June 25, 1935. D. E. STINES 2,006,186

RECTIFYING PROCESS

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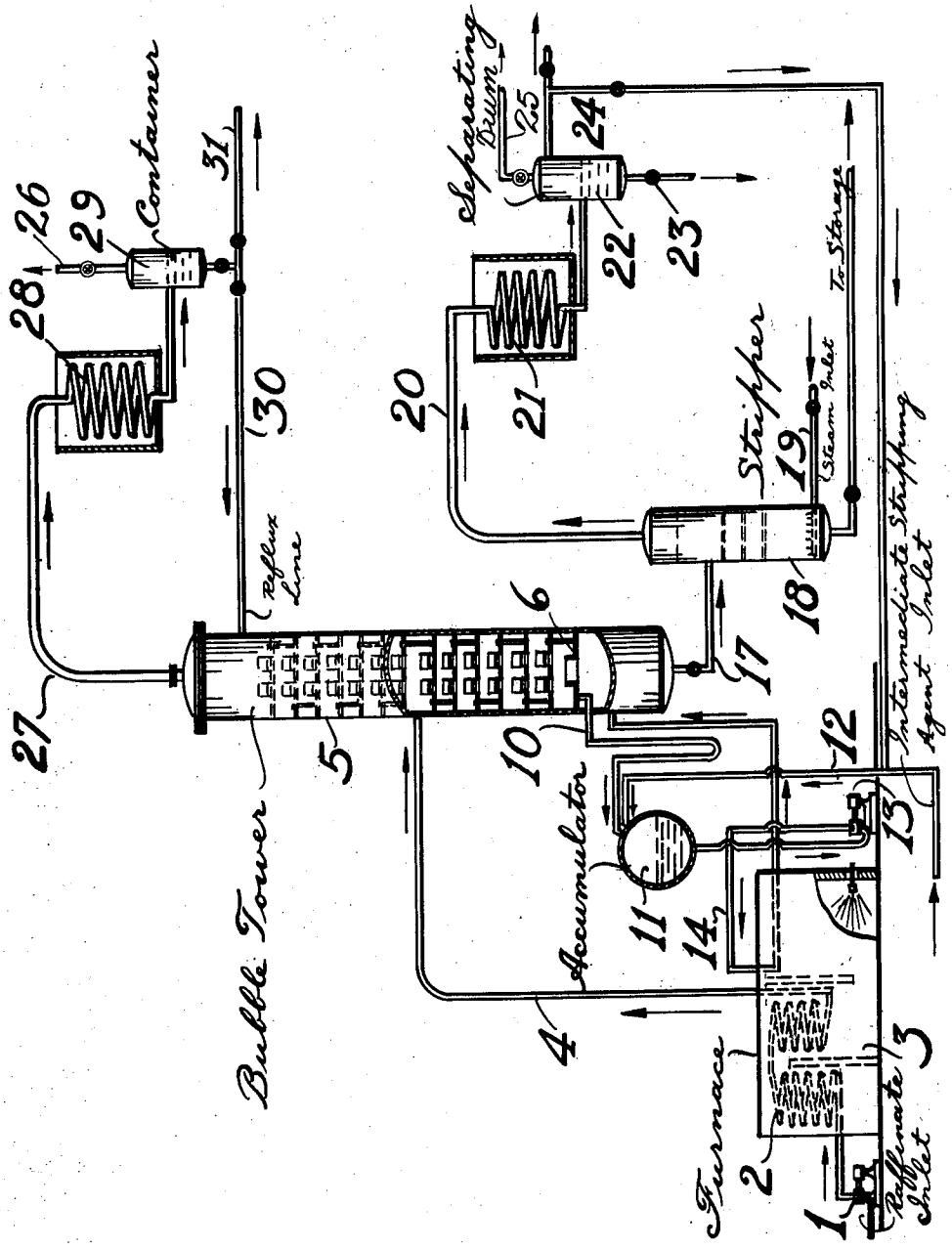

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RECTIFYING PROCESS

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12 Claims. (Cl. 196—94)

This invention relates to improvements in methods of separating relatively high and low boiling compounds from a mutual solution thereof.

It is an object of this invention to separate a mutual solution of two materials of quite widely different boiling points by fractionation so as to obtain one or both of the materials in a substantially pure state as regards contamination by the other material.

Another object of the invention is to accomplish separation of the materials while maintaining the quality of each of the materials.

Other objects will be apparent from the specification and from the accompanying drawing in which latter the figure is a diagrammatic representation of a preferred apparatus for carrying out the invention.

With a preferred embodiment in mind but without intention to limit the invention more than is required by the prior art, the raffinate from a solvent extraction treatment of lubricating oil distillate can be separated into two fractions, namely, substantially pure solvent and a substantially solvent-free petroleum fraction. Such a raffinate consists of approximately 10 percent of the solvent, which may be a pure compound boiling at 300° F., and 90 percent of a petroleum fraction having an initial boiling point of approximately 800° F. It is desired to recover the solvent at least 99 percent pure and to remove all except any negligible trace of the solvent from the lubricating oil fraction.

The raffinate is pumped by means of a pump 1 through a line 2 in a furnace 3. In passing through the furnace 3 the raffinate is heated to a temperature above the boiling point of the solvent and most of the solvent is vaporized. The heated raffinate is passed through a line 4 to an intermediate portion of a bubble tower 5. The bubble tower is provided with a plurality of bell caps and over-flow pipes according to the usual construction. The unvaporized portion of the raffinate flows downwardly through the stripping section of the bubble tower. As the raffinate flows downwardly through the bubble tower more and more of the solvent remaining in the raffinate is vaporized by the rising vapors so that the final over-flow which is caught upon a pan 6 is substantially free of solvent.

The over-flow is delivered from the pan 6 through a line 10 to an accumulator 11. An intermediate stripping agent is introduced into the accumulator 11 through a line 12. The intermediate stripping agent may comprise a narrow petroleum fraction having a boiling range of approximately 450 to 550° F., and constitutes an intermediate boiling fraction. The intermediate boiling fraction or stripping agent and the over-flow from pan 6 constitute a mixture which is pumped by a pump 13 through a line 14 into the radiant section of the furnace 3 where the mixture is heated to a temperature above the boiling point of the stripping agent whereby the major portion of the stripping agent is vaporized and a negligible amount of the high boiling raffinate fraction is vaporized. The partly vaporized discharge from line 14 is released into the bubble tower 5 below the pan 6 whereby the vapors of the stripping agent rise upwardly through the bubble tower and function to strip the low boiling compound from the feed plate over-flow. The bottom reboiling gives a source of substantially solvent free vapors in the lower portion of the bubble tower at temperatures not in excess of approximately 600° F. at atmospheric pressure. This temperature will not injure the lubricating oil fraction.

The liquid which accumulates in the bottom of the bubble tower consists substantially of the high boiling raffinate fraction with a small amount of the stripping agent and a negligible trace of the solvent or low boiling compound. This liquid is removed from the bubble tower at a temperature of approximately 600° F. and may contain a negligible amount of the solvent and perhaps 10 percent of compounds boiling in the range of the intermediate agent. This liquid is removed through a line 17 to a stripper 18 in which substantially all of the residual stripping agent is stripped out by steam which is introduced through a line 19. The over-head vapors from the stripper 18 are withdrawn through a line 20 to a condenser 21. The condensate is introduced into a separating drum 22 from which water is withdrawn through a line 23 while the stripping agent is withdrawn through a line 24 to a suitable place of storage, not shown. A vent line 25 balances the separator to the atmosphere or to a gas collecting system. Instead of stripping the light ends from the bottoms which are withdrawn from tower 5 through line 17 they may be recovered as an over-head cut upon the vacuum re-running of the lubricating oil fraction for finished oils.

The solvent or low boiling compound is withdrawn over-head from the bubble tower through a line 27 to a condenser 28. The condensed solvent is delivered to a container 29. A portion of the condensed solvent is returned through a line 30 to the upper portion of the bubble tower to

function as a refluxing agent. The remainder of the condensate is withdrawn through a line 31 to a place of storage, not shown. A vent line 26 balances the tower to the atmosphere or to a non-condensable collecting system by which the tower pressure is regulated.

An example of low and high boiling materials to be separated according to the present invention is the raffinate obtained in treating lubricating oils with undiluted phenol. The intermediate boiling substance may be in this case a petroleum fraction boiling between 450° to 550° F. When it is desired to separate SO_2 from a heavy petroleum oil obtained after extraction with liquid SO_2, the intermediate boiling substance may be either a naphtha boiling between 300° and 400° F. or it may be benzol or toluol. The solvent dewaxing of lubricating or cylinder oils also leads to solutions of one or more low boiling substances in the heavy oil. Thus, for example, the dewaxing may be carried out with a benzol acetone mixture in which case the light solvent of benzol and acetone is to be separated from the dewaxed oil. A heavy naphtha or a light gas oil may be used in this case as intermediate boiling substance. The same principle may be applied also to cases in which the high boiling material is a compound having a single boiling point.

While the invention has been described as applied in the treatment of raffinate from a solvent extraction treatment or dewaxing of lubricating oil distillate, it will be understood that it is intended to similarly separate compounds from any source having different boiling points. The process is applicable to the separation of pure compounds each having a single boiling point. It is applicable also to a solution of a pure compound and a fraction having a boiling range such that the boiling point of the pure compound lies outside of the boiling range of the fraction. The stripping agent can be a pure compound having a boiling point intermediate the boiling points of the relatively low and high boiling compounds. Similarly the stripping agent can be a material having a boiling range which falls within the temperature gap between the final boiling point of the overhead product and the initial boiling point of the bottoms product. Preferably the boiling point or boiling range of the stripping agent lies below and it preferably does not over-lap or include the boiling point or range of the high boiling compound and it similarly lies above and preferably does not overlap or include the boiling point or range of the low boiling compound. The stripping agent is usually so chosen that its presence in the bottoms product is not undesirable and/or so that it can be satisfactorily stripped out in the bottoms product in the usual manner.

By the process described the compounds are separated from each other by fractionation with a high degree of heat economy. The latent heat imparted to the stripping agent does work in vaporizing the low boiling component. The lowering of the temperature is accomplished by reason of some of the stripping agent dissolving in the bottoms product and thus raising its vapor pressure. The intermediate agent while not affecting the temperature at the top of a bubble tower lowers the temperatures throughout the lower or stripping section of the bubble tower. The effective fractionating pressure is lowered where the total fractionating pressure is atmospheric or is below or above atmospheric. Consequently any danger of undesirable deterioration or loss of quality of one of the compounds is minimized. By way of example such pressures as approximately 12 to 400 pounds per square inch may be employed.

Various changes may be made within the scope of the appended claims in which it is desired to claim all novelty inherent in the invention as broadly as the prior art permits.

I claim:

1. The method of separating a relatively high and a low boiling material from a solution thereof which comprises heating the solution above the boiling point of the lower boiling material, passing the heated solution through a fractionating zone, withdrawing the unvaporized fractions from the fractionating zone and mixing them with a substance having a boiling point between the boiling points of the materials, heating the mixture above the boiling point of the substance, injecting the heated mixture into an accumulating zone below the lower portion of the fractionating zone whereby vapors of the substance rising through the fractionating zone strip the lower boiling material from the higher boiling material, removing vapors of the substance and lower boiling material overhead from the fractionating zone and withdrawing the unvaporized bottoms and substance from the bottom of the accumulating zone.

2. The method of separating a relatively high and a low boiling material from a solution thereof according to claim 1 in which the process is carried out under an absolute pressure between 12 pounds per square inch and 400 pounds per square inch.

3. The method of separating a relatively high and a low boiling material from a solution thereof according to claim 1 in which the two materials of the solution and the intermediate boiling substance comprise three pure compounds, each having a definite boiling point.

4. The method of separating a relatively high and a low boiling material from a solution thereof according to claim 1 in which the two materials of the solution and the intermediate boiling substance comprise three fractions, each having a boiling range.

5. The method of separating a relatively high and a low boiling material from a solution thereof according to claim 1 in which the two materials of the solution and the intermediate boiling substance comprise a pure compound and two petroleum fractions.

6. The method of separating a relatively high and a low boiling material from a solution thereof according to claim 1 in which the two materials of the solution and the intermediate boiling substance comprise a petroleum fraction and two pure compounds.

7. The method of separating relatively high and low boiling materials from a solution thereof according to claim 1, in which one of the materials is a pure compound and has a given boiling point and the other material has a boiling range, and in which the boiling points of the substance and the materials do not over-lap.

8. The method of separating relatively high and low boiling materials from a solution thereof according to claim 1 in which the boiling ranges of the substance and one of the materials overlap.

9. The method of separating a relatively high and a low boiling fraction from a solution thereof, which comprises heating the mixture above the boiling range of the lower boiling fraction,

passing the heated mixture through a fractionating zone, withdrawing the unvaporized fractions from the fractionating zone and mixing them with a substance having a boiling range between the boiling ranges of the fractions, heating the mixture above the boiling range of the substance, injecting the heated mixture into an accumulating zone below the lower portion of the fractionating zone whereby vapors of the substance rising through the fractionating zone strip the lower boiling fraction from the high boiling fraction, removing vapors of the substance and lower boiling fraction over-head from the fractionating zone, and withdrawing the unvaporized bottoms and substance from the bottom of the accumulating zone and stripping the substance from the high boiling fraction.

10. The method of separating a relatively high and a low boiling material from a solution thereof according to claim 9 in which there is a temperature gap between the adjacent limits of the boiling ranges of any pair of the three fractions.

11. The method of separating a relatively high and a relatively low boiling material from a solution thereof, which comprises heating the solution above the boiling point of the lower boiling material, passing the heated solution through a fractionating zone, withdrawing the unvaporized fractions from the fractionating zone and mixing them with a substance having a boiling point between the boiling points of the materials, heating the mixture above the boiling point of the substance, passing vapors from the heated mixture through the fractionating zone whereby vapors of the substance rising through the fractionating zone strip the lower boiling material from the higher boiling material, removing vapors of the lower boiling material overhead from the fractionating zone, and withdrawing the unvaporized bottoms of higher boiling material and substance from the fractionating zone.

12. The method according to claim 11 in which the unvaporized bottoms withdrawn from the fractionating zone is passed through a stripping zone in which the substance is separated from the high boiling material.

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