

D. B. BROUGHTON

RECOVERY OF AROMATICS

INVENTOR:  
Donald B. Broughton

BY: *James R. Houston Jr.*  
*Harold W. Bergendorf*

INVENTOR:  
Donald B. Broughton

BY: *James R. Houston Jr.*  
*Harold W. Bergendorf*

ATTORNEYS

3,435,087

## RECOVERY OF AROMATICS

Donald B. Broughton, Evanston, Ill., assignor to Universal Oil Products Company, Des Plaines, Ill., a corporation of Delaware

Filed Oct. 21, 1965, Ser. No. 499,974

Int. Cl. C07c 7/10, 15/00

U.S. Cl. 260—674

12 Claims

### ABSTRACT OF THE DISCLOSURE

In the separation and recovery of aromatic hydrocarbons from a hydrocarbon feed containing at least 75% by weight of aromatics by countercurrent extraction with a sulfolane-type solvent to produce a raffinate phase containing non-aromatics and an extract phase containing aromatics, the improvement which comprises commingling with the feed, prior to its introduction into the extraction column, a sufficient amount of recycled raffinate phase to produce an overall feed mixture which is substantially immiscible with the solvent.

This invention relates to a process for the separation of hydrocarbon mixtures with the aid of a selective solvent for the purpose of separating and recovering the aromatic hydrocarbon components of the mixture. In one of its more specific applications, the process of the present invention serves to segregate the particular species of aromatic hydrocarbons, such as benzene, toluene and/or  $C_8$  aromatics from other classes of hydrocarbons that are normally contained in petroleum distillates, and utilizes a solvent which may be indefinitely recycled in the system, yields the desired hydrocarbon product in a state of high purity and separates the same substantially in its entirety from the feed stocks charged to the process.

The present invention is concerned with an improvement in the type of a separation process wherein a feed comprising a mixture of various classes of hydrocarbons having about 75% by weight of aromatic hydrocarbons is introduced into an extraction zone at an intermediate point thereof and is countercurrently contacted therein with a solvent selective for aromatic hydrocarbons, a raffinate phase comprising substantially all of the non-aromatic hydrocarbons in the feed stock is removed from one end portion of the extraction zone at which the solvent is introduced, a sufficient portion of said raffinate is recycled to said intermediate point to make the overall mixture of feed and recycled raffinate immiscible with the solvent, an extract phase comprising the aromatic components of the feed stocks dissolved in the selective solvent is removed from the other end portion of the extraction zone and the aromatic solute is subsequently recovered by stripping and fractionating the extract phase.

In accordance with the present invention, the extract phase is substantially freed, prior to its removal from the extraction zone, of substantially all of the non-aromatic components dissolved by the solvent from the feed stock by introducing a light hydrocarbon which is more volatile in the presence of the solvent than either the aromatic or raffinate components of the feed stock into the extraction zone at a point between the point of removal of extract phase and the point of introduction of the feed stock and displacing into the raffinate stream the residue of non-aromatic components dissolved in the solvent from the feed stock and replacing in the extract stream the more volatile light hydrocarbon, thereafter separating the more volatile light hydrocarbon contained in the resultant extract phase by stripping the extract after its removal from the extraction zone.

In a more specific embodiment this invention relates to a process for the separation and recovery of aromatic hydrocarbons from a hydrocarbon mixture having an aromatics content of at least 75% by weight which comprises: contacting the mixture with a sufficient amount of raffinate phase from a source hereinafter described to produce an overall mixture which is immiscible with a lean sulfolane-type solvent; introducing the lean sulfolane solvent into an upper point in the height of an extraction zone; introducing the overall mixture into an intermediate point in the height of the extraction zone and countercurrently contacting said overall mixture therein with the sulfolane solvent at an elevated temperature and at a pressure sufficient to maintain said solvent and said mixture in substantially liquid phase thereby forming a liquid raffinate phase and a liquid extract phase; removing the raffinate phase from the upper portion of said zone and recycling a sufficient portion of the raffinate phase back to said hydrocarbon mixture to produce said overall mixture; countercurrently contacting the extract phase in the lower portion of said zone with a light hydrocarbon which boils in the presence of the solvent at a temperature below the boiling point of the aromatic hydrocarbon being extracted but which remains in the liquid state at said temperature and pressure, said light hydrocarbon being introduced into a lower point of the extraction zone in sufficient amount to replace a substantial amount of the non-aromatic hydrocarbons of said mixture in the extract phase with said light hydrocarbons; removing from the lower portion of said zone the extract phase containing the light hydrocarbon, introducing the extract phase into a stripper column, removing an overhead stream from said column comprising the light hydrocarbon and returning the light hydrocarbon to the lower point of the extraction zone; removing a fat solvent stream comprising aromatic hydrocarbons and solvent from the bottom of the stripper column, introducing the column bottoms into a fractionating tower, removing a tower overhead stream therefrom comprising aromatics and a tower bottoms stream therefrom comprising lean solvent; and returning at least a portion of the tower bottoms stream to the upper point of the extraction zone.

Solvent extraction processes for the recovery of aromatic hydrocarbons from hydrocarbon mixtures have been known for many years. This is usually accomplished by processes such as extractive distillation or alternately by countercurrently contacting the feed with a solvent selected for aromatics in the liquid phase within an extractor. The present invention relates to the latter process. If the feed stock non-aromatic components present in the extract phase are not displaced in the extraction zone, the heavier of these components will not be readily stripped out of the aromatic-solvent mixture in the extractive stripper and due to their similarity of boiling points will dilute the ultimate aromatic purity. Thus it has been observed that when recovering aromatics from a  $C_6$ - $C_9$  feed using a sulfolane solvent, some of the  $C_9$  non-aromatics remain in the extract phase and eventually wind up in the  $C_8$  aromatic stream thereby reducing the purity of said  $C_8$  aromatic stream. This problem is further compounded by the increased use of hydrotreated pyrolysis oils as feed stocks for solvent extraction processes since the hydrotreating step tends to hydrogenate a portion of the aromatics thereby producing appreciable amounts of naphthenes, and these heavier naphthenes are more soluble in sulfolane than the corresponding heavy paraffins. If the heavy naphthenes and heavy paraffins are replaced with a light hydrocarbon, it is possible to remove the light hydrocarbons from the aromatics in the extractive stripper.

A significant problem encountered with the use of sulfolane-type solvents is that when the aromatics concentration of the hydrocarbon feed stock gets sufficiently high (above about 75% by weight) the feed stock phase becomes miscible with the solvent phase. Thus under these circumstances it becomes impossible to counter-

currently contact the solvent and the hydrocarbon phase in the liquid phase in an extractor and achieve the desired separation. In the past, attempts have been made to solve this miscibility problem by introducing the high aromatic content into the extractive stripper, sending the overhead stream from the stripper to the extractor (this overhead stream has a lower concentration of aromatics), contacting this overhead stream countercurrently with a lean solvent therein, and returning the extract phase from the extractor to the extractive stripper. Such a process is shown and claimed in U.S. Patent No. 3,146,190. However, this patented process does not allow the use of the light hydrocarbon backwash to remove the heavier non-aromatics from the extract phase and accordingly will make it very difficult to remove  $C_9$  paraffins and naphthenes from the solvent in the stripper, and will therefore reduce the purity of the ultimately separated aromatic hydrocarbons.

It is an object of this invention to overcome the foregoing problems in solvent extraction processes employing sulfolane-type solvents.

It is another object of this invention to introduce high aromatic content feeds directly into a liquid phase extractor and countercurrently contact a lean sulfolane-type solvent therein.

It is another object of this invention to use the lower portion of a liquid phase extractor to displace heavy non-aromatic hydrocarbons from an extract phase when using a hydrocarbon feed having an aromatic concentration of more than 75% by weight and a sulfolane-type solvent in a solvent extraction process for the separation and recovery of aromatic hydrocarbons.

It is a further object of this invention to increase the purity of the recovered aromatics in a sulfolane-type solvent extraction process using high aromatics content feed stocks.

It is still another object of this invention to increase the yield of the recovered aromatics in a sulfolane-type solvent extraction process using high aromatic content feed stocks.

It is another object of this invention to efficiently displace heavy non-aromatics from the extract phase with a light hydrocarbon in an extractor using a high aromatic content feed stock and a sulfolane-type solvent extraction process.

These and other objects will become more apparent especially in the light of the following detailed description.

Referring to the accompanying drawing, the feed is introduced into flow conduit 1 where it flows into an intermediate point in extractor 2. Preferably the feed is introduced into the extractor about  $\frac{1}{3}$  of the way along its height from the bottom. This permits the feed to contact the solvent in the upper  $\frac{2}{3}$  of the extractor and permits the extract phase to contact the light hydrocarbon backwash stream in the bottom  $\frac{1}{3}$  of the extractor. Additional non-aromatic hydrocarbon from a source described hereinafter flows through flow conduit 37 and mixes with the feed prior to entering extractor 2. This additional hydrocarbon flows at a sufficiently high rate to make the overall mixture entering the extractor immiscible with the solvent. Lean solvent is introduced into the upper part of extractor 2 through flow conduit 20 whereupon the lean solvent countercurrently contacts the overall mixture. The extractor is maintained at an elevated temperature and pressure sufficient to maintain the solvent and the feed mixture in the liquid phase. Since the solvent has a selectivity for the aromatics and the solvent is immiscible with the feed and additional hydrocarbon mixture, aromatics will preferentially dissolve into the solvent

phase. Thus, as the solvent passes downflow through the upper  $\frac{2}{3}$  of the extractor the aromatics concentration therein gradually increases. Appropriately, the solvent that is first introduced into the extractor is called lean solvent and the solvent containing dissolved aromatics is called the fat solvent. The feed and additional hydrocarbon mixture phase passes upflow through the extractor since it is less dense than the solvent phase and the extractor contains suitable means to efficiently contact the hydrocarbon phase and the solvent phase. As the feed hydrocarbon phase continues upflow through the extractor the aromatics and a small quantity of non-aromatics gradually dissolves into the solvent phase such that when the hydrocarbon phase reaches the top of the extractor, it contains essentially no aromatics and is called the raffinate phase. To raffinate phase is withdrawn from extractor 2 through flow conduit 23 where a portion of it is recycled back to the intermediate point of the extractor by flowing through pump 36 and flow conduit 37. This stream is the additional non-aromatic hydrocarbon referred to hereinbefore. The remaining portion of the raffinate phase continues through flow conduit 23 where it may be fractionated and/or water washed to remove small quantities of dissolved solvent. The fat solvent called the extract phase passes into the lower  $\frac{1}{3}$  of the extractor where it countercurrently contacts an immiscible light hydrocarbon stream called the backwash. The backwashing step results in the displacement from the extract phase into the backwash hydrocarbon phase of the non-aromatic feed components present in the extract phase. It must be remembered that sulfolane-type solvents will dissolve some non-aromatics along with the aromatics of the feed and the object of the backwashing is to displace the heavier feed non-aromatic hydrocarbons with the lighter hydrocarbons. The backwashing is accomplished by introducing the light hydrocarbon into the lower point of extractor 2 through flow conduit 11.

The backwashed extract phase is withdrawn from the lower portion of extractor 2 through flow conduit 3 where it is sent to extractive stripper 4. Stripper 4 is able to remove the non-aromatic components of the extract phase because the presence of the solvent in the stripper has less effect on the boiling point of the non-aromatic compounds dissolved in the solvent than it does on the aromatic compounds also dissolved in the solvent. In general, the solvent tends to retain in solution the more soluble aromatic component, even at temperatures considerably above the normal boiling point of the aromatics alone. However, when processing feed stocks of wider boiling ranges, the stripper will effectively remove the lighter non-aromatics overhead but will have difficulty in removing the heavier non-aromatics overhead without also vaporizing the lighter aromatics. Furthermore, when the heavier non-aromatics contain appreciable amounts of naphthenes this difficulty is increased since the solvent generally will tend to retain the naphthenes in solution more readily than the paraffins. In the presence of sulfolane, benzene and  $C_9$  naphthenes have about the same volatility and therefore it becomes impossible to remove  $C_9$  naphthenes in the stripper without also removing some  $C_8$  aromatics. If the  $C_9$  naphthenes are not removed in the stripper they remain in the aromatics product when the solvent is subsequently removed from the extract phase and due to their closeness in boiling point with  $C_8$  aromatics cannot be readily separated therefrom by ordinary fractionation. This of course results in low purity  $C_8$  aromatics. Therefore, the heavier non-aromatics are displaced by the lighter non-aromatic hydrocarbons in the backwash step to permit the lighter non-aromatics to be readily vaporized in the stripper. Light paraffins are less effective in displacing heavy naphthenes than light naphthenes and accordingly it is preferred that the light backwash material contain an appreciable concentration of naphthene—at least about

5%. When using saturated sulfolane as the solvent, the ratio of the solvent's selectivity for naphthene compared to a paraffin of the same carbon number is about 1.4 to 1.0. Also, as the molecular weight of the naphthenes increases, the selectivity of saturated sulfolane for said naphthene decreases by a factor of about 1.2 per carbon atom. Thus a light naphthene containing backwash material is efficient and highly satisfactory to displace the heavier non-aromatics from the extract phase. The stripper is operated at elevated temperatures and intermediate pressures (relative to the extractor pressure) in order to remove substantially all of the non-aromatics, some of the water and aromatics and a small amount of the solvent overhead. This overhead stream is removed from stripper 4 through flow conduit 5 where it passes through condenser 6, flow conduit 7 and into stripper receiver 8. The overhead material is condensed and separated into two phases therein, one a water-solvent phase and the other a light hydrocarbon stream. The light hydrocarbon phase is withdrawn from receiver 8 through flow conduit 11 where it is recycled back to the lower point of extractor 2. The water solvent phase settles in boot 9 where it is withdrawn therefrom through flow conduit 10 and is sent to solvent recovery means not shown. A portion of the bottoms fraction from the stripper flows through flow conduit 38 wherein it passes through reboiler heater 39 and returns to stripper 4. The remaining bottoms portion comprising aromatics and solvent is withdrawn from stripper 4 and passes through flow conduit 12, and into solvent recovery column 14.

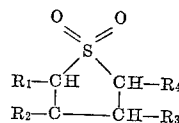
Column 14 is operated at low pressure (relative to the extractor pressure) and elevated temperatures to separate the solvent from the aromatics. The aromatics and a small amount of water are removed as a vapor overhead from column 14 and pass through flow conduit 15, condenser 16 and into column receiver 17. The overhead material is separated into a hydrocarbon phase comprising aromatics and a water phase. The aromatics are withdrawn from receiver 17 through flow conduit 18 where a portion thereof returns to column 14 as reflux while remaining portion of aromatics is withdrawn through flow conduit 19 as net product. Generally, the net product is sent to a series of fractionators to recover the individual aromatics as substantially pure components such as benzene, toluene, ortho-xylene, ethylbenzene, etc. A portion of the bottoms material from column 14 is withdrawn through flow conduit 40 where it passes through reboiler 41 before returning to column 14. The remaining bottoms portion comprising lean solvent is withdrawn through flow conduit 20 wherein a fraction thereof is recycled back to the upper point in extractor 2. The other fraction passes through flow conduit 13 and mixes with the extract phase prior to entering stripper 14. This is done to further improve the separation of aromatics and non-aromatics in the stripper due to extractive distillation since a larger quantity of solvent further increases the difference in boiling point between the non-aromatics and the aromatics in the extract phase. A small but discernible amount of decomposition of solvent and other sludge forming reactions may occur in the process and it is desirable to remove a small slipstream of lean solvent from flow conduit 20 through flow conduit 21 and regenerate the solvent in equipment not shown in order to prevent a build-up of sludge. The regeneration preferably is done by rerunning the solvent and thereafter returning the regenerated solvent to flow conduit 20 by means of flow conduit 22.

Since in some instances a portion of the light hydrocarbon backwash may pass through extractor 20 and leave in the raffinate phase out flow conduit 23, it would then be necessary to supply sufficient backwash material to make up for this loss. This is conveniently accomplished by passing the raffinate phase through flow conduit 7 and into stripper receiver 8 to supply any make

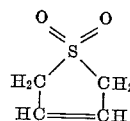
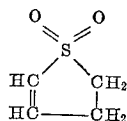
conduit 32, flow conduit 33 and reboiler 34 and returns overhead receiver 27. A portion of the condensed light raffinate is returned to splitter 24 through flow conduit 28 as reflux while the other portion is withdrawn through flow conduit 29. A fraction of this other portion of light raffinate is recycled through flow conduit 31, flow conduit 7 and into stripper receiver 8 to supply any make up necessary for loss of light hydrocarbon while the remaining fraction of light raffinate is withdrawn through flow conduit 30 as net product. A heavy raffinate portion is removed from the bottom of splitter 24 through flow conduit 32, flow conduit 33 and reboiler 34 and returns to splitter 24. The net heavy raffinate fraction is withdrawn through flow conduit 35. In the case of feed stocks which contain appreciable amounts of light naphthenes in the non-aromatic portion, these naphthenes will be adequate to efficiently displace the heavier non-aromatic components from the extract phase. In the case of feed stocks low in naphthenes, an outside source of naphthenes, preferably of lower boiling point than the components of the feed stock, may be initially loaded into the process and splitter 24 is operated to only remove overhead only the lower boiling light naphthenes which are totally recycled to stripper receiver 8.

Suitable feed stocks for the process of this invention comprises fluid hydrocarbon mixtures containing at least 75% by weight of aromatics. One source of such feeds is found in the debutanized or depentanized effluents from reforming reactors. Another source of such feeds is derived from pyrolysis process having light olefins as the principal product. A byproduct liquid is produced from these pyrolysis processes which is rich in aromatics. Generally, it is necessary to hydrotreat these pyrolysis liquids to saturate diolefins, olefins, etc. and to remove contaminants. Usually a small portion of the aromatics will also be hydrotreated to form naphthenes. Whatever the source, feed stocks in the carbon number range of from about C<sub>6</sub> to about C<sub>20</sub> are suitable for use in the process of this invention. Desirably, the feed stock is in the C<sub>6</sub> to C<sub>10</sub> range and preferably within the C<sub>6</sub> to C<sub>8</sub> range.

Although the process of this invention is applicable to many solvents having a selectivity for aromatics such as the glycols, sulfoxides, pyrrolidones, etc., preferably the solvent is a sulfolane-type. By this I mean that the solvent possesses a 5 membered ring, one atom of which is sulfur, the other four being carbon and having two oxygen atoms bonded to the sulfur atom. Generically, these solvents may be shown as having the following structural formula:



wherein R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> and R<sub>4</sub> are independently selected from the group consisting of a hydrogen atom, an alkyl group having up to 10 carbon atoms, an alkoxy radical having up to 8 carbon atoms and an arylalkyl radical having up to 12 carbon atoms. Other solvents preferably included within this process are the sulfolenes such as 2-sulfolene or 3-sulfolene which have the following structure:



The sulfolane solvents of this invention 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. A 2-sulfolene may be made similarly by isomerizing instead of hydrogenating the product resulting from condensing the conjugated diolefin with sulfur dioxide. Other useful solvents which have high selectivity

7  
for separating aromatics from non-aromatic hydrocarbons and may be employed in the process of this invention are 2-methylsulfolane, 2,4-dimethyl-sulfolane, methyl 2-sulfonyl ether, n-aryl-3-sulfonyl amine, 2-sulfonyl acetate, diethylene glycol, various polyethylene glycols, dipropylene glycol, various polypropylene glycols, dimethyl sulfide, N-methyl pyrrolidone and others.

The aromatic selectivity of the sulfolane solvents can be further enhanced by the addition of water to the solvent. Preferably the present solvents contain a small amount of water dissolved therein to increase the selectivity of the overall solvent phase for aromatic hydrocarbons over non-aromatic hydrocarbons without reducing substantially the solubility of the solvent phase for aromatic hydrocarbons. The presence of water in the solvent composition furthermore provides a relatively volatile material therein which is distilled from the fat solvent in the extractive stripper to vaporize the last traces of non-aromatic hydrocarbon from the fat solvent stream by steam distillation. The solvent composition preferably contains from about 0.5% to about 20% by weight of a diluent such as water preferably from about 5% to about 15% depending on the particular sulfolane solvent utilized and the process conditions at which the extractor and extractive stripper are operated.

The extractor is operated at elevated temperature and at a sufficiently elevated pressure to maintain the feed stock, solvent and light backwash streams in the liquid phase. Suitable temperatures are within the range of from 80° F. to about 400° F. and preferably from about 200° to about 300° F. Suitable pressures are within the range of from about atmospheric pressure up to about 400 p.s.i.g. and preferably from about 50 p.s.i.g. to about 150 p.s.i.g. It is preferable that the volume of backwash material introduced into the lower point in the extractor be at least 10% by volume of the extract phase leaving the extractor in order to effectively displace the heavier non-aromatic hydrocarbons from the extract.

The extractive stripper is operated at moderate pressures and sufficiently high reboiler temperatures to drive all the light backwash non-aromatic components and some of the aromatics water and solvent overhead. Typical stripper pressures are from atmospheric pressure to about 100 p.s.i.g. although generally the top of the stripper is preferably maintained at from about 1 p.s.i.g. up to about 20 p.s.i.g. The reboiler temperature is dependent upon the composition of the feed stock and the solvent. Generally for a C<sub>6</sub> to C<sub>9</sub> feed stock using saturated sulfolane, stripper bottom temperatures of from 275 to about 360° F. are satisfactory.

The solvent recovery column is operated at low pressures and sufficiently high temperatures to drive the aromatic hydrocarbons overhead and thus producing a lean solvent bottoms stream. Again the choice of operating conditions depends on the feed stock and the solvent composition. Preferably the top of the solvent recovery column is operated at vacuum of from about 100 to about 400 mm. mercury absolute. Low pressures must be employed in order to maintain a sufficiently low reboiler temperature to avoid thermal decomposition of the solvent. Preferably the reboiler temperature should be maintained below about 360° F. when using saturated sulfolane as the solvent.

The apparatus employed in the process of this invention may be any conventional convenient type known to those skilled in the art. For simplicity the drawing does not show all the pumps, tanks, heat exchangers, valves, bypasses, vents, reboilers, condensers, coolers, control valves, means for actuating control valves and other auxiliaries that may be necessary for the proper operation of the process but the inclusion of which will be evident to those skilled in the art.

The following example is presented to further illustrate the process of this invention but it is not intended to limit the invention to the materials used nor the operating conditions disclosed therein.

A hydrotreated ethylene pyrolysis co-product primarily in the C<sub>6</sub> boiling range is employed as the feed stock. The fresh feed is introduced into flow conduit 1 and thereafter into the intermediate point of extractor 2 at a rate of 75.9 moles/hr. consisting of 61.2 moles of benzene, 1.4 moles of toluene, and 13.3 moles of non-aromatics. The concentration of aromatics in the fresh feed is about 78% by weight. About 85% of the non-aromatic fraction consists of naphthenes. Saturated sulfolane containing about 5.2 mole percent water is introduced into the upper point of extractor 2 at a rate of 81.2 moles/hr. Light backwash containing about 30 mole percent naphthenes is introduced into the lower point of extractor 2 at a rate of 78.4 moles/hr. The backwash contains about 99 mole percent C<sub>6</sub>'s and the remainder primarily C<sub>7</sub>'s. A total of 27.4 moles/hr. of raffinate phase is withdrawn from flow conduit 23, of which 13.7 moles/hr. of raffinate passes through flow conduit 37, pump 36 and returns to flow conduit 1. The remaining net raffinate phase is withdrawn from extractor 2 through flow conduit 23 at a rate of 13.7 moles/hr. (solvent free basis) and contains 0.3 mole of benzene, 0.1 mole toluene, 23.2 moles of non-aromatics and 80.9 moles per hour of wet solvent. An additional 470.4 moles/hr. of lean solvent is mixed with the fat solvent and the resulting mixture is introduced into extractive stripper 4. An overhead stream is withdrawn from stripper 4 and into receiver 8 at a rate of 78.4 moles/hr. and contains 41.3 moles of benzene, 0.6 mole of toluene, and 36.5 moles of non-aromatics. This overhead material is condensed and the hydrocarbon phase is returned to the lower point in extractor 2 through flow conduit 11. A net bottoms stream is withdrawn from stripper 4 at a rate of 604.6 moles/hr. and sent directly to the solvent recovery column. An overhead hydrocarbon phase is withdrawn from receiver 17 through flow conduit 19 at a rate of 62.2 moles/hr. and contains 60.9 moles of benzene, and 1.3 moles of toluene.

The extractor is a rotary disc contactor and is maintained at a pressure of about 105 p.s.i.g. and a temperature of about 120° F. The stripper is maintained at a top pressure of about 3 p.s.i.g., a top temperature of about 230° F. and a reboiler temperature of about 320° F. The solvent recovery column is maintained at a top pressure of about 375 mm. mercury absolute, a top temperature of about 120° F. and a reboiler temperature of about 320° F.

It is expected that without the use of the light backwash stream there would be an appreciable concentration of non-aromatics in the stream withdrawn from flow conduit 43. It is also expected that without the recycle of a portion of the raffinate phase back to the feed inlet, the feed could not be directly introduced into the extractor and the light backwash stream could not be used without providing additional equipment.

I claim as my invention:

1. A process for the separation and recovery of aromatic hydrocarbons from a hydrocarbon mixture having an aromatics content of at least 75% by weight which comprises:

commingling the mixture with a sufficient amount of raffinate phase from a source hereinafter described to produce an overall mixture which is immiscible with a lean sulfolane type solvent;  
introducing a lean sulfolane solvent into an upper point in the height of an extraction zone;  
introducing the overall mixture into an intermediate point in the height of the extraction zone and counter-currently contacting said overall mixture therein with the sulfolane solvent at an elevated temperature and at a pressure sufficient to maintain said solvent and said mixture in substantially liquid phase to form a liquid raffinate phase and a liquid extract phase;  
removing the raffinate phase from the upper portion of said zone and recycling a sufficient portion of the raffinate phase back to said hydrocarbon mixture to produce said overall mixture;

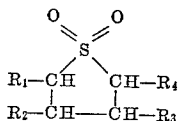
countercurrently contacting the extract phase in the lower portion of said zone with a light hydrocarbon which boils in the presence of the solvent at a temperature below the boiling point of the aromatic hydrocarbon being extracted but which remains in the liquid state at said temperature and pressure, said light hydrocarbon being introduced into a lower point of the extraction zone in sufficient amount to replace substantially all of the non-aromatic hydrocarbons of said mixture in the extract phase with said light hydrocarbons;

removing from the lower portion of said zone the extract phase containing the light hydrocarbon, introducing the extract phase into a stripper column, removing an overhead stream from said column comprising the light hydrocarbon and returning the light hydrocarbon to the lower point of the extraction zone;

removing a fat solvent stream comprising aromatic hydrocarbons and solvent from the bottom of the stripper column, introducing the column bottoms into a fractionating tower, removing a tower overhead stream therefrom comprising aromatics and a lower bottoms stream therefrom comprising lean solvent; and

returning at least a portion of the tower bottoms stream to the upper point of the extraction zone.

2. The process of claim 1 further characterized in that the solvent comprises a sulfolane of the general formula:



wherein R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> and R<sub>4</sub> are independently selected from the group consisting of a hydrogen atom, an alkyl group having up to 8 carbon atoms and an arylalkyl radical having up to 12 carbon atoms.

3. The process of claim 2 further characterized in that R<sub>1</sub>, R<sub>2</sub>, R<sub>3</sub> and R<sub>4</sub> are hydrogen atoms.

4. The process of claim 3 further characterized in that the sulfolane solvent contains up to 20% by volume of water.

5. The process of claim 1 further characterized in that the solvent comprises a sulfolene selected from the group consisting of 2-sulfolene and 3-sulfolene.

6. The process of claim 1 further characterized in that the unrecycled remaining portion of the raffinate phase is passed into a splitter fractionator wherein the remaining portion is separated into a light raffinate fraction and a heavy raffinate fraction and at least a portion

of the light raffinate fraction is returned to the lower point in the extraction zone.

7. The process of claim 1 further characterized in that a portion of the tower bottoms stream is returned to the upper point of the extraction zone and the other portion of the tower bottoms is mixed with the extract phase prior to entering the stripper column.

8. The process of claim 3 further characterized in that the light hydrocarbon comprises a fluid having from about 5 to about 7 carbon atoms per molecule.

9. The process of claim 3 further characterized in that the intermediate point in the extraction zone is located about 1/3 of the way along the vertical height from the bottom.

10. In a solvent extraction process for the separation and recovery of aromatic hydrocarbons in an extract phase, and a raffinate phase from a hydrocarbon feed mixture wherein the concentration of aromatics in the feed is sufficiently high to produce miscibility between the solvent and the feed, the improvement which comprises commingling with the feed a sufficient amount of non-aromatic hydrocarbons to reduce the aromatics concentration of the overall mixture to the point where said overall mixture is no longer completely miscible with the solvent, introducing said overall mixture directly into an extraction zone wherein the overall mixture is countercurrently contacted with the solvent, recovering from the extraction zone an extract phase and a raffinate phase, and recycling a sufficient amount of the raffinate phase to said feed to maintain the non-miscibility of said overall mixture as aforesaid.

11. The improved process of claim 10 further characterized in that the solvent comprises saturated sulfolane and the aromatic concentration in the hydrocarbon feed mixture is at least 75% by weight.

12. The improved process of claim 11 further characterized in that the aromatics concentration in the overall mixture is less than 75% by weight.

#### References Cited

##### UNITED STATES PATENTS

2,407,820	9/1946	Durum	260-674
2,770,663	11/1956	Grote	260-674
2,908,726	10/1959	Hutson	260-674

DELBERT E. GANTZ, *Primary Examiner*.

C. E. SPRESSER, JR., *Assistant Examiner*.

U.S. Cl. X.R.

208-312, 321, 325