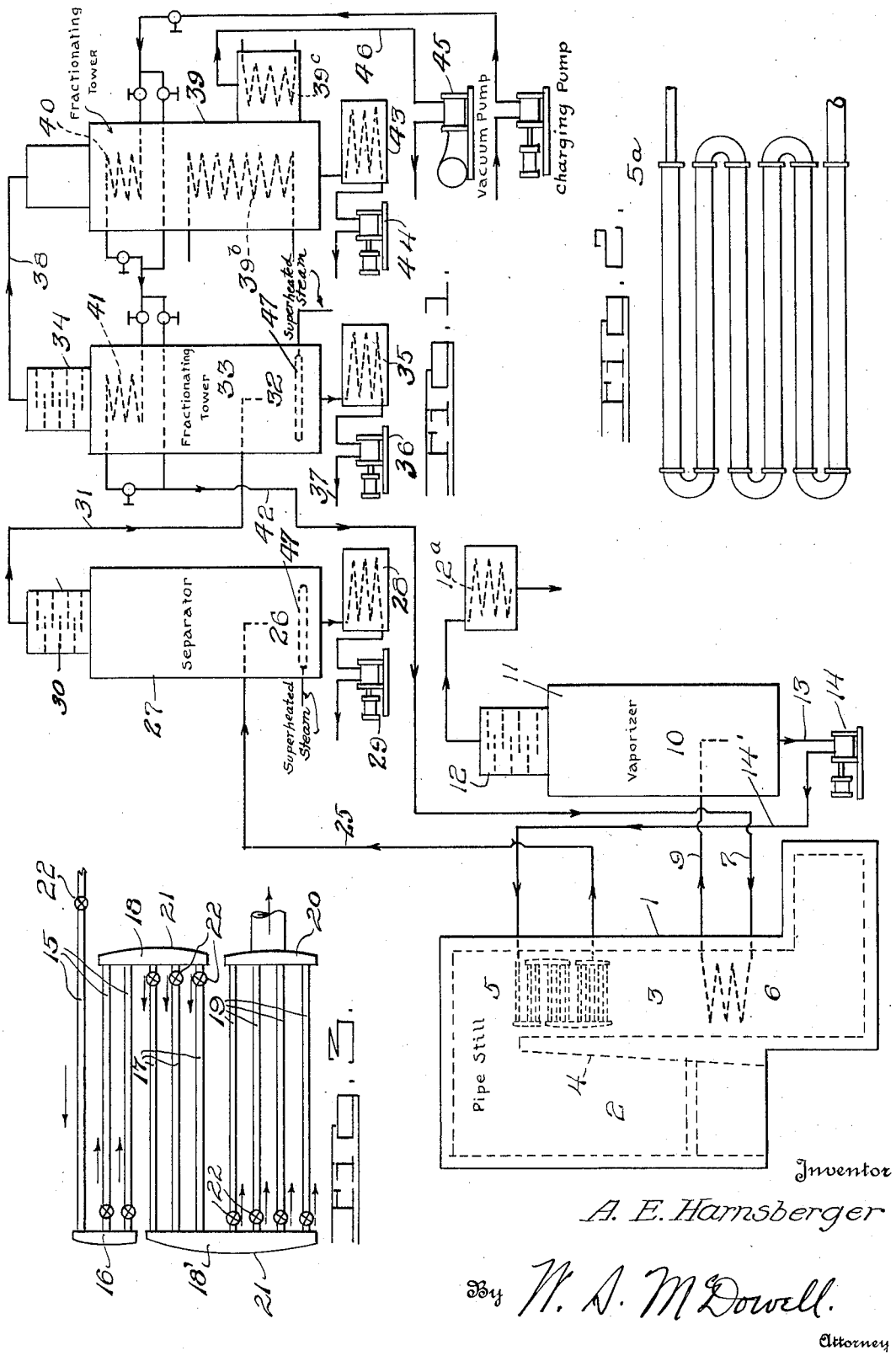


Fig. 1.

Fig. 2.

Fig. 3.

Inventor

A. E. Harnsberger

By W. S. McDowell.

Attorney

Patented Sept. 8, 1931

1,822,733

UNITED STATES PATENT OFFICE

AUDLEY E. HARNSBERGER, OF CHICAGO, ILLINOIS, ASSIGNOR TO THE PURE OIL COMPANY, OF CHICAGO, ILLINOIS, A CORPORATION OF OHIO

VACUUM DISTILLATION OF OILS

Application filed October 20, 1927. Serial No. 227,373.

This invention relates to an improved process for distilling mineral oils and is particularly directed to distillation processes wherein the oils under heat treatment are maintained under high vacuum conditions for the purpose of obtaining the desired vaporization thereof but without permitting any substantial cracking or molecular decomposition of the oils.

It is a primary object of the present invention to provide a vacuum distillation process wherein the primary heater employed is in the form of a pipe or tube still through which the oil is drawn and vaporized, the still being of such form as to provide for progressively increasing vapor space or cross-sectional pipe area from the oil inlet to the oil outlet end thereof, in order that a high vacuum maintained on the outlet end of the still will be permitted to penetrate back to the inlet end of said still so that the entire body of oil contained in the length of the still will be maintained under subatmospheric pressures. The presence of the vacuum throughout the entire length of the still is of primary importance in preventing local overheating of the oil while it is being circulated through the still, and it is in this respect particularly that the present invention has been devised.

Another object of the invention resides in a vacuum distillation process wherein the primary heater utilized is in the form of a tube still and includes pipe units or elements of uniform or constant cross-sectional pipe area throughout the length thereof, but wherein said pipe units are so formed that sufficient vapor area exists to minimize friction between the circulating oils and vapors and the walls of the pipe elements in order to permit the penetration of the vacuum carried on the outlet end of the still back to the oil inlet end thereof.

For a further understanding of the invention reference is to be had to the following description and the accompanying drawings, wherein:

Figure 1 is a diagrammatic view of the apparatus utilized in carrying out the present invention,

Figure 2 is a detail view of a slightly modified form of pipe still from that shown in Figure 1,

Figure 3 is an enlarged view of the heating unit of the pipe still shown in Figure 1.

Referring more particularly to the drawings the numeral 1 designates the setting of a primary heater of the type commonly known as a pipe or tube still. This heater is formed to include the usual combustion chamber 2 and a tube or pipe element chamber 3, the chambers 2 and 3 being separated by means of the usual bridge wall 4. Within the chamber 3 are upper and lower tube banks 5 and 6 respectively, and the lower bank has connected therewith a supply pipe 7, leading from a source of oil supply, and through which pipe the cold or preheated oil is pumped to the tube still. After circulating through the lower bank of tubes the temperature of the oil is raised, for example, to substantially 600 degrees Fah., wherein the lighter fractions of the oil readily vaporize, but without danger of cracking, and pass from the heater by way of an outlet pipe 9, which leads to a vapor chamber 10 provided in a vaporizer 11. The gases and vapors generated by this initial heating step pass overhead from the vaporizer and thence through a cooling coil 12^a, where the vapors are reduced to liquid form and may be suitably stored for future use.

The heavier or unvaporized fractions of the oil collect in the bottom of the vaporizers and are removed therefrom by way of a pipe line 13, in which is situated a pump 14 of any suitable type. This oil or condensate is then forced through the pipe line 14′ to the inlet side of the upper tube bank 5, where the oil is subjected to the higher temperatures prevailing in the tube still or primary heater, but wherein provision is made in the construction and form of this bank of tubes to prevent any appreciable or harmful decomposition of the oil.

The still is thus designed to operate on crude oil and consists essentially of two parts, namely, a preheating section in which the oil is heated above atmospheric pressure to a temperature at which but slight, if any, de-

structive decomposition will take place, and a sub-atmospheric section in which the final vaporization of the oil will be effected.

As an illustration of a specific form which the upper bank of tubes may take, the crude oil after being preheated through various heating apparatus, will enter the still at the inlet pass 15 thereof, where it is drawn through a plurality of parallel pipes, of constant or uniform cross-sectional area. The pipes comprising the pass 15 are connected with a header 16 and the course of tubes immediately below the inlet pass is composed of in this instance three parallel pipes 17. The pipes 17 are also of the same internal diameter or cross-sectional area as the pipes 15, but by providing a plurality of the same for each course, augmented vapor space or area is produced within the upper tube bank to provide for greater area to accommodate increased vapor formation. Similarly, the pipes 17 have their ends connected with a header 18, which is formed so that the tubes 17 will enter one end of the header 18′ for communication with four parallel pipes or tubes 19 which lead to the outlet header 20. The headers 16, 18, 18′ and 20 are provided with arcuate outer walls 21 by means of which the areas thereof are so proportioned as to give an even distribution of oil and vapor flowing from one set of pipes to the other, in order that substantially equal volumes of oil and vapor will flow constantly through all of the pipes or tubes. To further insure this feature the pipes or tubes 15, 17 and 19 may be provided with control valves 22, either automatically or hand controlled which enable the same outlet temperatures to prevail on the outlet end of corresponding pipes or tubes. These valves have been found useful in preventing what is known as the channelling of the oils or vapors through certain tubes and operate to maintain both uniform temperatures and flow through all of said tubes of any given pass.

In my co-pending application Serial Number 148,060, filing date November 12, 1926, now Patent No. 1,666,597, granted April 17, 1928, the tubes comprising the sub-atmospheric section of the heater are shown as possessing a progressively increasing diameter or cross-sectional pipe area from the oil inlet to the oil outlet of this tube section. In the present instance, however, the tubes are of constant cross-sectional area, but are arranged in the multiple order disclosed to accomplish substantially the same end, namely, enlarged vapor area toward the outlet of the still to accommodate vapor formation and reduce pipe friction. By this enlargement of area within the heater a vacuum maintained on the outlet of the still is enabled to penetrate back of the inlet thereof so that the oil in passing through the higher temperature zones of the still will be prevented from overheating and having the value thereof minimized in so far as the use thereof in lubricating stock is concerned.

By this construction the design of the still will be such that due to the increased number of tubes of the same diameter in the various courses of the still, friction loss of the oil passing through the tubes will be so lowered as to allow the vacuum to penetrate the tube bank to a point where the latter is heated to high or approximately cracking temperatures. Without this enlargement of area as the oil progresses through the tube bank, highest efficiency would not in all probability be obtained from a vacuum tube still, since the oil being under pressure could not vaporize except at high temperatures, and at such temperatures would have to carry sufficient heat to overcome the latent heat of vaporization when the vacuum zoning is reached. This would cause a drop in temperature as the oil leaves the tubes and apparently the oil would not be overheated. However, investigation would show that the oil was overheated, at least locally during some point in its passage through the tube bank. Another important advantage obtained by enlarging the vapor area of the tube bank as disclosed resides in the fact that experience has shown that when oil in the tube still vaporizes, very great back pressures are formed due to the vaporization of the oil, and the increased area is provided therefor in order to take care of the vapor volume and to allow for its proper circulation at proper velocity through the tube bank without local overheating.

The increasing vapor area from the inlet to the outlet end of the pipe still 5 is sufficient so that by the resulting decrease of pipe friction or back pressure of oil travelling in the still, four inches of mercury absolute of vacuum on the outlet of the still will penetrate to the inlet of the still, the above mentioned vacuum reducing gradually toward the inlet end of the still, but being present throughout the length of the latter. For example, it has been found that the process will produce unusual distillate such as of cylinder stock grade having a Baumé gravity of 27 and a viscosity in excess of 100 seconds Saybolt (at 210 degrees Fah.) with a flash test of 565 degrees Fah. and a fire test of 610 degrees Fah. and of good color when operating with a vacuum of 1.75 inches of mercury absolute on the outlet of the still, and showing 12 inches of mercury absolute on the inlet side, the temperature of the outlet oil being 690 degrees Fah. Various lighter distillates can be produced by reduction in the outlet temperature of the still, these latter distillates showing superior yields, color, flash and fire tests than those produced by ordinary methods.

A high vacuum still of this design is adapted not only for obtaining high viscosity dis-

tillates of exceptional color, but may be used to reduce a lubricating oil of good color to one of higher viscosity, this being accomplished at a low distillation temperature and consequent slight loss in color. For example, I have found it practical to take an oil of the following specification: Gravity 29.3, flash 500, fire 555, viscosity 69 Saybolt at 210 degrees, color 4½ NPA and reduce the same as follows: Gravity 28.5, flash 575, fire 650, viscosity 95 Saybolt at 210 degrees F., color 5½ NPA. The blending back of this oil with the distillate obtained shows practically the same color as the charging stock, disclosing that this still operates with a minimum of local overheating or destructive distillation.

Any suitable apparatus may be employed following the heating of the oil for effecting its fractionation under vacuum pressures. For example, the oil having been brought up to the desired temperature in the still is discharged from the upper bank 5 of the tube and is conducted through the line 25 to a vapor chamber 26 provided in a separator 27. In the particular form of the invention illustrated the heavier fractions are collected in liquid form in the bottom of the separator and are permitted to pass through a cooling coil 28 therein and are removed from the still by means of a condensate pump 29, which may be of the centrifugal or direct acting type, either steam or electric driven.

To further fractionate the discharge vapors, the top of the separator 27 is provided with a scrubber or dephlegmator 30, in which the vapors are scrubbed and then passed overhead through a vapor line 31 to a fractionating tower 32, which latter includes the usual separating chamber 33 and a superposed scrubber 34. The bottom of the separator or tower 32 is connected with a cooling coil 35, and a discharge pump 36 by means of a pipe line 37, through which the condensate formed in the separator 32 is circulated. The vapors present in the hydrocarbons led to the fractionating tower or separator 32 are permitted to pass through the scrubber 34 and thence by way of a pipe line 38 to a second fractionating tower or separator 39. In this tower there is provided a cooling or refluxing coil 40, through which cold oil may be circulated so as to effect the condensing of the heavier vapors within the tower 39. The coil 40 extends also to a similar coil 41 arranged in the other separating tower 32, and from this coil a pipe line 42 extends to the pipe line entering the lower tube bank 6. It will be apparent that by the provision of suitable valves the flow of oil through the coils 40 and 41 may be readily regulated so as to control the heat exchanging operation thereof in the separators 32 and 39. The final condensate collected in the tower 39 is led through a cooling coil 43 to a pump 44, which may be of the type indicated at 29 and 36. Additional cooling coils 39*b* and 39*c* are provided in the separator 39 to insure final condensation, and through which may be circulated oil, water or brine.

The vacuum in the system may be governed by means of a vacuum pump 45, which communicates with the separator 39 by way of a pipe 46. This pipe has its inlet end connected with the coil chamber 39*c*, through which a cold liquid may be circulated to provide maximum cooling of non-condensable vapors formed during the operation. It will be understood that as many of the fractionating towers or separators may be provided in the system as desired, depending upon the number of cuts or fractions wanted and that the specific form and arrangement of the fractionating equipment may be varied from that herein conventionally illustrated without departing from the principles of the invention. Any non-condensable vapors which may be formed in the operation of the system will be removed by means of the vacuum pump 45, which may be of the displacement type, hurling water type or steam ejector. The pump 45 thus serves to place the entire system under vacuum conditions as far back as the inlet end of the upper tube bank 5.

It will be seen that by the present invention a vacuum system of oil distillation is provided by which the operating temperatures may be maintained at safe levels to prevent the cracking or molecular decomposition of the oils handled by the system. The system thus produces distillate of improved viscosity and color, particularly well adapted for use in the formation and production of lubricating oils. I am aware that the general advantages accruing from vacuum distillation systems has been recognized, but to the best of my knowledge such prior systems have never employed the principle of extending the vacuum back to the point of introduction of the oil into the tube still, or to such positions where the temperatures employed might have the effect of cracking the oil were the vacuum not present, and wherein the oil is passed through the pipe still without reduction in its total weight.

Furthermore, the use of a tube type of heater instead of a shell or drum type, has the advantage that the oil exposed to hot metal travels at high velocity; increasing heat transfer and minimizes to a great extent, the likelihood of local overheating due to the sluggish flow thereof. In this respect the oil and vapors pass through the zone of maximum temperature in a period of not in excess of ten seconds, and usually at a much higher rate of travel. This feature of the invention will permit an outlet temperature of the oil leaving the still which may be above that wherein destructive decomposition takes place, the oil being held at this high temperature less than ten seconds before being cooled to a temperature below its destructive de-

composition point. Such practice, of course, represents an extreme condition, but use of which will cause a minimum of damage to the oil when vaporization of the heaviest fractions is desired, since destructive decomposition is somewhat dependent upon the time element, which is reduced to a point very much lower than that employed by previous methods. Under certain conditions I may resort to the use of steam or mechanical agitation in the various parts of the fractionating apparatus, but not in the still, to further assist in making oils conform to certain specifications in regard to viscosity and flash, and for this reason a coil 47 has been disclosed in the bottoms of the fractionating towers 27 and 32, and superheated steam may be inserted into these fractionating towers by means of such coils in controlled quantities to more completely govern the fractionating apparatus. However, no steam is introduced into the heating coil 5 nor is a given body of oil passing through said coil 5 reduced in weight or removed locally from various points of the coil.

In Figure 2 a modified form of the invention has been disclosed wherein the heater 5*a* is formed from a plurality of pipe units having constant cross-sectional pipe area throughout the entire length thereof but wherein the area of the inlet end of the bank is approximately the same as the area of the outlet end thereof. The area at the inlet end is somewhat in excess of that which the system may properly require but the construction permits the vacuum to penetrate back to the inlet end or a sufficient distance to permit of vaporization without decomposition when a tempearture of not in excess of 750° Fah. is maintained on the oil. This form of still is not as desirable as that of the increasing area type due to low heat transfer in the inlet passes and further to its increased initial cost, however, it does have the advantage of vacuum penetration and in this respect may be utilized to an advantage over standard types of stills.

What is claimed is:

1. In a method of distilling mineral oil, that procedure which consists of passing mineral oil through a heated zone in a stream of restricted cross section and splitting said stream into a plurality of substreams while maintaining the substreams of substantially equal cross section with respect to each other, and of substantially equal cross section with respect to the first named stream.

2. In a method of distilling mineral oil, that procedure which consists of passing mineral oil through a heated zone in a stream of restricted cross section and splitting said stream into a plurality of substreams while maintaining the substreams of substantially equal cross section with respect to each other, and of substantially equal cross section with respect to the first named stream, the composite paths traversed by such plurality of substreams being of such capacity that four inches absolute of vacuum maintained at the discharge end of the paths penetrates substantially to the inlet end thereof.

3. In a method of distilling mineral oil, that procedure which consists of passing mineral oil through a heated zone in a stream of restricted cross section and splitting said stream into a plurality of substreams while maintaining the substreams of substantially equal cross section with respect to each other, and of substantially equal cross section with respect to the first named stream, while maintaining that degree of vacuum at the discharge end of the composite path traversed by such plurality of streams as to cause said vacuum to exert its effect substantially to the inlet end of said path.

4. In a method of distilling mineral oil, that procedure which consists of passing mineral oil through a heated zone in a stream of restricted cross section and splitting said stream into a plurality of substreams while maintaining the said substreams of substantially equal cross section with respect to each other, and of substantially equal cross section with respect to the first named stream, and while individually controlling the flow of the several streams to maintain uniform flow and temperature at the discharge ends of said streams.

5. In a method of distilling mineral oil, that procedure which consists of passing mineral oil through a heated zone in a stream of restricted cross section and splitting said stream into a plurality of substreams while maintaining the substreams of substantially equal cross section with respect to each other and of substantially equal cross section with respect to the first named stream, while maintaining that degree of vacuum at the discharge end of the composite path traversed by such plurality of streams as to cause the vacuum to exert its effect substantially to the inlet end of said path, and while controlling the flow of the several streams to maintain uniform flow and temperature at the discharge end of the several streams.

6. In a method of distilling mineral oil, that procedure which consists of passing mineral oil through a heated zone in a stream of restricted cross section and splitting said stream into a plurality of substreams while maintaining the substreams of substantially equal cross section with respect to each other, and of substantially equal cross section with respect to the first named stream and maintaining such a degree of vacuum at the discharge ends of the paths as will penetrate back to the first named stream, said vacuum being in such degree that vaporization of the oil will take place when the oil is heated to a temperature of 750° F.

7. Apparatus for the distillation of mineral oil, comprising a pipe still formed of a plurality of groups of pipes and means for serially connecting the several groups of pipes, the number of pipes in the several groups successively increasing along the length of the still and the diameter of all of said pipes being substantially the same.

In testimony whereof I affix my signature.

AUDLEY E. HARNSBERGER.