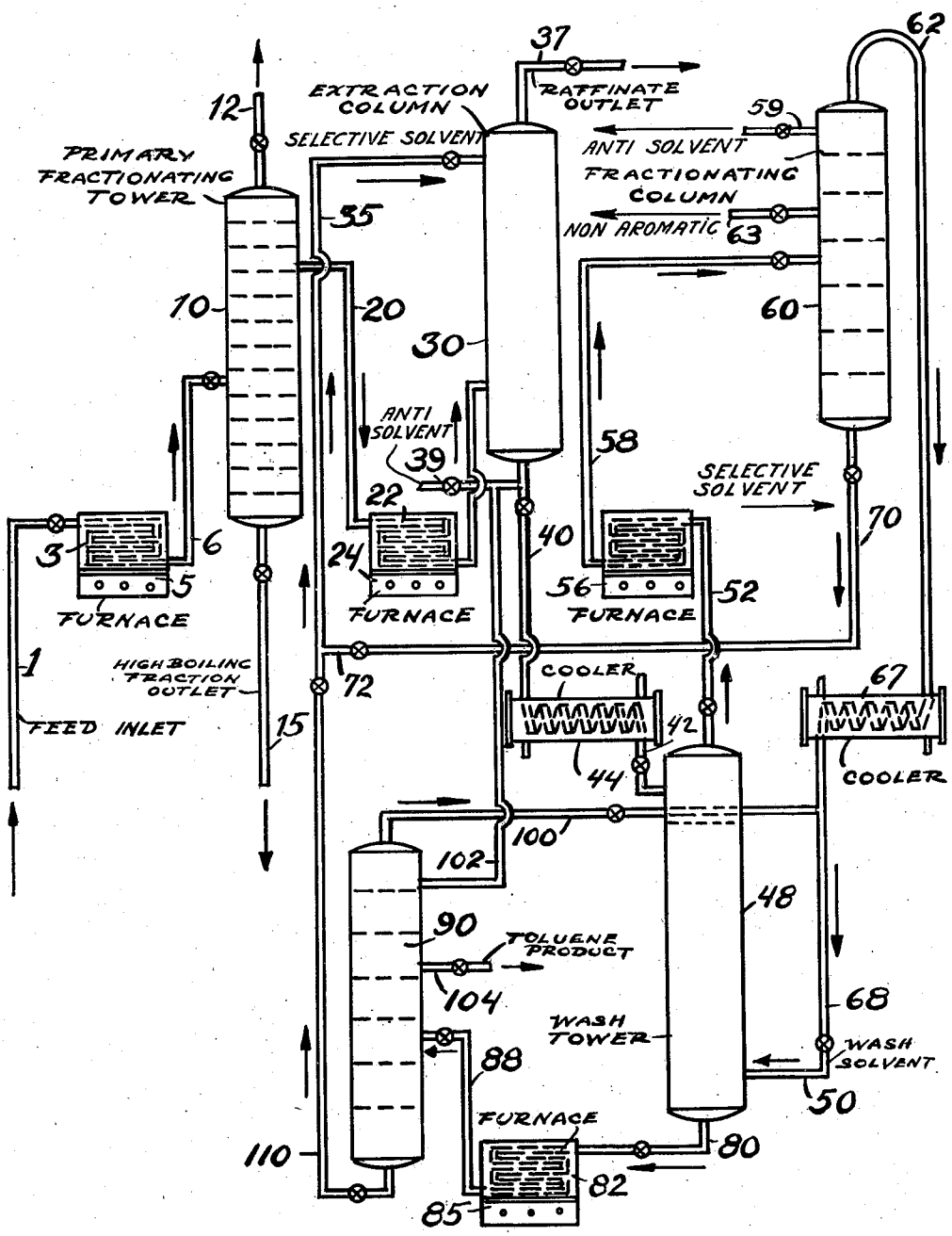
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VAPOR PHASE EXTRACTION

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VAPOR PHASE EXTRACTION

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The present invention relates to improvements in the art of extracting desirable hydrocarbons from a mixture thereof, and, more particularly, it relates to the fractionation of a relatively wide boiling range hydrocarbon fraction by vapor phase extraction to separate the individual types of components and to obtain high yields of each component in a relatively pure state.

Heretofore, in the ordinary vapor phase extraction of wide boiling range fractions, it has been possible to obtain one of the components in a fairly pure state with limited yield or to obtain a fairly high yield of this component with limited purity, but it was not possible to obtain both a high yield and high purity by vapor phase extraction alone. For example, in the recovery of toluene from a hydrocarbon fraction which had been subjected to reforming in the presence of hydrogen and a catalyst, it was not possible to obtain toluene by vapor phase extraction in a form sufficiently pure for use in nitration.

In order to concentrate substantially all of the toluene contained in a wide boiling stock by distillation in commercially available equipment, it is necessary to cut a fraction of approximately 50° F. boiling range (200° F.–250° F.). Vapor phase extraction of this cut will give products containing approximately 90% of the total toluene in 80–85% purity or an 80–85% yield of 90–95% purity. The associated hydrocarbons are, for the most part, olefins having boiling points in the range of 220° F.–250° F. and paraffins having boiling points generally higher than that of toluene. The olefins may be removed by acid-treating with associated relatively high losses of toluene. Total removal of paraffins can be accomplished only by an extremely effective distillation operation using equipment not generally available for commercial use. A portion of the toluene associated with the paraffin cuts may be recovered by recycling.

If the vapor phase extraction process is operated to take overhead only paraffins and the resulting hydrocarbons remaining in the extract phase are then stripped of solvent and subjected to a second vapor phase extraction, little improvement in purity of the aromatic phase is obtained. This phenomenon is due to the fact that the higher boiling paraffins and the olefins have approximately the same vapor pressure in the presence of the selective solvent as does the toluene. By subjecting the hydrocarbon material to alternate vapor phase extraction and distillation with recycling of the intermediate products, it would be possible to obtain substantially all of the toluene in a fairly pure state but an extremely complicated separating procedure would be necessary.

According to my present invention, I secure high yields of the desired component, such as toluene, in a high state of purity by a process which involves in brief compass distilling a wide boiling range feed stock and recovering therefrom a fraction containing substantially all of the desired hydrocarbon, such as toluene. This fraction is then subjected to vapor phase extraction to obtain the maximum yield of non-aromatic hydrocarbons overhead. The extract phase, which may be admixed with an added anti-solvent, is thereafter cooled and subjected to countercurrent washing with a paraffinic solvent having a boiling range different from that of the desired product and also from the higher boiling selective solvent. This washing operation is regulated so that practically all of the non-aromatic hydrocarbons initially present in the extract phase are washed out along with a very small amount of aromatics. The resulting extract phase is then distilled to separate the desired components, such as the toluene, from the wash solvent, the anti-solvent and the high boiling selective solvent.

The main object of my present invention, therefore, is to provide selective solvent means for recovering from a mixture of hydrocarbons, a desired component in high yield and possessing a high degree of purity.

A more specific object of my present invention is to solvent extract a mixture of hydrocarbons to secure a desired component in high yields and of a high degree of purity, in a manner effecting savings in equipment and utilities.

Other and further objects of my present invention will appear from the following more detailed description and claims.

The accompanying drawing shows diagrammatically an arrangement of apparatus elements in which a preferred modification of my invention may be carried into practical effect. I shall now set forth a specific example describing a preferred modification of my invention and in so doing I shall refer to the accompanying drawing for a better understanding of my invention.

Referring in detail, therefore, to the drawing, a hydrocarbon feed stock containing a quantity of toluene and having a wide boiling range, say a steam-cracked naphtha fraction containing toluene, is charged into the system thru line 1 and thence discharged into a fired coil 3 disposed in a furnace 5 where the fraction is heated to vaporize the charging oil and thence with-

drawn thru line 6 and discharged into a primary fractionating column 10. From fractionating column 10 a low boiling fraction is withdrawn thru line 12 and a high boiling fraction is withdrawn thru line 15. These fractions are rejected from the present system. A middle fraction boiling within the range of from 180° F. to 260° F. is withdrawn thru line 20 and heated in a fired coil 22 disposed in a furnace 24 to a temperature of approximately 220° F. to substantial vaporization, and thence discharged into extraction column 30 where it is treated with a solvent, such as phenol discharged from line 35 into tower 30. A raffinate phase is recovered from column 30 thru line 37. This raffinate phase contains substantially no aromatics but consists essentially of paraffins and olefins plus a small amount of solvent, and is processed to recover the solvent which is returned to the system in equipment not shown.

An extract phase is withdrawn from column 30 thru line 40 where it may be admixed with an anti-solvent, such as water, up to 20% of the volume of phenol. This anti-solvent is added thru line 39 and is added for the purpose of reducing the solubility of non-aromatics in the phenol. Thence the mixture is discharged into a cooler 44 where it is cooled to a temperature of 60° F.–120° F. and thence discharged thru line 42 into a wash tower 48 where it is washed with liquid pentane fed into said tower thru line 50. An overhead fraction, comprising the liquid pentane with a small amount of solvent, non-aromatic hydrocarbons and minor amounts of toluene from tower 48, is withdrawn overhead thru line 52 and charged to a furnace 56 where it is heated to partial vaporization (around 400° F.–600° F.), then withdrawn thru line 58 and discharged into a fractionating column 60 from which three overhead fractions are recovered as follows: first, a wash solvent fraction which is withdrawn thru line 62, cooled in a cooler 67, and returned thru lines 68 and 50 to wash tower 48; a side stream fraction consisting of the anti-solvent which is withdrawn thru line 59 and is returned by means not shown to line 39 and hence to tower 30; a side stream fraction consisting of non-aromatic together with a small amount of aromatic hydrocarbons is withdrawn thru line 63; this fraction is normally rejected from the system but may be recycled to line 1 for further treatment; and a bottoms fraction which is withdrawn thru line 70, the said bottoms fraction comprising the solvent which is recycled thru lines 72 and 35 to extraction tower 30. Where it is desired to recover in pure form, one or more of the non-aromatic hydrocarbons contained in the feed to the vapor phase extraction, the material withdrawn thru line 63 may be combined with the vapor phase raffinate line 37 and the resulting mixture is then subjected to vapor phase extraction. The resulting extract phase may be processed by washing in the liquid phase as previously described.

The washed extract phase is withdrawn from wash tower 48 thru line 80, thence heated in a coil 82 disposed in a furnace 85, thence withdrawn thru line 88 and discharged into a fractionating column 90 from which wash solvent is recovered thru line 100 and recycled thru lines 68 and 50 to wash tower 48. A side stream comprising the anti-solvent is withdrawn thru line 102 and recycled to line 39 for further use in the wash column 48. Toluene is recovered as a side stream thru line 104. The solvent is recovered from the bottom of still 90 thru line 110 and recycled to line 35 and thence into extraction tower 30.

The toluene recovered from line 104 is usually of satisfactory quality, but in order to meet the rigid requirements for nitration grade material, it may be processed by distilling and acid-treating to remove a small amount of contaminants in a known manner in equipment not shown.

In the foregoing specific example given I have illustrated the invention as applied to a steam-cracked naphtha for toluene recovery. My invention is applicable to any feed stock containing toluene. Other aromatics may be recovered in a similar manner from suitable feed stocks. Furthermore, my invention includes the recovery of other hydrocarbons from mixtures thereof, such as the recovery of butadiene, from a C_4 hydrocarbon mixture. In the example given, phenol is the aromatic selective solvent and H_2O is employed as the anti-solvent. Other selective solvents include furfural, cresylic acid, etc. Other anti-solvents include low molecular weight aliphatic alcohols including polyhydric alcohols.

Good results are obtained in recovering toluene from hydrocarbon oils by operating in the following range of conditions in the extraction zone:

Proportion of phenol	100 to 300 volume per cent of feed
Temperature in extraction tower	Feed inlet—220° F. Tower top—210° F. Tower bottom—320° F.
Pressure	Atmospheric

In the wash tower 5–15 vol. per cent of H_2O based on the phenol is employed.

The amount of wash solvent is 50 to 200 volume per cent of the volume of extract. The temperature in this tower may be from 70° F. to 100° F.

I claim:

1. The method of recovering desired aromatic hydrocarbon components from a naphtha containing said aromatic components and non-aromatic components which comprises vaporizing said naphtha, fractionating the vapors, recovering a naphtha fraction containing said desired aromatic components and having a boiling range spreading over up to about 80° F., contacting said fraction substantially vaporized with about 100% to 300% by volume of a liquid selective solvent for the desired aromatic components at temperatures ranging from about 210° F. to about 320° F. to produce an extract phase containing said desired aromatic components together with about 5% to 10% of non-aromatic components and a vaporous raffinate, separating said extract phase from said vaporous raffinate, adding about 5% to 15% by volume based on the amount of selective solvent of an antisolvent to said extract phase, contacting said extract phase with about 50% to 200% by volume of a paraffinic wash solvent for said non-aromatic components in a washing zone at a temperature of about 70° to 100° F. to wash out of said extract phase practically all of said non-aromatic components together with a minor amount of the desired aromatic components, said wash solvent having a boiling range different from that of said desired aromatic components, withdrawing said extract phase from said washing zone and recovering said desired aromatic components from said extract phase by distillation.

2. The method set forth in claim 1 in which the selective solvent is phenol and in which proc-

ess water is added to the extract phase recovered from the extraction zone prior to the washing step.

3. The method set forth in claim 1 in which the wash solvent is pentane.

4. The method of recovering toluene from a naphtha containing toluene and non-aromatic components which comprises obtaining a naphtha fraction boiling within the range of from about 180° F. to about 260° F., vaporizing said fraction, contacting said vaporied fraction with about 100% to 300% by volume of a liquid selective solvent for toluene at a temperature ranging from about 210° F. to about 320° F. under such conditions as produce a liquid extract phase containing toluene together with about 5% to 10% non-aromatic components and a vaporous raffinate, separating said extract phase from said vaporous raffinate, adding an antisolvent to said extract phase, contacting said extract phase with about 50% to 200% by volume of a paraffinic wash solvent in a washing one at a temperature of about 70° F. to 100° F., in such a manner that practically all of the non-aromatic components together with a minor amount of toluene are washed out of said extract phase, said wash solvent having a boiling range different from that of toluene, withdrawing said extract phase from said washing zone and recovering toluene therefrom by distillation.

5. The method set forth in claim 4 in which the selective solvent is phenol in liquid form.

6. The method set forth in claim 4 in which the selective solvent is liquid phenol and in which process water is added to the phenol withdrawn from the extraction zone which water serves as an anti-solvent, and thereafter the mixture of anti-solvent and phenol extract is discharged into the washing zone.

7. The method set forth in claim 4 in which the wash solvent is pentane.

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