

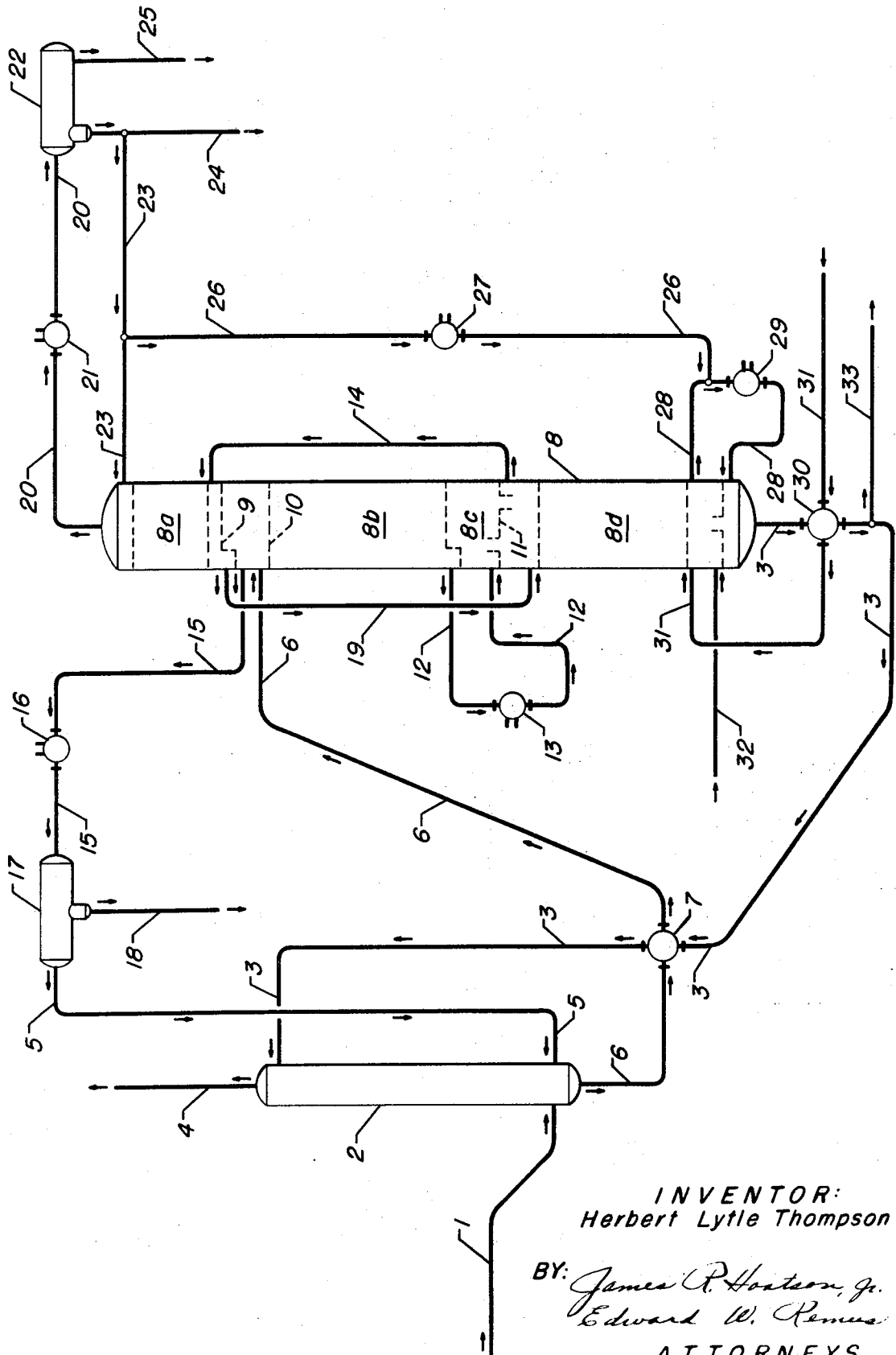
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H. L. THOMPSON

3,702,295

AROMATIC HYDROCARBON RECOVERY METHOD

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3,702,295

AROMATIC HYDROCARBON RECOVERY METHOD

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U.S. Cl. 208—321

12 Claims

ABSTRACT OF THE DISCLOSURE

High purity aromatic hydrocarbons are extracted from a mixture of aromatic and non-aromatic hydrocarbons by a combination of solvent extraction, stripping of the aromatic extract and the rectification of a vapor side cut from the aromatic stripping in the presence of a water reflux.

BACKGROUND OF THE INVENTION

The method of the present invention relates to the solvent extraction and recovery of aromatic hydrocarbons from hydrocarbon feedstocks containing aromatic and nonaromatic hydrocarbons. The present invention more particularly relates to the recovery of aromatic hydrocarbons from an extract phase provided by an aromatic-selective, solvent extraction process, particularly when sulfolane solvent is utilized therein.

Conventional processes for the recovery of high purity aromatic hydrocarbons such as benzene, toluene and xylenes (BTX) from various hydrocarbon feedstocks including catalytic reformate, hydrogenated pyrolysis gasoline, etc., utilize an aromatic selective solvent. These processes produce an extract phase containing aromatic hydrocarbons, solvent and contaminating amounts of non-aromatic hydrocarbons from which the aromatics must be recovered.

Two of the more prevalent methods of effecting selective aromatic extraction, utilize either a glycol or sulfolane type solvent. However, in the last decade the industry has utilized a sulfolane type system as its preferred mode of operation because of certain well known, desirable advantages which a sulfolane system possesses in comparison to a glycol type system. More particularly, the industry has often found it to be advantageous to convert existing glycol type units into sulfolane type units to increase their efficiency. Typical of the prior art glycol units which are being converted to a sulfolane type operation is that process embodied within U.S. Pat. No. 2,730,558. This cited glycol type operation utilizes a solvent extraction zone in conjunction with a stripping column which comprises a flash zone, a vaporizing zone and a stripping zone operating in a manner sufficient to substantially remove solvent and contaminating quantities of non-aromatic hydrocarbons from the extract phase recovered from the solvent extraction zone. However, it has been difficult in commercially operating plants to fully utilize this type of stripping column when the extraction operation is changed from a glycol type operation to a sulfolane type operation. One of the problems in the conversion from a glycol to a sulfolane operation revolves around the inability to effectively remove the sulfolane solvent from the aromatics recovered as a vapor side stream from the described stripping column, since it is impractical to water wash sulfolane solvent from the aromatic hydrocarbons as is utilized in a glycol type operation. Accordingly, the refiner typically installs a recovery fractionation column for separating aromatic hydrocarbons and sulfolane solvent since the stripping column which was converted from a glycol type operation does not produce, by itself, a relatively solvent-free aromatic extract. Thus, the conversion of a glycol type

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operation to a sulfolane type operation as currently practiced by the art requires installation of additional equipment and other capital items.

SUMMARY OF THE INVENTION

Therefore, it is an object of this invention to provide a method for the recovery of aromatic hydrocarbons from an extract phase produced in a solvent extraction operation particularly when using sulfolane as the aromatic selective solvent.

It is another object of this invention to provide a method for the recovery of aromatic hydrocarbons from a mixture of aromatic and non-aromatic hydrocarbons in a facile and economical manner.

It is a particular object of this invention to provide a method for readily converting and operating existing prior art processes originally built for glycol type solvents when converted to a sulfolane type solvent, and as a result, produce an efficient sulfolane type operation without the addition of appreciable amount of equipment.

In an embodiment, therefore, the present invention relates to a method for recovering aromatic hydrocarbons from an extract phase from an aromatic selective solvent extraction zone which, in a preferred embodiment, utilizes sulfolane as an aromatic selective solvent. The aromatic hydrocarbons are recovered by introducing the extract phase containing aromatic hydrocarbons, aromatic-selective solvent and contaminating non-aromatic hydrocarbons into an upper section of a stripping zone and introducing steam into a lower section of the stripping zone. From the upper section of the stripping zone, a first vapor fraction, comprising water and non-aromatic hydrocarbons, is withdrawn while a second vapor fraction comprising aromatic hydrocarbons and solvent is withdrawn from the intermediate section of the stripping zone. The second intermediate vapor fraction is then passed into a lower section of a rectification zone with a third vapor fraction comprising aromatic hydrocarbons and water being withdrawn from an upper section of this rectification zone. This third vapor fraction is then condensed to provide a relatively non-aromatic and solvent-free aromatic hydrocarbon stream and a liquid water stream. At least a portion of the liquid water stream is passed as reflux into the upper section of the rectification zone. Withdrawn from the lower section of the rectification zone is a liquid stream comprising solvent and water which is passed into the intermediate section of the stripping zone. A lean solvent stream is withdrawn from the lower section of the stripping zone and is preferably suitable for use in a solvent extraction zone.

In a further more limited embodiment, an intermediate liquid fraction from the stripping zone is reboiled, preferably by withdrawing a liquid fraction from the intermediate section of the stripping zone, heating the fraction and passing it back to the same intermediate section.

By utilizing the method set forth, aromatic hydrocarbons relatively free of contaminating amounts of non-aromatic hydrocarbons and solvent is produced by minor modifications to a conventional glycol type operation, without the necessity of installing a separate recovery column for the separation of aromatic hydrocarbons and solvent.

Other objects, embodiments and more detailed description of the foregoing embodiments will be found in the following more detailed description of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

Hydrocarbon feedstocks suitable for utilization in the method of the present invention include many different

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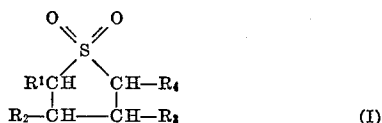
aromatic-non-aromatic mixtures having a substantially high enough concentration of aromatic hydrocarbons to economically justify the recovery of the aromatic hydrocarbons as a separate product stream. The present invention is particularly applicable to hydrocarbon feed mixture containing at least 25% by weight aromatic hydrocarbons. Typical aromatic feedstock charged to an extraction step will contain from about 30% to about 60% by weight aromatic hydrocarbons with aromatic hydrocarbon concentrations as high as 95% being suited in some instances. A suitable carbon range for the hydrocarbon feedstock is from about 6 carbon atoms per molecule to about 20 carbon atoms per molecule, and preferably from 6 to 10 carbon atoms per molecule.

One suitable source of hydrocarbon feedstock is a depentanized fraction from the effluent from a conventional catalytic reforming process unit for the reforming of a naphtha feedstock. Another suitable source of feedstock is the liquid by-product from a pyrolysis gasoline unit which has been hydrotreated to saturate olefins and diolefins, thereby producing an aromatic hydrocarbon concentrate suitable for the solvent extract technique described herein. The preferred feedstock recovered from a catalytic reforming unit, comprises single ring aromatic hydrocarbons of the C₆-C₈ range which are also mixed with corresponding boiling range paraffins and naphthenes which are present in the product from a catalytic reforming unit.

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 since water is preferably included in the solvent composition for enhancing its selectivity. In general, the solvent 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 are preferably 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 satisfactory 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.

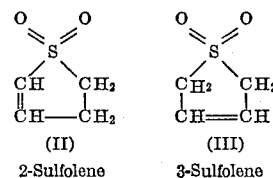
A preferred solvent which is utilized in the method of the present invention is a solvent of a sulfolane type. This solvent possesses a five membered ring containing one atom of sulfur and four atoms of carbon, with two oxygen atoms bonded to the sulfur atoms of the ring. Generically, the sulfolane-type solvents may be indicated as having the following structural formula:



wherein R₁, R₂, R₃, and R₄ are independently selected from the group comprising a hydrogen atom, an alkyl group having from one to ten carbon atoms, an alkoxy

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radical having from one to eight carbon atoms, and an arylalkyl radical having from one to twelve carbon atoms. Other solvents which may be included within this process are the sulfolenes such as 2-sulfolene or 3-sulfolene which have the following structures:



Other typical solvents which have a high selectivity for separating aromatics from non-aromatic hydrocarbons and which may be processed within the scope of the present invention are 2-methylsulfolane, 2,4-dimethylsulfolane, methyl - 2 - sulfonyl ether, n - aryl - 3 - sulfonyl amine, 2-sulfonyl acetate, diethylene glycol, various polyethylene glycols, dipropylene glycol, various polypropylene glycols, diglycol amines, dimethyl sulfoxide, N-methyl pyrrolidone, etc. The specifically preferred solvent chemical which is processed within the scope of the present invention is sulfolane, having the following structural formula:



As previously indicated the aromatic selectivity of the solvent can usually be enhanced by the addition of water to the solvent. Preferably, the solvents utilized in the practice of this invention contain small quantities of water in order to increase the selectivity of the overall solvent phase for aromatic hydrocarbons without reducing substantially the solubility of the solvent phase for aromatic hydrocarbons. The presence of water in the solvent composition further provides a relatively volatile material which is distilled from the solvent in the subsequent stripper following the extraction zone, more fully discussed hereinafter, which vaporizes the last traces of non-aromatic hydrocarbons from the solvent stream by steam distillation. Accordingly, the solvent composition of the present invention preferably contains from about 0.1% to about 20% by weight water and, preferably, about 0.5 to about 1.0% by weight depending upon the particular solvent utilized and the process conditions at which the extraction zone and the extractor-stripper are operated.

Aromatic hydrocarbons contained in the foregoing feedstocks are recovered by introducing the hydrocarbon feedstock into a solvent extraction zone maintained under solvent extraction conditions including the presence of an aromatic selective solvent of the type discussed. Solvent extraction conditions and techniques are generally well known to those trained in the art and vary, depending on the particular aromatic selective solvent utilized. Therefore, for brevity, from hereon in the discussion will be generally limited to the preferred solvent, sulfolane. Since those trained in the art can extrapolate to other solvents based on the teachings given herein for sulfolane, the scope of the present invention is not to be unduly limited to this single solvent species.

The solvent extraction zone provides an extract phase comprising solvent having aromatic hydrocarbons and a minor amount of non-aromatic hydrocarbons dissolved therein and a raffinate phase comprising non-aromatic hydrocarbons. Typically, the raffinate is water washed to remove any sulfolane which may be entrained therein. Preferably, the extraction conditions utilized are correlated to maintain the solvent and hydrocarbons passed to the extraction zone in the liquid phase so as to embody a

liquid phase solvent extraction. The conditions, apparatus, and mode of operation associated with the solvent extraction zone are well known to those trained in the art. For example, see *Petroleum Refiner*, No. 8, vol. 38, September 1959, pages 185-192, the teachings of which are specifically incorporated by reference herein.

Also embodied within the solvent extraction zone is the concept of displacing heavier non-aromatic hydrocarbons from the extract phase at the lower end of the solvent extraction zone by utilizing the known technique of a non-aromatic hydrocarbon containing reflux at that point. By displacing the heavy non-aromatics with light non-aromatics, the resulting non-aromatics are more readily separable from the aromatics in the subsequent stripping zone to be discussed later. It is preferred that this reflux stream comprise relatively light non-aromatic hydrocarbons but significant quantities of aromatic hydrocarbons, i.e., 30% to 60% by weight, may be present in the reflux stream. The exact amount of reflux introduced into the lower section of the solvent extraction zone varies depending on the degree of non-aromatic hydrocarbon rejection desired in the extraction zone. Preferably the reflux is at least 10% by volume of the extract phase so as to insure effective displacement of the heavy non-aromatic hydrocarbons from the extract phase into the raffinate. According to the process of the present invention at least a portion, if not all, of the light non-aromatic reflux required is provided by a non-aromatic fraction removed as overhead from an upper section of a hereinafter described stripping section. This fraction is withdrawn as a vapor and contains water (steam) which is preferably condensed and removed before the non-aromatics are passed as reflux to the solvent extraction zone.

The solvent extraction zone is operated under conventional conditions including elevated temperature and a sufficiently elevated pressure to maintain the solvent reflux streams and hydrocarbon charge in the liquid phase. When utilizing sulfolane, suitable temperatures are about 80° F. to about 400° F., preferably about 175° F. to about 300° F., and suitable pressures are about atmospheric to about 400 p.s.i.g., preferably about 50 to 150 p.s.i.g. Solvent quantities should be sufficient to dissolve substantially all of the aromatic hydrocarbons present in the solvent extraction zone feed. Preferred are solvent to feed ratios, by volume, of about 2:1 to about 10:1 when utilizing a C₆-C₈ range naphtha cut as feed.

The extract phase from the solvent extraction zone comprising sulfolane solvent, aromatic hydrocarbons and contaminating non-aromatic hydrocarbons is introduced into an upper section of a stripping zone to remove therein, the non-aromatic hydrocarbons. This separation is accomplished by introducing steam, as a stripping medium, into a lower section of the stripping zone in amounts necessary to remove essentially all of the contaminating amounts of non-aromatic hydrocarbons from the extract phase as a first vapor fraction which is withdrawn from the upper section of the stripping zone. This vapor fraction comprises water (steam), non-aromatic hydrocarbons, and a lesser amount of aromatic hydrocarbons. This vapor fraction is preferably cooled and condensed to form an aqueous phase and a hydrocarbon phase. This hydrocarbon phase is then recovered and passed to the lower section of the solvent extraction zone to serve as the described light non-aromatic reflux and to recover the aromatic hydrocarbons contained in the original vapor fraction withdrawn from the upper portion of the stripping column. Since the stripping zone acts as an extractive stripper, lean sulfolane solvent may be added to a lower portion of the stripping zone to enhance the separation of the non-aromatics from the aromatics.

The aromatic hydrocarbons are recovered by withdrawing from an intermediate section of the stripping zone a second vapor fraction comprising aromatic hydrocarbons and solvent. This vapor fraction is relatively free of non-aromatic hydrocarbons, i.e., less than 1000

p.p.m., by weight on a hydrocarbon basis, and preferably less than 500 p.p.m. Preferably, to insure a more efficient separation, a liquid fraction from the intermediate section of the stripping zone is reboiled. This can be readily accomplished by inserting a heat exchange means within the column proper. However, when converting existing glycol units to sulfolane units, the reboiling of the intermediate liquid fraction is accomplished by withdrawing the liquid fraction from the stripping zone, heating the liquid in an external heating means and returning the heated liquid to the same locus of the stripping zone as it was withdrawn. This intermediate reboiling is particularly important when relatively large amounts of reflux comprising water and sulfolane solvent are passed from a lower section of a hereinafter described rectification zone to an intermediate section of the stripping zone. In essence, by withdrawing an overhead vapor fraction and an intermediate vapor fraction as described, the stripping zone actually embodies two stripping zones; an upper zone for stripping non-aromatic hydrocarbons from the solvent and aromatics and a lower zone for stripping the aromatics from the sulfolane solvent. Withdrawn from the lower section of the stripping zone is a lean sulfolane solvent stream which is preferably suitable for utilization in the solvent extraction zone.

The exact conditions to be utilized in a stripping zone of the type described are broadly within a temperature range of about 200° F. to about 500° F. and a pressure range of about 50 mm. Hg absolute to about 25 p.s.i.g. As is well known to those trained in the art, more exact processing conditions are a function of a myriad of variables, particularly feed compositions, aromatic purity desired and aromatic recovery sought. However, based on the teaching herein, it is within the scope of one trained in the art to readily develop specific processing conditions for a given feedstock. In the particular instance of the conversion of a glycol type unit to a sulfolane type unit, the existing glycol stripper, if of the type described in U.S. Pat. No. 2,730,558, is not usually designed for subatmospheric conditions. Therefore, processing conditions, after conversion, are within the broad temperature range of 200° F. to 500° F., preferably about 300° F. to about 375° F. and a pressure range of about 1 p.s.i.g. to about 25 p.s.i.g.

The second vapor fraction comprising aromatic hydrocarbons, water and solvent which was withdrawn from an intermediate section of the stripping zone is then passed to a lower section of a rectification zone to separate therein the aromatic hydrocarbons from the sulfolane solvent. This separation is accomplished by maintaining the rectification zone under conditions including a temperature of about 100° F. to about 400° F. and a pressure of about 50 mm. Hg to about 25 p.s.i.g., preferably 5 p.s.i.g. to about 20 p.s.i.g., and withdrawing from an upper section of the rectification zone a vapor fraction relatively free of solvent comprising aromatic hydrocarbons and water (steam). This vapor fraction is condensed and the aromatics recovered are relatively free of non-aromatics and sulfolane solvent. At least a portion of the liquid water formed when the vapor fraction was condensed is passed to the upper section of the rectification zone to help effectively remove the sulfolane solvent from the aromatics. Withdrawn from a lower section of the rectification zone is a liquid stream comprising solvent and water. This stream is then passed, as reflux, to an intermediate section of the stripping zone to recover therein the sulfolane solvent for use in the extraction zone. The aromatic hydrocarbons recovered from the condensed rectification zone overhead may be further processed by means well known to those trained in the art, including clay treating and fractionation, to recover the individual hydrocarbon species such as benzene, toluenes and mixed xylenes.

The described stripping zone and rectification zone can be constructed as separate, grassroots units; however, the

present invention finds particular utility in converting existing glycol units to sulfolane units. Referring to U.S. Pat. No. 2,730,558, and stripping column 26 illustrated therein, flash section A of column 26 is readily converted to a rectification zone by the addition thereto of sufficient vapor-liquid contacting plates. Section B and C of column 6 are likewise converted to a dual function stripping section by the addition of appropriate internal vapor-liquid contacting trays, liquid trap out trays and a side cut reboiler.

DESCRIPTION OF THE DRAWING

The process of the present invention can be most clearly described by reference to the attached drawing, schematically illustrating the recovery of benzene and toluene from a C₆-C₇ mixture of aromatic and non-aromatic hydrocarbons. Of necessity, certain limitations must be present in a flow diagram of the type presented and no intention is made thereby to limit the scope of this invention to particular solvents, concentrations, weights, operating conditions, etc. Some miscellaneous appurtenances including valves, pumps, separators, reboilers, etc., have been eliminated. Only those vessels and lines necessary for a complete and clear understanding of the process of the present invention are illustrated, with any obvious modifications made by those possessing expertise in the art of aromatic solvent extraction, particularly of the sulfolane type, being included within the generally broad scope of the present invention.

Referring now to the attached schematic diagram, a C₆-C₇ cut depentanized reformat, containing 297 units (moles per hour) of aromatic hydrocarbons and 1127 units of non-aromatic hydrocarbons at a temperature of 100° F., is passed via line 1 to the lower section of solvent extraction zone 2. Solvent extraction zone 2 is a conventional sulfolane type extraction zone and is maintained under a pressure of about 125 p.s.i.g. Entering an upper portion of solvent extraction zone 2 via line 3 is a lean solvent stream, from a source to be described later, containing 7205 units of sulfolane and 1095 units of water at a temperature of 210° F. Within solvent extraction zone 2, the hydrocarbon and sulfolane streams are contacted to produce a raffinate phase, removed via line 4, containing 4 units of aromatic hydrocarbons, 1127 units of non-aromatic hydrocarbons, and about 12 units of sulfolane solvent. The sulfolane solvent contained in the raffinate phase removed via line 4 is readily recovered by passing this stream to a raffinate wash column not shown. Removed via line 6 from the bottom portion of solvent extraction zone 2 at a temperature of about 187° F. is an aromatic extract phase containing 371 units of aromatic hydrocarbons, 272 units of non-aromatic hydrocarbons, 1095 units of water and 7192 units of sulfolane. This mixture is indirectly heat exchanged in heat exchanging means 7 with lean solvent flowing in line 3 recovered from recovery column 8 in a manner to be described later.

The aromatic extraction phase, now at a temperature of 238° F. is passed, via line 6, to a unitary recovery column 8. Recovery column 8 comprises a rectification zone 8a, an upper stripping zone 8b, an intermediate zone 8c, and a lower stripping zone 8d. Contained within recovery column 8 is a series of vapor liquid contacting plates 10, a liquid trap out plate 11 and an imperforate means 9 which isolates rectification zone 8a from upper stripping zone 8b. It is to be noted that with the exception of the added internals mentioned, recovery column 8 is essentially identical to the stripping column illustrated in U.S. Pat. No. 2,730,558. 1546 units of water, typically recovered from a water still, not illustrated, enter via line 31 and are converted to steam in steam generator means 30 by indirect heat exchange with lean solvent leaving the bottom section of recovery column 8 via line 3. Steam leaves steam generator means 30 at a tem-

perature of 265° F. and enters the lower portion of recovery column 8. If desired, fresh or regenerated sulfolane solvent may be added via line 32. A liquid stream is removed from the bottom portion of recovery column 8 via line 28 and passed to reboiling means 29 and heated to a temperature of about 350° F. and passed back to the lower section of the column. This produces a bottoms temperature of about 346° F. and a bottoms pressure of about 12 p.s.i.g. Removed from intermediate section 8c via line 12 are about 94 units of liquid at about 278° F., which are heated in indirect heat exchange means 13 to a temperature of about 320° F. and passed back to section 8c via line 12. Also removed from intermediate section 8c via line 14 is a vapor fraction at a temperature of 310° F., containing 293 units of aromatic hydrocarbons, 1665 units of water, and 25 units sulfolane which are passed via line 14 to a lower portion of rectification zone 8a. Removed from the upper portion of upper stripping zone 8b, via line 15, is a vapor fraction, at a temperature of about 220° F. and a pressure of about 6 p.s.i.g. containing 2 units of sulfolane; 180 units of water; 272 units of non-aromatic hydrocarbons and 78 units of aromatic hydrocarbons. This vapor phase is condensed by cooling stream 15 in heat exchange means 16 to a temperature of about 100° F. The resulting two-phase mixture is passed via line 15 to liquid settler 17 from which the condensed water is removed via line 18 and is passed to a water still, not illustrated, to recover dissolved amounts of sulfolane. The hydrocarbons form a separate phase which are removed via line 5 and passed to a lower section of solvent recovery zone 2 to serve as a light non-aromatic reflux which displaces any of the heavy non-aromatic hydrocarbons which may be contained in the extract phase removed from solvent extraction zone 2.

Removed from an upper section of rectification zone 8a via line 20, is a vapor stream at a temperature of 220° F. containing 1795 units of water (steam) and 293 units of aromatic hydrocarbons. It is to be noted that this stream is essentially free of sulfolane solvent and non-aromatic hydrocarbons. Vapor stream 20 is condensed in heat exchange means 21 by cooling the vapor to a temperature of about 100° F. The resulting two-phase liquid mixture is passed to separation means 22 wherein the aromatic hydrocarbons and water are separated. Removed from separation zone 22 via line 25 is an aromatic extract of benzene and toluene which, when further fractionated to yield a pure benzene and toluene species, will produce a benzene fraction containing less than .05 percent by weight non-aromatic hydrocarbons and a toluene fraction containing less than 0.02 percent by weight non-aromatic hydrocarbons. Water is removed from separation means 22 via line 24. 400 units of this water are withdrawn from line 24 via line 23 and passed thereby to the upper portion of rectification zone 8a. The remaining portion of water is passed via line 24 to serve as a wash medium for removing solvent from the raffinate phase removed via line 4 from solvent extraction zone 2. If desired, a portion of the water recovered from separation means 22 can be withdrawn via line 26 heated in heat exchange means 27, converted to steam therein and passed via line 26 to a lower portion of stripping section 8d by commingling with the liquid withdrawn via line 28 and reboiled in heat exchange means 29. Withdrawn from lower section of rectification zone 8a via line 19 is a liquid phase at a temperature of about 230° F. containing 270 units of water and 25 units of sulfolane which is passed to intermediate section 8c of recovery column 8. As previously indicated, lean solvent suitable for use in solvent extraction zone 2 is removed via line 3 and is used to indirectly heat incoming steam in steam regeneration means 20 and extraction zone extract in heating means 7. If desired, a portion of the solvent may be withdrawn via line 33 for regeneration which insures solvent purity and removes any C₉ and C₁₀ aromatics contained in the process.

It is seen from the foregoing description that the method of the present invention converts a conventional glycol type unit to a sulfolane type unit capable of producing nitration grade aromatic hydrocarbons without the necessity of extensive addition of other equipment such as a recovery column which the art traditionally has installed when converting glycol type units to sulfolane type units. This is accomplished by utilizing the described sequence of process flows and by converting an existing glycol type unit into a stripping zone having two stripping sections contained therein; one for stripping non-aromatic hydrocarbons from the extract phase, and a lower stripping section for stripping out sulfolane solvent from the non-aromatic free, aromatics. However, since the lower section does not fully remove all of the sulfolane solvent under the conditions which are practically attainable therein, complete sulfolane removal is attained by further rectification of the recovered aromatic extract containing a minor amount of sulfolane in the presence of an aqueous reflux.

I claim as my invention:

1. A method for recovering aromatic hydrocarbons from an extract phase from an aromatic-selective, solvent extraction zone which comprises the steps of:

- (i) introducing the extract phase containing aromatic hydrocarbons, contaminating non-aromatic hydrocarbons, and aromatic-selective solvent into an upper section of a stripping zone;
- (ii) introducing steam into a lower section of the stripping zone;
- (iii) withdrawing from the upper section of the stripping zone a first vapor fraction comprising water and non-aromatic hydrocarbons;
- (iv) withdrawing from an intermediate section of the stripping zone a second vapor fraction comprising aromatic hydrocarbons, water and solvent;
- (v) passing the second vapor fraction into a lower section of a rectification zone;
- (vi) withdrawing from an upper section of the rectification zone a third vapor fraction comprising aromatic hydrocarbons and water;
- (vii) condensing the third vapor fraction to provide a relatively non-aromatic and solvent free aromatic hydrocarbon stream and a liquid water stream;
- (viii) passing at least a portion of the liquid water stream into the upper section of the rectification zone;
- (ix) withdrawing from the lower section of the rectification zone a liquid stream comprising solvent and water and passing said liquid stream into an intermediate section of the stripping zone; and
- (x) withdrawing from the lower section of the stripping zone a lean solvent stream.

2. The method of claim 1 wherein an intermediate liquid fraction from the stripping zone is reboiled.

3. The method of claim 2 wherein the intermediate liquid fraction is withdrawn from the intermediate section of the stripping zone and is heated and passed back to the intermediate section of the stripping zone.

4. The method of claim 1 wherein a portion of the liquid water stream from step (vii) is converted to steam and passed into the lower section of the stripping zone.

5. The method of claim 1 wherein the lean solvent stream is suitable for use in the solvent extraction zone.

6. The method of claim 1 wherein the solvent comprises a sulfolane type solvent.

7. The method of claim 1 wherein the solvent comprises sulfolane.

8. A method for recovering C_6 - C_8 aromatic hydrocarbons from a feedstock containing C_6 - C_8 aromatic hydrocarbons and non-aromatic hydrocarbons which comprises the steps of:

- (i) introducing said feedstock into a solvent extraction

zone maintained under aromatic extraction conditions including the presence of lean sulfolane solvent in an amount sufficient to separate the feedstock into a raffinate phase containing non-aromatic hydrocarbons and an extract phase comprising sulfolane having aromatic hydrocarbons and contaminating quantities of non-aromatic hydrocarbons dissolved therein;

- (ii) passing said extract phase into an upper section of a stripping zone maintained under stripping conditions including the introduction of stripping steam into a lower section of the stripping zone, passing of a hereinafter specified first liquid reflux stream into an intermediate section of the stripping zone, and the reboiling of a liquid fraction from an intermediate section of the stripping zone to provide

- (a) an upper vapor fraction comprising water and non-aromatic hydrocarbons;
- (b) an intermediate side cut vapor fraction, relatively free of non-aromatic hydrocarbons, comprising aromatic hydrocarbons, water and a minor amount of sulfolane; and
- (c) a bottoms fraction comprising sulfolane suitable for reuse in the solvent extraction zone of step (i);

- (iii) passing said intermediate vapor fraction into a lower section of a rectification zone maintained under conditions including the presence of a hereinafter specified second liquid reflux stream passed into an upper section of the rectification zone to provide

- (a) an overhead vapor fraction relatively free of non-aromatic hydrocarbons and sulfolane comprising aromatic hydrocarbons and water; and
- (b) a bottoms liquid fraction comprising sulfolane and water;

- (iv) passing said bottoms liquid fraction from the rectification section to the stripping zone as the specified first liquid reflux stream;

- (v) condensing said overhead vapor fraction to provide a non-aromatics and sulfolane free aromatic phase and a water phase; and

- (vi) passing at least a portion of said water phase into the upper section of the rectification zone as the specified second liquid reflux stream.

9. The method of claim 1 wherein a portion of the water phase from step (v) is converted to steam and passed to a lower section of the stripping zone.

10. The method of claim 1 wherein the upper vapor fraction from the stripping zone of step (ii) (a) is condensed to form a water containing phase and a hydrocarbon, non-aromatic containing phase with the hydrocarbon non-aromatic containing phase being passed to a lower section of the solvent extraction zone.

11. The method of claim 1 wherein fresh sulfolane solvent is introduced into a lower section of the stripping zone.

12. The method of claim 1 wherein the stripping conditions include a pressure of about 1 p.s.i.g. to about 25 p.s.i.g. and a temperature of about 200° F. to about 500° F.

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DELBERT E. GANTZ, Primary Examiner

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U.S. Cl. X.R.

208—325; 260—674 S.E.

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,702,295 Dated November 7, 1972

Inventor(s) Herbert Lytle Thompson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

In the Claims:

Claims 9 through 12, first line of each,
"claim 1" should read -- claim 8 --.

Signed and sealed this 26th day of November 1974.

(SEAL)
Attest:

McCOY M. GIBSON JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents