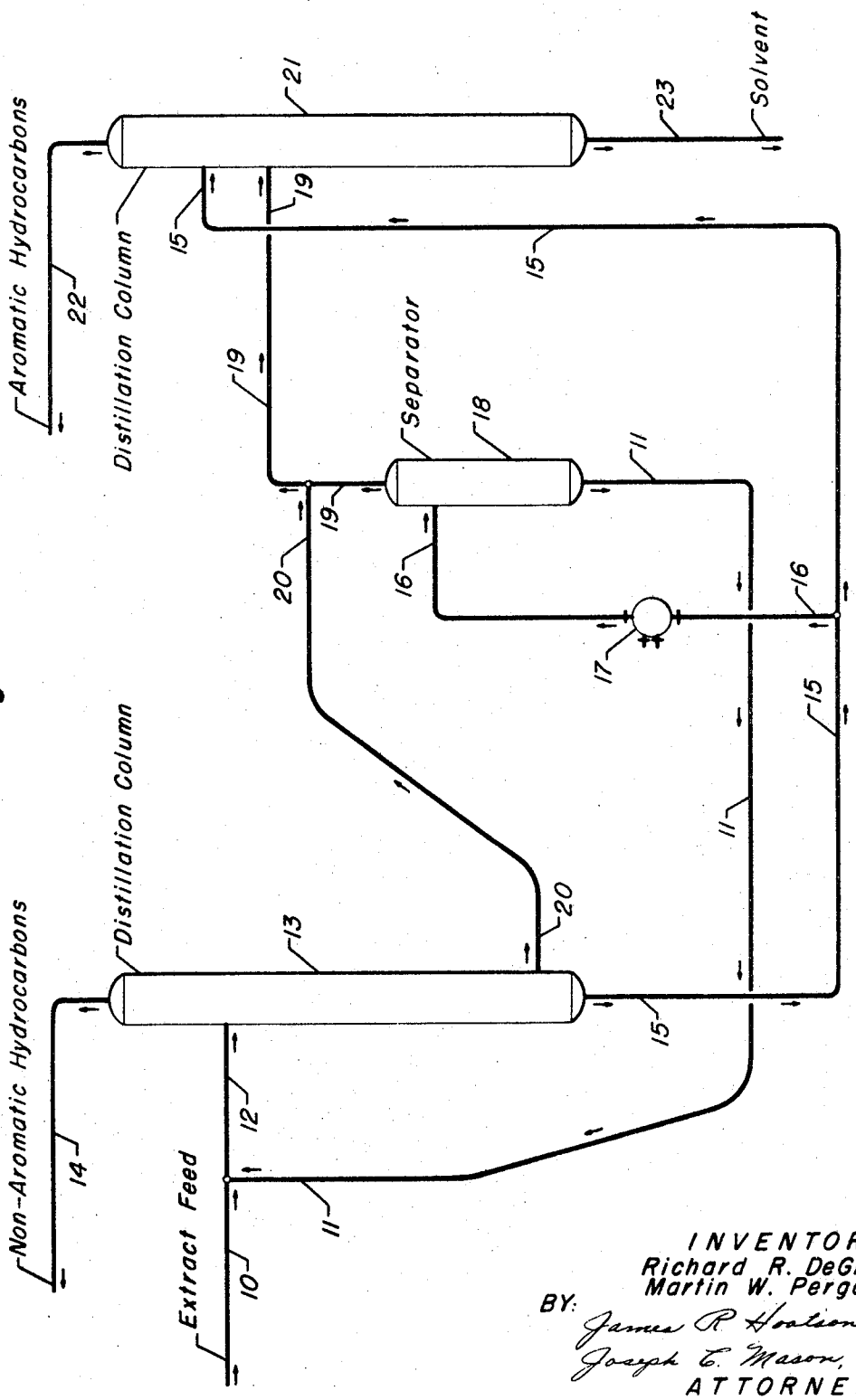
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SOLVENT EXTRACTION OF AROMATIC HYDROCARBONS

Filed May 29, 1967 2 Sheets-Sheet 1

Figure 1



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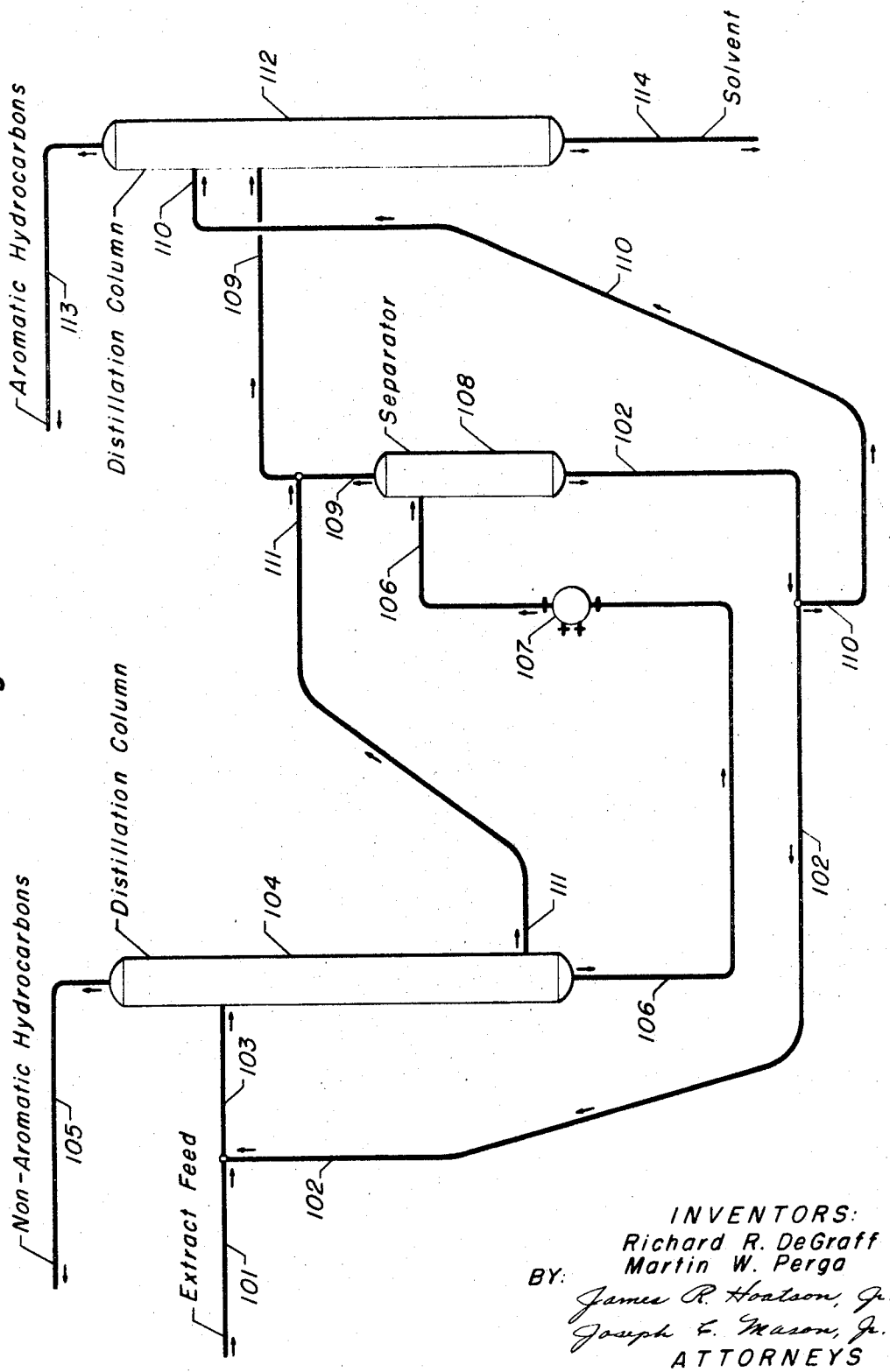
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SOLVENT EXTRACTION OF AROMATIC HYDROCARBONS

Filed May 29, 1967 2 Sheets-Sheet 2

Figure 2



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United States Patent Office 3,466,344 Patented Sept. 9, 1969

3,466,344

SOLVENT EXTRACTION OF AROMATIC HYDROCARBONS

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Filed May 29, 1967, Ser. No. 641,771
Int. Cl. C07c 15/00, 7/08
U.S. Cl. 260—674 10 Claims

ABSTRACT OF THE DISCLOSURE

Method for fractionating the extract phase from an aromatic solvent extraction operation. The extract is subjected to extractive distillation followed by separate distillation of the solvent-aromatic stream for recovery of the aromatic hydrocarbons. A preflash of the solvent-aromatic stream prior to the aromatic recovery column is effectively utilized.

Background of the invention

This invention relates to the recovery of aromatic hydrocarbons via the solvent extraction of aromatic components from a suitable feedstock. It particularly relates to the recovery of aromatic hydrocarbons from the extract phase from such a solvent extraction operation. It specifically relates to the distillation of a solvent phase, such as sulfolane, having aromatic hydrocarbons dissolved therein in order to recover the aromatic hydrocarbons such as benzene, toluene, and xylene therefrom.

It is known in the art that a conventional process for the recovery of high purity aromatic hydrocarbons of, say, nitration grade from various feedstocks including catalytic reformates is liquid-liquid extraction utilizing a solvent such as diethylene glycol or sulfolane, each of which has high selectivity for the aromatic hydrocarbon components contained in the feedstock. Typically, in the practice of such prior art process, a hydrocarbon feed mixture is contacted in an extraction zone with an aqueous solvent composition which selectively dissolves the aromatic component of the hydrocarbon feedstock thereby forming a raffinate phase comprising one or more non-aromatic hydrocarbons and an extract phase containing dissolved aromatic components. The extract phase is then separately distilled yielding an overhead distillate containing only a portion of the extracted aromatic component, a sidecut fraction comprising aromatic hydrocarbons, and a bottoms fraction comprising lean solvent suitable for reuse in the extraction zone. Frequently, to prevent losses of the solvent, the raffinate phase is washed with water in a washing zone in order to remove solvent from the raffinite phase.

Also not infrequently, the extract phase is subjected to extractive distillation in order to remove a contaminating quantity of non-aromatic hydrocarbons from the extract phase. This extractive distillation operation is normally performed in order to make possible the recovery of nitration grade aromatic hydrocarbons such as benzene and toluene. Therefore, a typical prior art process for the recovery of aromatic hydrocarbons encompasses a solvent extraction step, an extractive distillation step, and a final distillation step for the recovery of high purity aromatic hydrocarbons from the solvent phase.

In the extractive distillation operation it is common practice to add to the extract phase considerable quantities of additional solvent so that the relative volatilities between the non-aromatic hydrocarbon components and aromatic hydrocarbon components are substantially increased in order to effectuate an almost complete separation between the two via distillation. This, of course, requires a distillation column of some complexity utilizing large quantities of utilities such as steam for heat input in order to properly perform the distillation. However, the extractive distillation column is severely limited in the amount of heat input which is possible because care must be taken to minimize the quantity of aromatics in the overhead fraction which would represent a loss in yield of desirable aromatic hydrocarbons by virtue of adding inefficiencies to the extraction operation to which the overhead stream is normally returned. Accordingly, the extractive distillation operation achieves a balance between the desire to remove non-aromatic hydrocarbons from the aromatic hydrocarbons and the desire to maximize the recovery of the aromatic hydrocarbons.

In similar fashion, the operation of the aromatic recovery column is one of achieving proper thermal balance. It would be desirable to have the feed to the aromatic recovery column as high as possible so that a minimum amount of reboiler heat need be added to the column. It is also desirable to control the temperature of the feed since relatively small changes in temperature have a large effect on the heat balance of the column.

Summary of the invention

It is therefore an object of this invention to provide a method for the recovery of aromatic hydrocarbons from the extract phase of a solvent extraction operation in a more facile and economical manner.

It is another object of this invention to provide a method for fractionating a sulfolane solvent having dissolved therein aromatic and non-aromatic hydrocarbons in a facile and economical manner.

Thus, according to one embodiment of this invention there is provided a method for recovering aromatic hydrocarbons from the extract phase from a solvent extraction zone which comprises the steps of: (a) introducing an extract phase containing solvent having aromatic hydrocarbons dissolved therein, said extract also contaminated with non-aromatic hydrocarbons, into a first distillation zone in the presence of a hereinafter specified added solvent stream; (b) withdrawing from said first zone an upper stream comprising said contaminants and a lower stream comprising solvent and aromatic hydrocarbons; (c) passing a portion of said lower stream into a separation zone maintained under conditions sufficient to produce a vapor fraction containing aromatic hydrocarbons and a liquid fraction comprising solvent; (d) introducing said vapor fraction and the other portion of said lower stream into a second distillation zone under conditions sufficient to produce a distillate fraction comprising aromatic hydrocarbons and a bottoms fraction comprising solvent suitable for reuse in said extraction zone; and (e) returning said liquid fraction of Step (c) to Step (a) as said added solvent.

Another embodiment of the invention includes the method hereinabove wherein said solvent comprises sulfolane.

Still another embodiment of this invention includes the method hereinabove wherein a vapor intermediate sidecut stream containing aromatic hydrocarbons is withdrawn from said first distallation zone, admixed with said vapor fraction of Step (c), and passed together into said second distillation zone as specified.

It is noted from the hereinabove brief description of the present invention, relative to the prior art, that significant economies of operation are achieved by the novel and simple expedient of placing a separation means between the extractive distillation column and the aromatic recovery column. The function of the separation means is to remove a considerable portion of the solvent from

having to be stripped and/or distilled in the aromatic recovery column. This means that the aromatic recovery column may be smaller thereby requiring less capital monies for equipping the plant. In addition, the introduction of aromatic hydrocarbons in vapor phase and, in particular, the utilization of the preferred expedient of withdrawing a vapor sidecut from the extractive distillation column, permits a significant increase in the temperature of the material being charged to the aromatic recovery column. Here again the result is to require less reboiler heat and less equipment for the aromatic recoverey column, and to require the introduction of less stripping steam therein which in effect reduces the column size requirements to effectuate the same recovery of high purity hydrocarbons.

The hydrocarbon feed mixture which may be separated by the improved method of the present invention comprises many different aromatic-nonaromatic mixtures. Typically, feedstocks applicable to the solvent extraction technique include hydrocarbon distillate fractions (usually boiling within or near the gasoline boiling range) of natural gasoline or straight-run petroleum distillates and especially comprises reformed naphthas which are rich in aromatic hydrocarbons and which are particularly valuable as a source of such mononuclear aromatic hydrocarbons as benzene, toluene, and xylene. Thus, the desired aromatic hydrocarbons may comprise benzene; toluene; benzene and toluene; toluene and xylene; and benzene, toluene and xylene. In each case, however, it is understood that the feedstock contains nonaromatic hydrocarbons as well as aromatic hydrocarbons. The process of the present invention is specifically directed to the separation of hydrocarbon feed mixtures comprising benzene and toluene and to feed mixtures comprising toluene and xylene as the aromatic hydrocarbon. Typically, the feedstocks for the present invention as charged to the aromatic extraction step will contain from about 30% to about 60% by weight aromatic hydrocarbons; although, aromatic hydrocarbon concentrations as high as 95% by weight may be used in some cases. The quantity of added solvent is directly proportional to the aromatic content of feed.

Solvent compositions which may be utilized in the practice of the present invention are those selected from the classes which have high selectivity for aromatic hydrocarbons. These aromatic selective solvents generally contain one or more organic compounds containing in their molecule at least one polar group such as a hydroxyl, amino, cyano, carboxyl, or nitro radical. In order to be effective, the organic compounds of the solvent composition having the polar radical must have a boiling point substantially greater than the boiling point of water which, preferably, is included in the solvent composition for enhancing its selectivity; and, in general, must also have a boiling point substantially greater than the end boiling point of the aromatic component to be extracted from the hydrocarbon feed mixture.

Organic compounds suitable for use as part of the solvent composition preferably are selected from the group of those organic containing compounds which include the aliphatic and cyclic alcohols, cyclic monomeric sulfones, the glycols and glycol ethers, as well as the glycol esters and glycol ether esters. The mono- and poly-alkylene glycols in which the alkylene group contains from 2 to 3 carbon atoms such as ethylene glycol, diethylene glycol, triethylene glycol and tetraethylene glycol, propylene glycol, dipropylene glycol, and tripropylene glycol, as well as the methyl, ethyl, propyl, and butyl ethers of the glycol hydroxyl groups, and the acetic acid esters thereof, constitute a particularly preferred class of organic solvents useful in admixture with water as the solvent composition for use in the present invention. An illustrative glycol comprises triethylene glycol.

Additionally, excellent results may be obtained utilizing the cyclic monomeric sulfone, such as tetrahydrothiophene-1,1-dioxide. Still further, an organic compound particularly useful in the practice of this invention is a sulfolane which may be made by condensing a conjugated diolefin with sulfur dioxide and then subjecting the resulting product to hydrogenation, alkylation, hydration and/or other substitution or addition reactions. Typically, organic compounds belonging to the sulfolane class are 2-sulfolene, 2-methyl sulfolane, 2,4-dimethylsulfolane, 2,4-dimethyl-4-sulfolane, methyl-3-sulfonyl ether, ethyl-3-sulfonyl sulfide, and others.

The preferred organic compounds which are utilized in the solvent composition of the present invention are admixed with from 2% to 25% by weight water. Thus, a preferred solvent composition comprises 75% to 98% by weight polyalkylene glycol, e.g. triethylene glycol, and 2% to 25% by weight water.

The apparatus embodied in the practice of the present invention may be any conventional or convenient type known to those skilled in the art. Also, the operating conditions suitable for the practice of this invention are conventional and well known to those skilled in the art with the exception of the precise temperatures and pressures for operating the extractive distillation column and the aromatic recovery column according to the teachings of this invention. In any event, from the teachings presented herein and from a general knowledge of the art, those skilled in the art will be able to choose the proper operating conditions to achieve the benefits ascribed to the practice of the present invention. The amount of solvent composition utilized in admixture with an appropriate feedstock should be at least sufficient to dissolve the constituent to be extracted. It may be desirable to use a considerable excess over the theoretical amount of solvent composition necessary especially when maximum purity and maximum recovery of aromatic hydrocarbons are desired. Usually, in the extraction step the solvent composition to feed ratios will range from about 1:1 to about 20:1 by volume, preferably, from about 5:1 to about 15:1 by volume. A summary of the conditions necessary for the practice of the sulfolane solvent type of operation may be found in Petroleum Refiner, volume 38, No. 9, September 1959, pages 185–192, the entire disclosure of which is incorporated herein by reference.

The solvent extraction step as previously mentioned is well known and may utilize apparatus of any type suitable for effecting counter-current contact between two liquid phases at least partially, but not wholly miscible with each other and wherein the relatively more dense solvent may be brought into intimate contact with the relatively less dense hydrocarbon phase. Thus, the extraction zone which produces the solvent extract which is used as feedstock to the practice of the present invention may comprise a packed column or may contain a series of horizontal plates through which the liquid solvent flows in dispersed form and in countercurrent flow relationship to the ascending hydrocarbon stream.

Description of the drawing

FIGURE 1 is a schematic representation of apparatus for practicing one embodiment of the invention wherein only a portion of the extractive distillation column bottoms is passed into the separation zone.

FIGURE 2 is a schematic representation of apparatus for practicing another embodiment of the invention wherein the entire bottoms stream from the extractive distillation column is passed into the separation zone.

With reference to FIGURE 1, an extract phase from a conventional solvent extraction zone is passed into the system via line **10**. The extract phase has aromatic hydrocarbons dissolved therein and due to inherent inefficiencies of any commercial solvent extractive step this extract phase is also contaminated with a small amount of nonaromatic hydrocarbons. The extract feed stream in line **10** is mixed with an added solvent stream from line **11**,

the source of which is more fully described hereinafter. The combined extract feed stream plus added solvent is passed via line **12** into extractive distillation column **13** which is maintained under distillation conditions.

Operating conditions in distillaton column **13** are conventional (and dependent on the composition of the feed to the column) in that sufficient heat must be added to the column in order for a separation to take place between the non-aromatic hydrocarbons and the aromatic hydrocarbons dissolved in the solvent. Typical operating conditions for distillation column **13**, suitable when using a sulfolane-type solvent, include a pressure from 90 mm. Hg to 15 p.s.i.g., an overhead temperature from 140° F. to 330° F., and a bottoms temperature from 170° F. to 355° F., typically about 350° F.

Operating under these conditions a non-aromatic hydrocarbon upper stream is withdrawn from column **13** via line **14**. A lower stream comprising solvent and aromatic hydrocarbons is withdrawn, preferably, from the bottom of column **13** via line **15** and passed at least in part into separator **18** via line **16**, preferably through heat exchanger **17** wherein, additional heat is added to this fraction prior to entering separator **18**. The remainder of the lower stream comprising solvent and aromatic hydrocarbons is passed in continuing line **15** into distillation column **21**, generally, at an upper location therein.

Separator **18** is maintained under conditions sufficient to produce a vapor fraction in line **19** containing aromatic hydrocarbons and a liquid fraction in line **11** comprising solvent having a reduced aromatic hydrocarbon content. As previously mentioned, the liquid fraction comprising significantly denuded solvent is returned via line **11** into admixture with the incoming extract feed from line **10**.

The vapor fraction containing aromatic hydrocarbons is passed in continuing line **19** into distillation column **21**, preferably, at a locus below the locus selected for the liquid solvent stream entering distillation column **21** from line **15**. In some cases, it may be preferable to introduce this vapor fraction at a locus above the liquid solvent locus or to admix these streams and feed them together into column **21**.

Operating conditions for separator **18** include a temperature from 170° F. to 350° F., typically, 300° F., and a somewhat reduced pressure from the pressure of distillation column **13**, generally, from 100 mm. Hg to 700 mm. Hg, typically, 40 mm. Hg. Proper choice of operating conditions within these limits will produce, for example, as a vapor fraction in line **19** from 1% to 11% of the feed in line **16**. It was found in the practice of this invention that this separation operation produced a concentrate of aromatic hydrocarbons in line **19** in vapor form. It follows, therefore, that the aromatic content of the solvent in the liquid fraction leaving separator **18** via line **11** contained, for example, from 0.2% to 6.2% by weight aromatic hydrocarbons and, typically, 3.2% aromatics while the material in line **15** contained, for example, from 10% to 14% aromatic hydrocarbons.

As a preferred operation of this embodiment of the invention, a vapor sidecut fraction is withdrawn from distillation column **13** via line **20**. The locus for withdrawal is selected so that the material in line **20** contains a concentration of aromatic hydrocarbons and is of sufficient purity from an aromatic standpoint so that no contaminating non-aromatic hydrocarbons will appear in the distillation fraction from distillation column **21**. As a practical mode of operation, the vapor fraction in line **20** is admixed with the vapor fraction in line **19** and these are passed together into distillation column **21**, as previously mentioned. As used herein, however, the term "together" is intended to embody the situation where the two vapor fractions are indeed passed separately into distillation column **21** depending upon the desires of those skilled in the art of design of distillation columns. Generally, the material in line **20** will contain, for example, from 50% to 99% aromatic hydrocarbons, typically 94% at 10 p.s.ig. and 335° F., and will be withdrawn at a temperature of, say, from 170° F. to 350° F., typically, about 335° F.

Distillation column **21** is operated in conventional manner for the separation of aromatic hydrocarbons from the selective solvent. Depending upon the volatility characteristics of the solvent, the operating conditions are chosen so that extremely high purity aromatic hydrocarbons may be withdrawn from distillation column **21** via line **22**. Lean solvent suitable for reuse in the extraction process is withdrawn from distillation column **21** via line **23**. Typical operating conditions for distillation column **21** when utilizing sulfolane as the solvent and desiring to recover, for example, benzene and toluene as the aromatic hydrocarbons include an overhead temperature from 120° F. to 306° F., a bottoms temperature from 300° F. to 350° F., and a separating column pressure of from 90 mm. Hg to 760 mm. Hg. Those skilled in the art from the general knowledge and from the teachings presented herein will know how to choose the proper operating conditions, depending on the composition of the feed to column **21**, to effectuate recovery of high purity aromatic hydrocarbons from the solvent.

It is to be noted from the above description that the function of separator **18** is to significantly reduce the distillation load on column **21**. The practical effect of this function is to reduce the column size and operating expense for the recovery of the aromatic hydrocarbons from the solvent. Exchanger **17**, previously mentioned, is an optional function, but in the practice of this invention it is frequently desirable to elevate the temperature of the material in line **16** so that a maximum amount of aromatic hydrocarbons may be flashed from the solvent phase. The optional use of exchanger **17** in conjunction with the withdrawal of a vapor phase via line **20**, has the benefit of increasing the preheat of the feed to column **21**, thereby further reducing the heat input requirements to column **21** and/or significantly reducing the amount of stripping steam necessary in column **21**. Exchanger **17**, of course, may be an indirect heat exchanger using either steam, hot oil, and the like, as the heating medium.

With reference to FIGURE 2, another embodiment of the present invention includes the introduction of an extract feed stream, as previously mentioned, into the system via line **101**. Added solvent is mixed with the feed stream from **102** and the mixture of extract feed and the added solvent is passed via line **103** into distillation column **104**. For purposes of illustration, operating conditions in distillation column **104** are substantially the same as those presented in the description of the embodiment of FIGURE 1. By way of emphasis, the operating conditions, of course, are dependent upon the characteristics of the solvent utilized and the characteristics of the aromatic and non-aromatic hydrocarbons contained in the solvent phase. The particular operating conditions presented previously were for the case of sulfolane as the solvent and benzene and toluene as the desired aromatic hydrocarbons. Returning to FIGURE 2, the non-aromatic hydrocarbon contaminants are removed from extractive distillation column **104** via line **105**. A lower stream comprising solvent and aromatic hydrocarbons is removed preferably from the bottom of column **104** via line **106** and passed entirely through exchanger **107** into separator **108**. Suitable operating conditions are maintained in separator **108** to produce a vapor fraction containing aromatic hydrocarbons in line **109** and a liquid fraction comprising solvent having reduced aromatic content in line **102**. As previously mentioned, a portion of the material in line **102** is passed via continuing line **102** into admixture with the incoming feed in line **101**. The amount of material returning for admixture with the feed depends on the feed composition to column **104** but, typically, may be from 5% to 75% of the total material removed as liquid from separator **108**. The other portion of the liquid fraction from separator **108** is passed from line **102** via

line **110** into distillation column **112**, generally, at a locus in the upper portion of column **112**.

The vapor fraction containing a concentrate of aromatic hydrocarbons is passed via line **109** into distillation column **112**, generally, at a locus below the locus selected for entry of the liquid stream from line **110** into column **112**. Alternate methods of introduction, previously mentioned with reference to column **21** of FIGURE 1, may also be used.

As a preferred optional operation, a vapor fraction is withdrawn from distillation column **104** via line **111** and for practical operation admixed with the vapor fraction in line **109** and passed together into distillation column **112**. As previously described, the material in line **111** is selected to contain aromatic hydrocarbons dissolved in solvent of such purity that substantially no non-aromatic hydrocarbons are transferred into distillation column **112**.

Operating conditions in distillation column **112** are maintained sufficient to separate high purity aromatic hydrocarbons as a distillate fraction in line **113** and a lean solvent fraction suitable for reuse in the solvent extraction step in line **114**.

One of the advantageous features it was discovered in the practice of the embodiments of the invention discussed hereinabove is the achievement of a considerably elevated temperature in the bottom of distillation column **13** and **104**. By utilizing the vapor sidecut withdrawal feature from this extractive distillation column, a considerable increase in temperature of the bottoms may be achieved without increasing the loss of aromatic hydrocarbons in the non-aromatic hydrocarbon fraction removed from this column. By operating at a considerably increased temperature at the bottom of the extractive distillation column, considerably less heat is required in final aromatic recovery distillation columns **21** and **112**. Operating in conjunction with the increased distillation column bottoms temperature, the separator between the two distillation operations permits a considerable reduction in the quantity of material which normally is passed through the aromatic recovery column. The economic benefits from this combined operation are lower capital expenses for the aromatic recovery column and lower operating expenses for the column.

Preferred embodiment

From the teachings presented hereinabove, the preferred embodiment of this invention includes the method for recovery of aromatic hydrocarbons from the extract phase from a solvent extraction zone which comprises the steps of: (a) introducing an extract phase containing solvent having aromatic hydrocarbons dissolved therein, said extract also contaminated with non-aromatic hydrocarbons, into a first distillation zone in the presence of a hereinafter specified added solvent stream; (b) withdrawing from said first zone an upper stream comprising said contaminants and a lower stream comprising solvent and aromatic hydrocarbons; (c) passing entirely said lower stream into a separation zone maintained under conditions sufficient to produce a vapor fraction containing aromatic hydrocarbons and a liquid fraction comprising solvent; (d) introducing said vapor fraction and a portion of said liquid fraction into a second distillation zone under conditions sufficient to produce a distillate fraction comprising aromatic hydrocarbons and a bottoms fraction comprising solvent suitable for reuse in the solvent extraction zone; and, (e) returning the other portion of said liquid fraction of Step (c) to Step (a) as said added solvent.

A distinctly preferred embodiment of this invention includes the preferred method hereinabove wherein the solvent comprises sulfolane and a vapor intermediate sidecut stream containing aromatic hydrocarbons is withdrawn from said first distillation zone, admixed with said vapor fraction of Step (c), and passed together into said second distillation zone as specified.

The invention claimed:

1. Method for recovering aromatic hydrocarbons from the extract phase from a solvent extraction zone which comprises the steps of:

(a) introducing an extract phase containing solvent having aromatic hydrocarbons dissolved therein, said extract also contaminated with non-aromatic hydrocarbons, into a first distillation zone in the presence of hereinafter specified added solvent stream;

(b) withdrawing from said first zone an upper stream comprising said contaminants and a lower stream comprising solvent and aromatic hydrocarbons;

(c) passing a portion of said lower stream into a separation zone maintained under conditions sufficient to produce a vapor fraction containing aromatic hydrocarbons and a liquid fraction comprising solvent;

(d) introducing said vapor fraction and the other portion of said lower stream into a second distillation zone under conditions sufficient to produce a distillate fraction comprising aromatic hydrocarbons and a bottoms fraction comprising solvent suitable for reuse in said extraction zone; and,

(e) returning said liquid fraction of Step (c) to Step (a) as said added solvent.

2. Method according to claim **1** wherein said solvent comprises sulfolane.

3. Method according to claim **1** wherein a vapor intermediate sidecut stream containing aromatic hydrocarbons is withdrawn from said first distillation zone, admixed with said vapor fraction of Step (c), and passed together into said second distillation zone as specified.

4. Method according to claim **1** wherein said conditions for separation in Step (c) include an elevated temperature and reduced pressure.

5. Method according to claim **3** wherein said portion of lower stream in Step (c) is heated by indirect heat exchange prior to passage into said separation zone; and, wherein said solvent comprises sulfolane.

6. Method for recovering aromatic hydrocarbons from the extract phase from a solvent extraction zone which comprises the steps of:

(a) introducing an extract phase containing solvent having aromatic hydrocarbons dissolved therein, said extract also contaminated with non-aromatic hydrocarbons, into a first distillation zone in the presence of a hereinafter specified added solvent stream;

(b) withdrawing from said first zone an upper stream comprising said contaminants and a lower stream comprising solvent and aromatic hydrocarbons;

(c) passing entirely said lower stream into a separation zone maintained under conditions sufficient to produce a vapor fraction containing aromatic hydrocarbons and a liquid fraction comprising solvent;

(d) introducing said vapor fraction and a portion of said liquid fraction into a second distillation zone under conditions sufficient to produce a distillate fraction comprising aromatic hydrocarbons and a bottoms fraction comprising solvent suitable for reuse in the solvent extraction zone; and,

(e) returning the other portion of said liquid fraction of Step (c) to Step (a) as said added solvent.

7. Method according to claim **6** wherein said solvent comprises sulfolane.

8. Method according to claim **6** wherein a vapor intermediate sidecut stream containing aromatic hydrocarbons is withdrawn from said first distillation zone, admixed with said vapor fraction of Step (c), and passed together into said second distillation zone as specified.

9. Method according to claim **7** wherein said conditions for separation in Step (c) include an elevated temperature and reduced pressure.

10. Method according to claim **8** wherein said lower stream of Step (c) is heated by indirect heat exchange

prior to passage into said separation zone; and, wherein said solvent comprises sulfolane.

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U.S. Cl. X.R.

208—321, 325