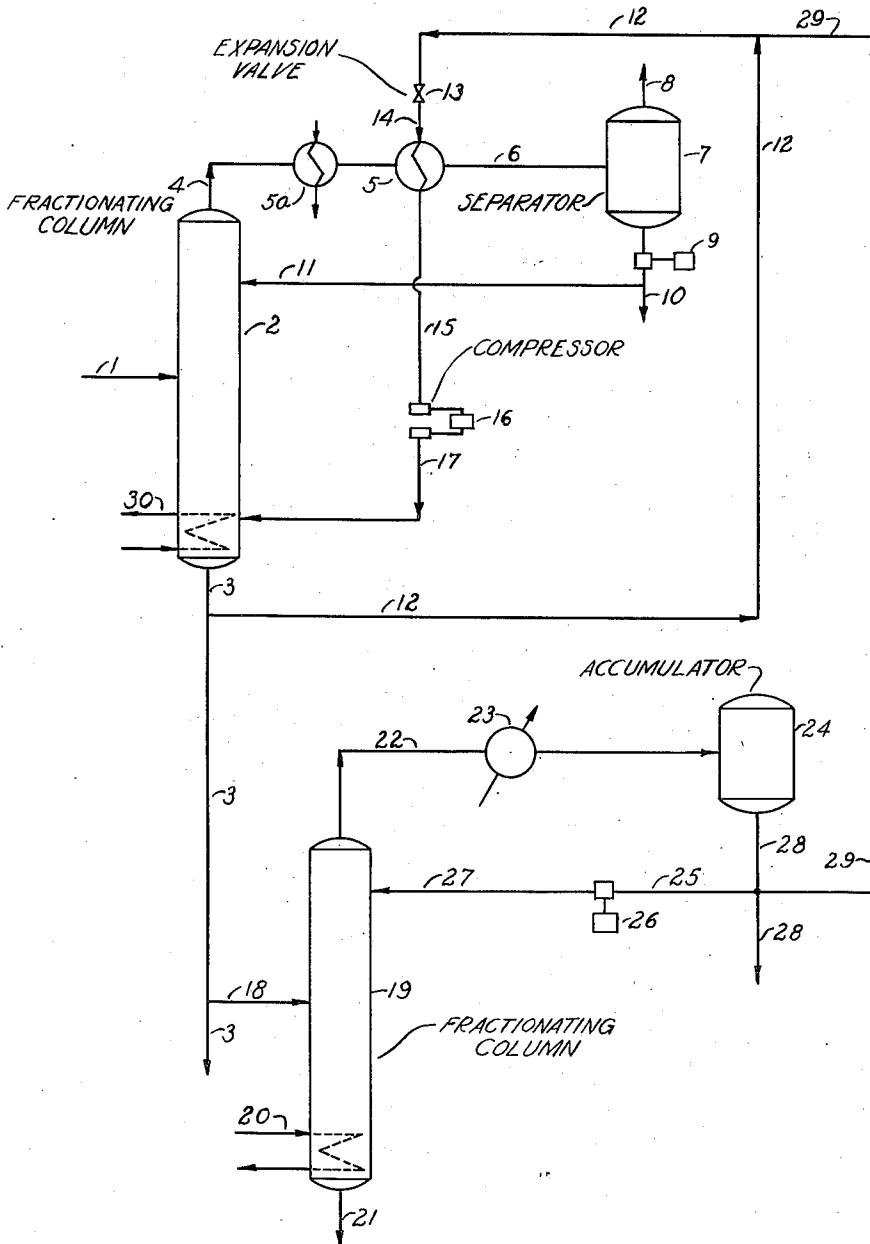


Aug. 24, 1943.

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FRACTIONAL DISTILLATION

2,327,643

Filed Aug. 16, 1941



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2,327,643

FRACTIONAL DISTILLATION

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Application August 16, 1941, Serial No. 407,137

7 Claims. (Cl. 62-175.5)

This invention relates to distillation, and is particularly concerned with a method of supplying heat to the reboiler, and abstracting heat from the reflux condenser, of a fractionating column. Although not limited thereto, the invention may be applied with particular advantage to the distillation of light hydrocarbon mixtures having relatively narrow boiling ranges such as, for example, mixtures of iso- and normal butane.

One of the objects of the invention is to provide an economical method of reboiling and refluxing a fractionating column.

Another object of the invention is to reduce the cost of operating a fractionating column at a high reflux ratio, so as to accomplish inexpensively the separation of closely boiling constituents. Other objects of the invention will appear during the course of the description hereinafter given.

It is contemplated by the invention to withdraw liquid from a fractionating column, at some point below the feed inlet, and to reduce the withdrawn liquid to a pressure at which it will have a boiling point lower than the condensing temperature of the vapors issuing from the top of the column. The liquid is then indirectly contacted with the overhead vapors, and in vaporizing, cools and condenses them. Thereafter it is recompressed to its original pressure and returned to the lower part of the column, at the higher temperature to which it is raised by the compression, so supplying heat for reboiling the column.

A more detailed description of a preferred embodiment of the invention will now be given by way of illustration and with reference to the accompanying drawing, which is a diagrammatic flow sheet. It is to be understood that the embodiment of the invention shown and described is exemplary only.

Referring now to the drawing, material to be fractionated is introduced at any suitable pressure and temperature through line 1 into a fractionating column 2, and is subjected to conventional fractionation therein. The overhead product of the fractionation is withdrawn from the top of the column through line 4 and passed serially through heat exchangers 5a and 5 wherein it is cooled and wherein at least a portion of it is condensed. The condensate and any material remaining uncondensed pass through line 6 into an accumulator or separator 7, wherein disengagement of any uncondensed material from the condensate is allowed to take place. The uncondensed material, if such there be, is withdrawn from separator 7 through line 8 and disposed of

as desired while the condensate is withdrawn through line 10 and picked up by pump 9. A quantity sufficient for refluxing purposes is returned to the top of the column through line 11 while the remainder, constituting the liquid overhead product of the distillation, is disposed of as desired through line 10.

The net production of bottoms product from the distillation operation is withdrawn from the base of column 2 through line 3 and disposed of as desired. An additional quantity of bottoms product, which is subject to continuous recycling to the column as hereinafter described, is also withdrawn through line 3 and diverted through line 12 to a valve 13 through which it is expanded and partially vaporized at a lower pressure, such that upon being subsequently passed through line 14 into indirect contact with the column overhead vapors in heat exchanger 5 it will be completely vaporized, and will extract heat from the column overhead vapors to bring about condensation thereof. Immediately upon being reduced in pressure, sufficient of the bottoms product material will vaporize to effect cooling of the whole quantity down to the boiling point which it has at the reduced pressure, and as previously noted this boiling point must be below the condensation temperature of the vapors to be condensed in 5. The bottoms product material vaporized in heat exchanger 5 passes through line 15 to a compressor 16 wherein it is raised to its original pressure and wherein it simultaneously undergoes a substantial rise in temperature. The heated compressed vapors travel through line 17 and are re-introduced into the base of column 2. They supply heat to the material in the base of the column for reboiling purposes and subsequently undergo condensation, eventually being withdrawn again as previously described. In the event that the quantity of material required to be so re-circulated for condensing purposes in exchanger 5 is less than the amount needed for reboiling purposes after recompression, I may supply the deficiency by means of a conventional reboiler such as a steam heated coil 30 located in the base of column 2. If on the other hand the quantity of material re-circulated is desired to suffice for reboiling purposes and this amount is insufficient for condensing the reflux and product, the deficiency is made up by means of the auxiliary condenser 5a, to which any suitable extraneous refrigerant may be supplied. Otherwise condenser 5a is unnecessary and may be omitted.

The procedure described is most advanta-

geously applied to distillation processes which operate at pressures above atmospheric, but it may be applied if desired to atmospheric or even vacuum distillations. The utility of the invention is particularly great in connection with the separation of binary mixtures, the components of which differ only slightly in their boiling points. In the separation of normal from iso-butane for example, the column top temperature may differ by as little as 24° F. from the bottom temperature, and a relatively high reflux ratio and a large amount of reboiler heat are necessary. Under such circumstances it is necessary to reduce the pressure of the bottom product re-circulated through line 12 relatively little in order to make possible the vaporization thereof in contact with the overhead vapors, and a correspondingly small compression ratio is necessary to return the vaporized material to the base of the column. It is characteristic of narrow boiling range fractionation that large amounts of heat must be added and withdrawn at closely adjacent temperature levels. Ordinarily, of course, the generation of heat by gas compression is not particularly economical, but under the circumstances outlined, in which the compression serves the dual purpose of providing heat and refrigeration, the arrangement is a very economical one.

Under other circumstances I may employ my invention in connection with a two-stage distillation process involving re-distillation of the bottoms product from a primary fractionating column. A suitable arrangement for practicing this embodiment is shown in the drawing, and comprises a line 18 for transferring the bottoms product of a distillation column 2 to a secondary column 19 which is operated in a conventional manner to deliver a final bottom product through line 21. Reboiling of column 19 is accomplished by means of steam heated coil 20, and overhead vapors are withdrawn through line 22 for condensation in condenser 23. The condensate is collected in an accumulator 24 and reflux is returned through line 25, pump 26 and line 27 to the top of column 19. The net production of overhead product from column 19 is withdrawn through line 28, and an additional quantity which is to be constantly recirculated as hereinafter described is passed through line 29 into line 12 for reduction in pressure through valve 13, in exactly the same manner as has been explained with reference to the material re-circulated from line 3 through line 12. The material returned through line 29 for cooling and re-boiling purposes will serve equally as effectively as the original bottoms from column 2, of which it is a derivative. If desired, both the bottoms from column 2 and the overhead material from column 19 may be re-circulated and admixed in any desired proportions for joint service in cooling the overhead and reboiling the bottom of column 2. The re-circulation of overhead material from 19 is not so advantageous as the re-circulation of bottoms from 2, because it involves the refractionation of the material re-circulated.

It will be understood that the reboiler heat and reflux refrigeration provided by the invention are not necessarily utilized at the extreme bottom of the tower and in the external reflux condenser, respectively. It is frequently desirable to provide for reflux cooling at intermediate points of a tower above the feed inlet, and for reboiling at intermediate points below the feed inlet. In accordance with the invention, by suitable selection of the point of withdrawal and

the pressure to which the withdrawn material is expanded, refluxing and reboiling at intermediate points may be easily achieved. For example, I may withdraw liquid from a column at a point between the bottom and the feed inlet and after expansion thereof to a sufficiently low pressure and temperature, pass the expanded material through a coil located in the column between the feed inlet and the top. In this manner I may condense distillate vapors as reflux intermediate their traverse of the column. Thereafter the expanded and vaporized material may be returned to the column preferably at or adjacent its point of withdrawal, for reboiling purposes.

In order to illustrate more fully the manner in which the invention may be practiced, an example will now be given. A mixture of ethane and ethylene may be fractionated by means of the apparatus shown in the drawing, using only column 2 and associated elements for the purpose. The operation is suitably conducted at a column pressure of 250 pounds per square inch absolute, a top temperature of -32° F. and a bottom temperature of 9° F. Material (ethane) withdrawn from the bottom of the column may then be expanded to a pressure of 110 pounds per square inch absolute, at which part of it will vaporize and its temperature will fall to approximately -42° F. After abstracting heat from the ethylene vapors in a condenser such as 5 and recompression to 250 pounds absolute the ethane re-circulated would be at approximately 25° F. When re-introduced into the base of the column this ethane would produce a reboiling effect equivalent to the vaporization by direct heating of slightly more than an equivalent amount of ethane, because the return vapors would be superheated slightly (from 9° to 25° F.). It would be fortuitous, depending chiefly upon the reflux ratio, the feed composition, and the conditions of the feed entering the column, if the reboiling and refluxing provided by any rate of ethane re-circulation in accordance with the invention happened to be exactly in balance for such an operation. More commonly, either auxiliary reboiling heat or reflux refrigeration will be required.

Under the conditions of the example, the re-circulation of 18,300 pounds per hour of ethane would provide about 2,340,000 B. t. u. per hour of reflux refrigeration and 3,180,000 B. t. u. per hour of reboiler heat. The ethane recompressor would consume approximately 239 shaft horsepower.

I claim:

1. In the fractional distillation of a volatile mixture of relatively narrow boiling range involving the cooling of distillate vapors by indirect heat exchange with a cooling medium to condense at least a portion of said vapors and the use of thus-produced condensate as reflux in the fractionating zone, the improvement which comprises withdrawing from the fractionating zone liquid which has been enriched by fractionation with the heaviest component of the feed, reducing thus-withdrawn liquid to a pressure at which its boiling point will be below the condensation temperature of distillate vapors to be condensed, using material thus reduced in pressure as cooling medium to accomplish condensation of distillate vapors and thereby vaporizing material so used, compressing thus-produced vapors to the pressure existing in the

fractionating zone and returning thus-compressed vapors to said zone.

2. A fractional distillation process for a volatile liquid of relatively narrow boiling range which comprises withdrawing liquid from a fractionating zone and reducing the pressure thereon to effect partial vaporization and cooling thereof, using thus cooled material to cool vapors produced in the process and to produce a condensate therefrom, thereafter raising thus-used material to its original pressure and returning it to said zone.

3. In the distillation of a volatile mixture of narrow boiling range in a fractionating column to produce a bottoms product consisting substantially entirely of a single component of said mixture, the improvement which comprises withdrawing liquid from said column at a point below the feed inlet and using said liquid at a pressure lower than that maintained in said column as a cooling medium to cool by indirect heat transfer and condense distillate vapors while said liquid is under a pressure at which it has a boiling point below the condensation temperature of said vapors, whereby said liquid is vaporized, compressing the thus-produced vapors and returning them to the column.

4. In the distillation of a volatile mixture of narrow boiling range in a fractionating column to produce a bottoms product consisting substantially entirely of a single component of said mixture, the improvement which comprises withdrawing bottoms product liquid from said column and reducing the pressure thereon sufficient to lower the temperature and boiling point thereof below the condensation temperature of the column overhead vapors, exchanging heat between bottoms product liquid at said reduced pressure and overhead vapors to bring about condensation of said vapors and vaporization of said liquid, compressing vapors thus produced and introducing the compressed vapors into the lower portion of said column to effect reboiling of said column.

5. In the fractional distillation of a volatile mixture of relatively narrow boiling range involving the cooling of distillate vapors by indirect heat exchange with a cooling medium to condense at least a portion of said vapors and the use of thus-produced condensate as reflux in the fractionating zone, the improvement which comprises using as said cooling medium a material obtained by re-distillation of the less volatile product of said distillation, said material while so used being under a pressure low enough to permit vaporization thereof at the condensation temperature of said distillate vapors to be condensed, and introducing said material after said use into the fractionating zone adjacent the point of withdrawal of said less-volatile product.

6. In the distillation of a volatile mixture of narrow boiling range, the improvement which comprises withdrawing liquid from a fractionating zone, separating liquid thus withdrawn into light and heavy fractions, reducing the pressure on said light fraction to effect partial vaporization and cooling thereof and using the material thus reduced in pressure as a cooling medium for vapors produced in the fractionating zone to effect condensation thereof, thereafter increasing the pressure on the thus used material and returning it to said fractionating zone.

7. A fractional distillation process for a volatile liquid of relatively narrow boiling range which comprises withdrawing liquid from a fractionating zone and reducing the pressure thereon to effect partial vaporization and cooling thereof, using thus cooled material to cool vapors produced in the process and to produce a condensate therefrom, thereafter increasing the pressure on the thus used material and returning it to said fractionating zone.

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