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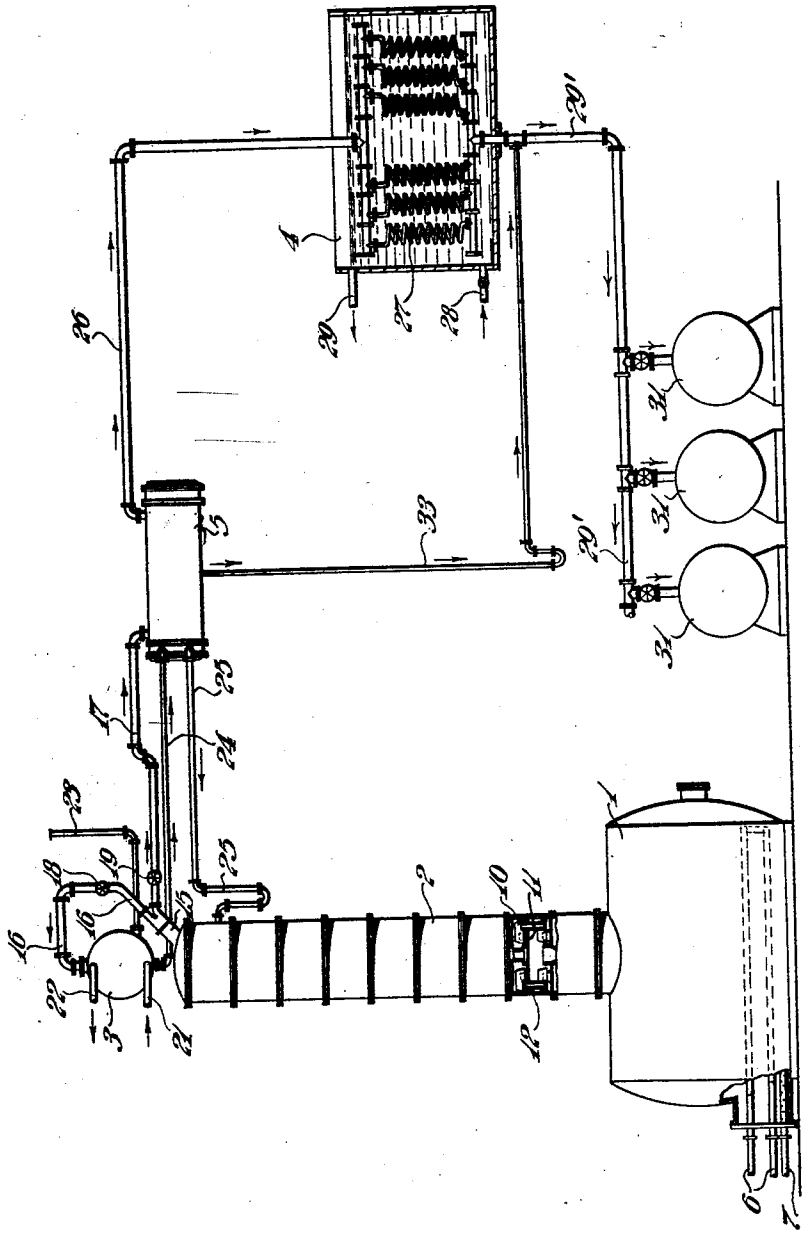

INVENTOR.
Fred Denig.

BY Jesse R. Langley
ATTORNEY.

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FRED DENIG, OF PITTSBURGH, PENNSYLVANIA, ASSIGNOR TO THE KOPPERS COMPANY, A CORPORATION OF DELAWARE

METHOD OF AND APPARATUS FOR OIL DISTILLATION

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My invention relates to the distillation of hydrocarbon oil for the separation of constituents thereof in relatively uncontaminated form. It has an especial relation to the distillation of hydrocarbon oil such, for example, as that recovered from coal gas and consisting largely of a mixture of benzol, toluol and xylols, for the recovery therefrom of such constituents in relatively uncontaminated form. My invention may, however, be employed in the distillation of other analogous oils.

In the methods and apparatus of the prior art, intermittent distillation of crude light oil of the character described above is necessarily accompanied by frequent regulation throughout the course of the distillation in accordance with the nature of the principal constituent being separated at any one time. In intermittent distillation operations of this character, the various constituents are volatilized in the order of their respective boiling points, the constituents having the lowest boiling points being volatilized first. Due to the varying temperatures at which these constituents condense, it has been necessary in the past to provide dephlegmation apparatus therefor which is capable of temperature regulation according to the condensation temperatures of the various constituents, the volatilization of which varies throughout the course of distillation.

Such dephlegmation has been accomplished in the past by subjecting the vapors evolved during the distillation operation to indirect contact with a cooling fluid such, for example, as water, to effect a partial condensation, and the temperature regulation has been effected by varying the amount or temperature of water delivered to the dephlegmating apparatus.

Dephlegmation of this character has two other disadvantages. In the first place, the amount of condensate collected in the dephlegmator and returned as reflux to the distilling apparatus is necessarily only a fraction of the total condensate evolved, and is usually much less in amount than the amount of material which is permitted to leave the dephlegmator. Any attempt to increase the amount of condensate in the dephlegmator inevitably causes a material lengthening of the distillation process.

In the second place, it necessarily follows from a consideration of the nature of the partial condensation taking place in a dephlegmator of this type that the refluxed liquid differs somewhat from the vapors evolved, being in fact of somewhat lower volatility.

Consequently, both the amount and nature of the refluxed liquid in a process of the character indicated above leave much to be desired, and even with large fractionating apparatus, have made it difficult or impossible in the past to obtain "pure products" (that is, commercially pure benzol, toluol and the like) within a reasonable time.

Furthermore, in such prior processes, it has been difficult or impossible to avoid the production of large amounts of intermediate fractions, which cause considerable loss of time and necessitates re-distillation.

An object of the present invention is to provide a method of and apparatus for distillation of hydrocarbon oil of the character described, in which the regulation of fractionating or dephlegmating temperature required throughout the course of the distillation operation is self-controlled according to the constitution of the vapors being evolved at any time.

My invention has for other objects such other operative advantages and results as may hereinafter be found to obtain.

In the copending application of Joseph Van Ackeren, Serial No. 480,647, filed September 9, 1930, there is described and claimed an intermittent distillation operation and apparatus therefor in which the oil to be dis-

tilled is maintained throughout the distillation at its boiling point in the usual manner and the vapors thereby evolved from the oil are conducted through a rectifying column such, for example, as a series of conventional bell-and-tray sections, passing through the same in countercurrent with a suitably proportioned flow of reflux oil.

Reflux oil is introduced to the rectifying column at a point sufficiently removed from the heating vessel to accomplish an adequate rectification of the vapors, and prior to such introduction is subjected to an exchange of heat from the vapors passing through the rectifying column at substantially the same time.

In the aforesaid copending application of Joseph Van Ackeren, this heat exchange is effected by withdrawing a portion of the reflux oil from the rectifying column and commingling the thus withdrawn reflux oil (which issues from the column at a temperature substantially equal to that of the vapors passing therethrough) with a quantity of fresh reflux oil and then returning the mixture to the rectifying column at a point above the point of withdrawal of said portion of the reflux oil. The fresh reflux oil preferably consists of a portion of condensate obtained by cooling the rectified vapors issuing from the top of the rectifying column.

In this manner there is accomplished a self-regulation of the temperature and nature of the reflux oil, whereby an exceptionally efficient rectification is made possible without the usual necessity for frequent regulation throughout the course of the distillation operation as the temperatures of distillation increase.

According to my present invention, an exchange of heat between the vapors produced in the distillation and the reflux oil is accomplished by dividing the stream of rectified vapors issuing from the top of the rectifying column into two portions, one of which is immediately subjected to a cooling operation sufficient to condense all condensible constituents of the same, the condensate being then subjected to indirect heat exchange with the remaining portion of the vapors and returned to the still as reflux oil, while the remaining portion of the vapors, after such heat exchange with the condensate obtained from the first-mentioned portion, is condensed in the usual manner.

In this manner, the increase of temperature of the vapors in the rectifying column causes a corresponding increase in the temperature of the reflux oil added to the same and in spite of the change of temperature throughout the operation, no regulation on the part of the operator is required, the process and apparatus being to this extent self-regulated.

Moreover, it is possible in this manner to overcome the disadvantages of the usual type of dephlegmation operation, in which the reflux oil returned to the column necessarily differs from the constituent being volatilized. It will be obvious that according to my invention, the reflux oil added at any time is substantially identical with the principal constituent being recovered at the same time.

In order that my invention may be clearly set forth and understood, I now describe, with reference to the accompanying drawing, a preferred form in which it is accomplished and embodied. In this drawing,

The single figure is a more or less diagrammatic view, partly in elevation and partly in vertical section, of apparatus for distilling hydrocarbon oil in accordance with the present invention. Referring to the drawing, the apparatus employed consists principally of a still tank or vessel 1, a rectifying column 2, condensers 3 and 4 and a vapor-to-oil heat exchanger 5. The still tank 1 is of conventional design and is provided with means for heating the same, for example, a number of indirect steam coils 6 and a perforated conduit 7 for introducing steam directly to the charge within the still tank.

The rectifying column 2 is in the present instance shown as comprising a plurality of bell-and-tray sections of conventional type and is preferably located directly over the still tank 1 or is in any event so connected therewith as to receive therefrom vapors evolved during the distillation of a hydrocarbon oil.

Each bell-and-tray section is provided with a suitable number of bells 10 located over vapor uptakes 11, and one or more liquid downflow pipes 12 so disposed as to provide a liquid seal for the bells 10.

Vapors are withdrawn from the top of the rectifying column 2 through a vapor conduit 15 having branches 16 and 17 with valves 18 and 19, respectively.

The vapor branch conduit 16 communicates with the condenser 3 which is of the usual indirect type and is provided with cooling water inlet and outlet conduits 21 and 22, respectively. The condenser 3 is also provided with an outlet conduit 23 which serves to remove non-condensible constituents of the vapors passing through the condenser 3 and communicates at its lower portion with a conduit 24 which serves to remove oils condensed from the vapors passing through the condenser 3.

The conduit 24 communicates with the vapor-to-oil heat exchanger 5 which in turn communicates with a sealed oil conduit 25 serving to return oil from the heat exchanger 5 to a point at or near the top of the rectifying column 2.

The vapor branch conduit 17 also communicates with a vapor space within the heat exchanger 5 which in turn communicates

through a vapor conduit 26 with coils 27 located within the condenser 4, which is provided with a cooling water supply conduit 28 and an overflow conduit 29 so disposed as to keep the coils 27 immersed in cooling water in the condenser 4.

The coils 27 communicate at their lower ends with a condensate line 29′ leading through suitable connections to a plurality of receiving tanks 31.

The vapor space within the vapor-to-oil heat exchanger 5 also communicates through a sealed conduit 33 with the condensate line 29′.

In the operation of the method of the present invention, the still tank 1 is charged with a suitable quantity of the oil to be distilled and heat is supplied, at first through the indirect steam coils 6 and later through the direct steam coils 7 when necessary, to maintain the oil at its boiling point and thus to effect a continuous vaporization of constituents of the oil, the removal and recovery of which in uncontaminated form are desired.

The vapors evolved during the distillation and which vary throughout the course of the distillation from the vapors of the lowest boiling constituent of the oil to the vapors of the highest boiling constituent of which recovery is contemplated, pass upward through the rectifying column 2 and are subjected to a countercurrent flow of reflux oil introduced through the conduit 25 to the rectifying column 2.

By reason of such contact, vapors are stripped of their higher boiling constituents so that except during the inevitably immediate periods, said vapors are rectified to contain substantially only the lowest boiling constituent of the vapors present in the system at any time, while any of said low boiling constituent may in spite of such rectification be present in the reflux oil is caused to be revolatilized in the absence of the condensation of higher boiling constituents of the vapors. In this manner what may be described as a double rectifying action is accomplished.

Upon issuing from the rectifying column 2, a portion of the rectified vapors enters the condenser 3 where they are subjected to indirect contact with a flow of cooling water of such temperature and in such quantity as to effect a condensation of substantially all of the condensible constituents thereof.

The condensate thereby secured, and which is substantially identical in constitution with the vapors issuing from the rectifying column 2 is then subjected to an exchange of heat from the remaining portion of the vapors issuing from the rectifying column 2 and is then returned to the rectifying column 2 as reflux. It will be obvious that reflux oil obtained in this manner approximates both in temperature and constitution the vapors within the rectifying column 2 at the point of introduction of the reflux oil and that any changes in temperature or constitution of the reflux oil will effect a corresponding change in the nature and temperature of the reflux oil so that an extremely efficient rectification is made possible.

The condensate obtained from the condenser 4 is delivered to the receiving tanks 31 in the usual manner in accordance with the nature of such condensate.

That is to say, so long as the condensate consists of substantially pure benzol it is delivered to one of the tanks 31 set apart for the purpose of receiving the same and when the condensate begins to show signs of contamination with toluol which is the next highest boiling constituent of the mixture being distilled, it is delivered to another of the tanks 31 set apart for the purpose of receiving the intermediate condensate. Subsequently, upon substantially complete disappearance of benzol from the condensate, the latter is then delivered to still another of the tanks 31 set apart for the purpose of receiving toluol and so on throughout the distillation.

According to the present invention, the amounts of intermediate distillates obtained may be considerably reduced in comparison with the amounts of distillates obtained according to methods now generally employed and this constitutes an advantage of the present invention. However, such intermediates are conveniently disposed of by introducing them together with quantities of crude oil to be distilled in the following or any subsequent distillation operation.

Moreover, the valves 18 and 19 are regulated by the operator to maintain a proper balance between the reflux oil and the vapors removed for condensation in accordance with the requirements of the particular oil being distilled.

Such operation is well within the skill of the ordinary operator and it is ordinarily possible for such an operator to so regulate these valves at the beginning of the distillation that no further regulation during the course of the distillation operation is required.

It will therefore be obvious that my invention dispenses with the tedious requirements of regulation of a dephlegmating operation that have been necessary in the past and makes provision for a self-regulation of the temperature and nature of the reflux oil.

Where it is desirable, however, to reduce the amount of intermediate condensate obtained, the present procedure may be modified in accordance with that described and claimed in the aforesaid copending application of Joseph Van Ackeren. According to this method, all of the distillate evolved from the column is returned thereto as reflux during the periods in which it is impossible to

avoid the production of an intermediate condensate.

To illustrate this modification of the present process, it may be assumed that at a given time the distillation operation is producing substantially uncontaminated benzol, the reflux being returned to the rectifying column consists also of substantially uncontaminated benzol. When the amount of benzol in the vapors within the rectifying column 2 falls to such a point that the condensate begins to show contamination with toluol, the next higher boiling constituent, the valve 19 is closed and all of the vapors issuing from the top of the column 2 are subjected to condensation in the condenser 3 and the condensate being then recirculated as reflux.

Since the vapors leaving the top of the column during this period are necessarily richer in benzene (the most volatile constituent now present) and the liquid in the top of the column, the reflux returned to the column will also be richer in benzene than the liquid with which it mixes. For this reason, benzene will be caused to be concentrated in rectified form in the uppermost trays.

When this condition is established, the valve 19 is reopened and the valve 18 closed until the rectified benzol in the upper part of the rectifying column passes over the condenser 4. After a short period, it becomes impossible to prevent the collection of an intermediate benzol in toluol fraction and the valve 18 is then opened, the valve 19 remaining open. The intermediate fraction obtained during the time the valve 18 is closed is delivered to the intermediate receiver selected for the collection of this material. A condensate then changes sharply to substantially pure toluol and is delivered to the appropriate receiver.

A similar operation may be performed, when so desired, during each of the intermediate periods inevitably encountered during the course of the distillation. After all of the highest boiling fraction which it is possible to obtain in satisfactorily pure form has been driven over, the distillation is ended. The intermediates then recovered during the distillation are then mixed with a fresh batch of oil for redistillation in the manner indicated hereinabove.

In this instance it is necessary to continue to deliver the condensate to the intermediate receiving tank for a short time after the resumption of normal operation in order to prevent the introduction into the toluol receiver of such small amounts of benzol as may be contained in the upper trays of the column 2 at the time the valve 19 is closed.

While my invention has been described hereinabove with respect to a preferred form of apparatus and a preferred method of procedure, it will be obvious to those skilled in the art that it is capable of considerable modification in detail and is therefore not limited to such illustrative examples but may be variously employed and practiced within the scope of the claims hereinafter made.

I claim as my invention:

1. In a method of distilling hydrocarbon oil of the class described, the improvement which comprises charging a batch of the oil into a vessel, applying heat thereto to maintain the oil at its boiling point, conducting vapors thereby evolved through a rectifying column, withdrawing rectified vapors from said column, cooling a portion thereof to condense the same, subjecting the condensate thereby obtained to heat exchange with another portion of said vapors, and then returning the thereby heated condensate to said rectifying column as reflux.

2. Apparatus for distilling hydrocarbon oil of the class described which includes a vessel adapted to receive a batch of said oil, means for applying heat thereto to maintain the oil at its boiling point, a rectifying column adapted to receive thereby evolved vapors therefrom, means for withdrawing rectified vapors from said column, a condenser located in the path of a portion of said rectified vapors, means for subjecting the condensate obtained therein to heat exchange with the remaining portion of said rectified vapors, and means for returning the thereby heated condensate from said heat exchanging means to said rectifying column as reflux.

3. The method of distilling crude light oil recovered from a gas and comprising a mixture of at least two constituents having different boiling points, which comprises; charging a batch of said oil into a vessel; applying heat thereto to maintain the oil at its boiling point; conducting vapors thereby evolved through a rectifying column; withdrawing vapors from said column; during such time as said vapors contain substantially only the lower-boiling constituent, separating said vapors into two portions, cooling one portion to condense the same, subjecting the condensate thereby obtained to heat exchange with said other portion, and then returning said thereby heated condensate to said rectifying column as reflux, and separately condensing the other portion of said vapors; when said vapors commence to include considerable amounts of the higher-boiling constituent, condensing all of the vapors withdrawn from said column and returning the condensate to said column as reflux, while continuing the distillation, until the vapors of the lower-boiling constituent remaining in said column are caused to be concentrated in the upper portion of said column; then removing the vapors from said column and condensing the same without returning the condensate to said column, until said vapors no longer contain a substantial

amount of the higher-boiling constituent; and then separating the vapors from the column into two portions, condensing one portion, subjecting the condensate to heat exchange with the other portion, returning the thereby heated condensate to the column as reflux and separately condensing said other portion of said vapors.

In testimony whereof, I have hereunto subscribed my name this 28th day of February, 1931.

FRED DENIG.