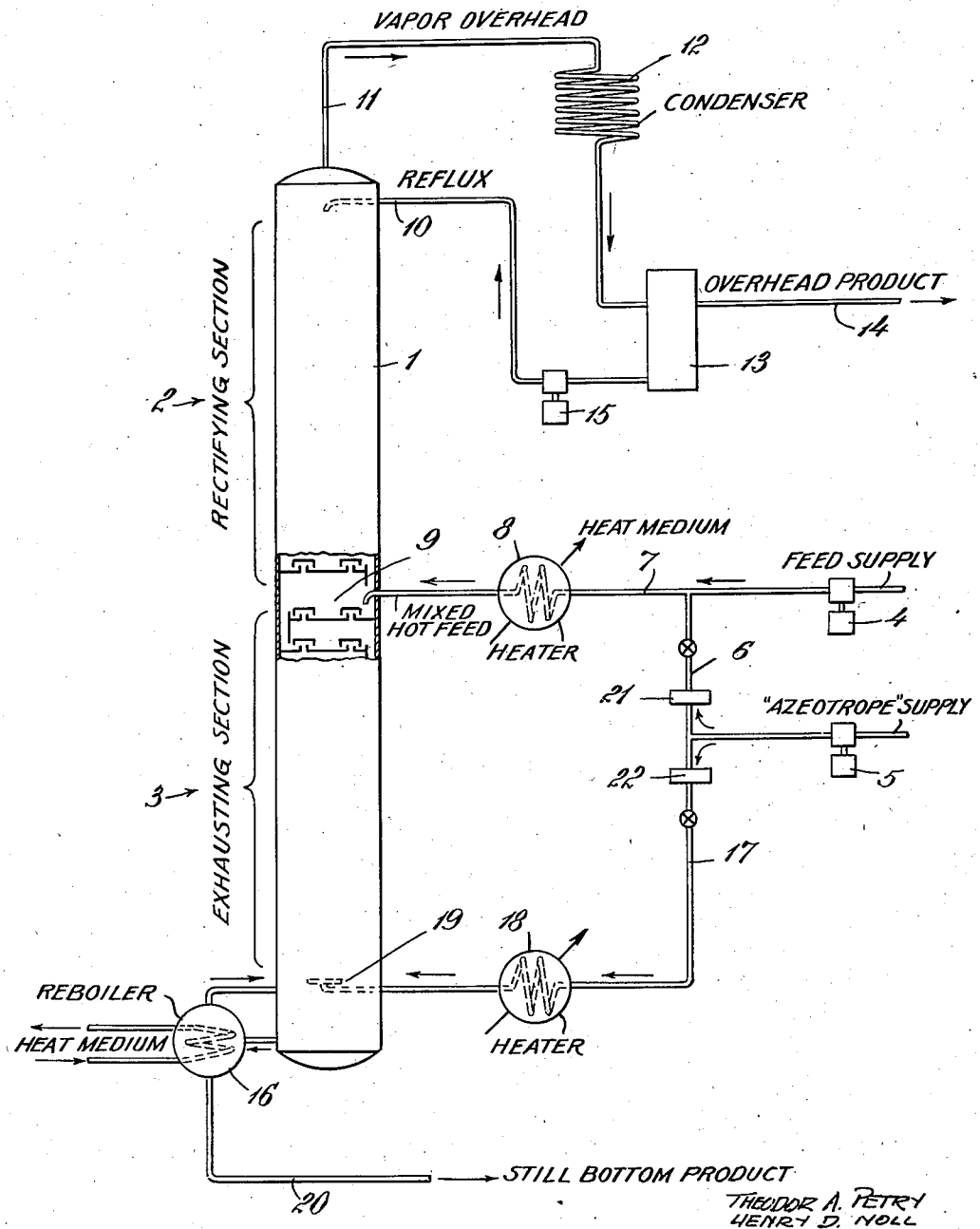
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SEPARATION OF HYDROCARBONS

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THEODOR A. PETRY
HENRY D. NOLL
RUSSELL LEE
INVENTORS

BY Myron J. Burkhard
ATTORNEY

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SEPARATION OF HYDROCARBONS

Theodor A. Petry, Henry D. Noll, and Russell Lee, Wenonah, N. J., assignors to Socony-Vacuum Oil Company, Incorporated, New York, N. Y., a corporation of New York

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4 Claims. (Cl. 202—42)

This invention has to do with the separation of aromatic and non-aromatic hydrocarbons of substantially similar boiling points by methods of azeotropic distillation, wherein the distillation is conducted in the presence of an added material, herein referred to as an azeotropic agent, which forms constant boiling mixtures with the non-aromatic hydrocarbons which constant boiling mixtures boil below the boiling point of the aromatic hydrocarbon or of any constant boiling mixture which may be formed therewith.

In such distillations, the aromatic material is left as a still bottom product. The purity of bottoms product may be increased, by the use of increased amounts of azeotropic agent or higher reflux ratios or both. Increased amounts of azeotropic agent cause losses of still bottom product to overhead which cannot be prevented by the use of high rates of reflux. High rates of reflux are undesirable because they increase the heat load and decrease the capacity of the distillation tower.

This invention has for its object the provision of a method for conducting such azeotropic distillations wherein a lessened use of reflux is permitted with the attainment of a given purity of still bottom product and in which the azeotropic agent addition is under relatively precise control. It has for a major object the use of at least a portion of the azeotropic agent by introducing it to the system in vapor form.

The usual form of process for conducting such azeotropic processes has heretofore been that of introducing a heated feed mixture, consisting of the desired aromatic material, the undesired non-aromatic material, and a predetermined amount of azeotropic agent at some point between the ends of a continuously operating fractionating column, from the bottom of which the desired aromatic material is withdrawn, and from the top of which the constant boiling mixture of non-aromatic and azeotropic agent is withdrawn. The top of the column is refluxed with condensed overhead product, and heat is applied to the exhausting column by means of a reboiler.

This invention is based upon a modification of this system in which a portion of the azeotropic agent is introduced in vapor form into the exhausting column to act as a stripping medium therein.

The invention may be understood by reference to the attached drawing, the single figure of which shows the process in diagrammatic form. In this drawing 1 is a fractionating column adapted for continuous operation consisting of a rectification section 2 and an exhausting section 3. A feed material is brought to the system by pump 4 and an additive or azeotropic material by pump 5. A portion of the azeotropic material passes through pipe 6 to join the feed in pipe 7, the resulting mixture being heated to distillation temperature in heater 8 and introduced to the column at feed level 9. The vapors rising from 9 through the rectification section 2 under the influence of wet reflux introduced at 10 are freed of still bottom product and are withdrawn by pipe 11 and pass through condenser 12 to receiver 13 whence overhead product is withdrawn at 14 and reflux is returned to the column by pump 15. Still bottoms product descending through the exhausting section 3 is freed of overhead product by the joint action of the heat introduced through reboiler 16 and of azeotropic agent in vapor form derived from pipe 17, vaporized in heater 18 and introduced as vapor at 19. Still bottom product is withdrawn by pipe 20.

This form of operation has the decided advantage over the previous operation in that the presence of azeotropic agent in vapor form in the exhausting column enables a greater degree of separation of still overhead product from still bottom product with the same overall amount of azeotropic material being used in the system without increase in reflux ratio. A further very significant advantage rises from the very precise control over the amount of azeotropic agent which is introduced to the system. Since the overall efficiency of the system depends upon rather precise control of this amount, it may be quite conveniently achieved here by a constant setting of volume passed at control point 21 in pipe 6 and a variable control responsive to variations in the system, either manual or automatic, at control point 22 acting upon a lesser amount of azeotropic agent, say on about 10% of the total.

Referring specifically to the separation of toluene from a close cut fraction of petroleum origin, in the presence of methanol, to which this system of operation is quite adapted, and assuming a feed of about 45% concentration of toluene, for 100 volumes of feed, about 60–80 volumes of methanol, total, would be utilized, of which about 6–8 volumes or less would be passed into the bottom of the column as vapor. In a column of about 24 theoretical plate efficiency, the operation would produce as a still bottom product toluene of about 98% purity, 90% of the available toluene being recovered, with a reflux ratio of less than 7:1 based on total hydrocarbon feed.

In this specification and the claims, the term "constant boiling mixture" is made use of in full recognition of the fact that the non-aromatic hydrocarbons so removed are themselves a mixture and when considered from the standpoint of precision the term "constant boiling" is inexact. However, for any commercial distillation, and particularly for a continuously operating commercial distillation, the variations are sufficiently small that the material may be readily handled from the design and operating standpoint as a constant boiling mixture, and the term is used with that understanding.

Since with hydrocarbon fractions having low aromatic contents it is frequently desirable to conduct the azeotropic distillation in two steps, recovering as a still bottom from the first step a fraction more concentrated in aromatic and as a still bottom from the second step the desired aromatic in a condition of relatively high purity, the distillation conditions in the two steps being similar except for the necessary variations in azeotropic agent amount, etc., dependent upon the different concentrations of aromatic in the feed the invention is to be understood as being applicable equally to such two-step azeotropic distillation operations.

We claim:

1. That method of conducting a distillation separation of an aromatic hydrocarbon material from non-aromatic hydrocarbons of substantially similar boiling point in the presence of an added material which forms constant boiling mixtures with the non-aromatic hydrocarbons which constant boiling mixtures boil below the aromatic hydrocarbon or any constant boiling mixture formed therewith which comprises heating a feed stock comprising the aromatic and non-aromatic hydrocarbons to distillation temperature, introducing said feed to a continuously operating fractionating column between the ends thereof, adding a portion of the said added material to the feed prior to heating, and adding the remainder of the said added material in vapor form near the bottom of the exhausting section of said fractionating column.

2. That method of conducting a distillation separation of an aromatic hydrocarbon material from non-aromatic hydrocarbons of substantially similar boiling point in the presence of an added material which forms constant boiling mixtures with the non-aromatic hydrocarbons which constant boiling mixtures boil below the aromatic hydrocarbon or any constant boiling mixture formed therewith which comprises heating a feed stock comprising the aromatic and non-aromatic hydrocarbons to distillation temperature, introducing said feed to a continuously operating fractionating column between the ends thereof, adding a major portion of the added material to the feed prior to heating, and adding the remainder of the said added material in vapor form near the bottom of the exhausting section of said fractionating column.

3. That method of separating substantially pure toluene from a close cut petroleum fraction containing the same which comprises heating the petroleum fraction to distillation temperature and introducing it to a continuously working fractionating column between the rectifying and exhausting sections thereof, and supplying methanol to the distillation system in sufficient quantity to effect a separation of non-aromatics from toluene, in which a major portion of the methanol is introduced into the petroleum fraction prior to heating thereof, and a minor portion of the methanol is introduced in vapor form near the bottom of the exhausting section of said column.

4. That method of conducting a distillation separation of an aromatic hydrocarbon material from non-aromatic hydrocarbons of substantially similar boiling point in the presence of an added material having the azeotropic distillation characteristics of methanol under the conditions of operation appropriate for toluol recovery, which forms constant boiling mixtures with the non-aromatic hydrocarbons which constant boiling mixtures boil below the aromatic hydrocarbon or any constant boiling mixture formed therewith, which comprises heating a feed stock comprising the aromatic and non-aromatic hydrocarbons to distillation temperature, introducing said feed to a continuously operating fractionating column between the ends thereof, adding a portion of the said added material to the feed prior to heating, and adding the remainder of the said added material in vapor form near the bottom of the exhausting section of said fractionating column.

THEODOR A. PETRY.
HENRY D. NOLL.
RUSSELL LEE.