United States Patent [19]
Kubek et al.

[11] **3,779,904**
[45] **Dec. 18, 1973**

[54] PROCESS FOR THE SEPARATION OF AROMATIC HYDROCARBONS FROM A MIXED HYDROCARBON FEEDSTOCK

[75] Inventors: **Daniel John Kubek**, North Tarrytown; **Alexander Jean-Marie Kosseim**, Yorktown Heights; **George Solomon Somekh**, New Rochelle, all of N.Y.

[73] Assignee: **Union Carbide Corporation**, New York, N.Y.

[22] Filed: **Dec. 21, 1971**

[21] Appl. No.: **210,404**

[52] **U.S. Cl.** .... **208/333**, 208/321, 208/323, 260/674 SE
[51] **Int. Cl.** **C10g 21/16**, C07c 7/10
[58] **Field of Search** .... 260/674 SE; 208/321, 208/323, 333

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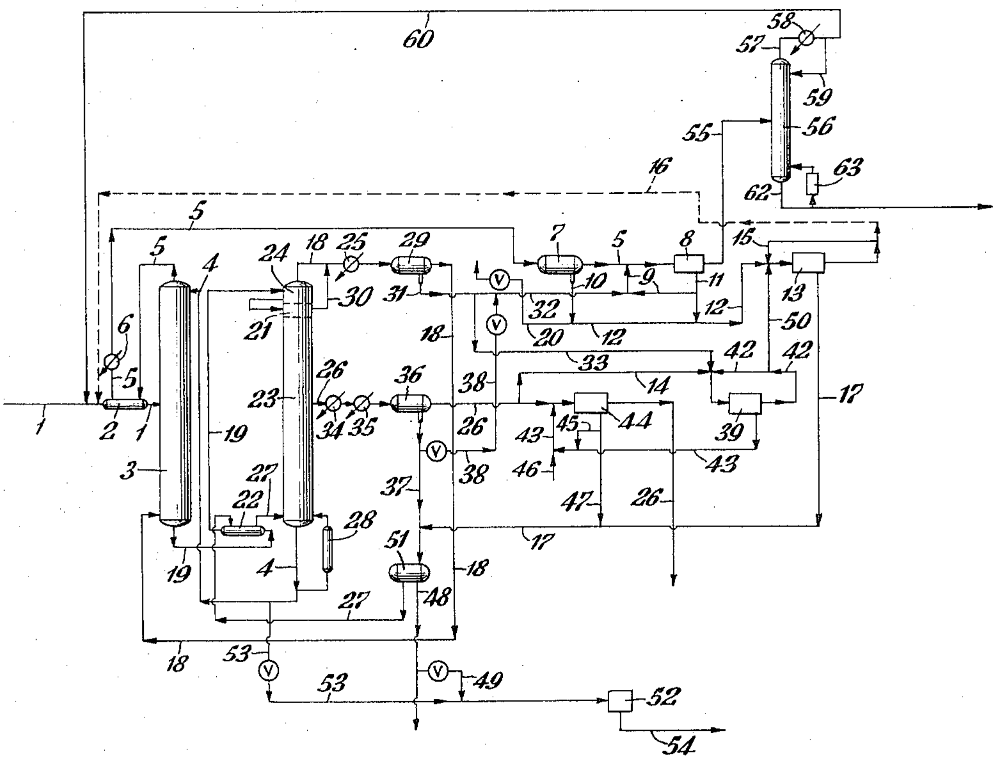
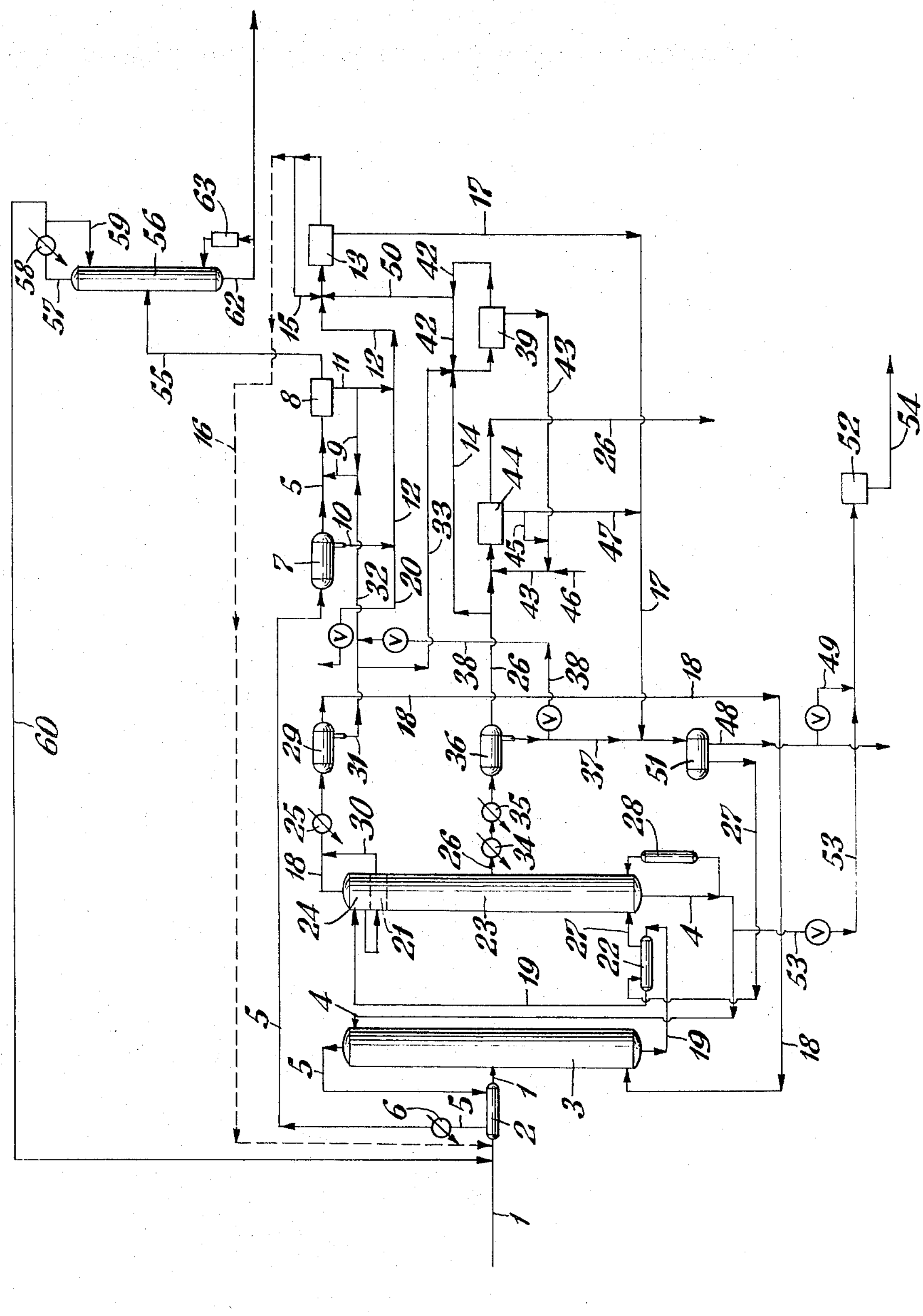
Primary Examiner—Delbert E. Gantz
Assistant Examiner—C. E. Spresser
Attorney—Paul A. Rose et al.

[57] ABSTRACT

The process involves a combination of continuous solvent extraction-steam distillation for the recovery of aromatic hydrocarbons from a mixed feedstock. The feedstock is contacted with a solvent-water mixture at temperatures in the range of about 75° to 200°C and the extract and raffinate streams are separated into their components. The purity of the aromatics recovered from the extract is improved by introducing light aliphatic hydrocarbons consisting essentially of aliphatic and cycloaliphatic hydrocarbons of no more than five carbon atoms with the feedstock at the middle theoretical stage of the extraction column. The light aliphatics are introduced in amounts in the range of 5 to 12 percent, based on the weight of the feedstock.

16 Claims, 1 Drawing Figure

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PROCESS FOR THE SEPARATION OF AROMATIC HYDROCARBONS FROM A MIXED HYDROCARBON FEEDSTOCK

FIELD OF THE INVENTION

This invention relates to an improvement in a process for the separation of aromatic hydrocarbons from a fixed hydrocarbon feedstock and, more particularly, to the recovery of high purity aromatic hydrocarbons in high yields while making efficient use of process components.

DESCRIPTION OF THE PRIOR ART

With the advent of the benzene-toluene-C_8 aromatics fraction (known and hereinafter referred to as BTX) as the principal raw material in the manufacture of petrochemicals, outstripping ethylene in this regard, and the increased demand for aromatics as a component in gasoline to increase its octane rating and thus reduce or eliminate the need for lead, which has been under fire as a pollutant, aromatics separation processes availed of in the past have come under close scrutiny with an eye toward improving process economics.

Improved economics can be translated into, among other things, the lowering of heating requirements and the more effective use of process components as aids in the separation process.

Various processes have been used for aromatics separations in systems of the single extractor-single distillation column (or stripper) type, which have one particular step, among others, in common, i.e., the reflux hydrocarbons are derived from the distillation of the extract and are recycled into the lower portion of the extraction column below the bottom plate or below the lowest theoretical stage. To improve the purity of the aromatic products, lower boiling aliphatics, not precisely the light aliphatics defined below, have been added to this reflux. These lower boiling aliphatics were obtained from fractionation of the feedstock, raffinate, and/or reflux and always introduced with or as a part of the reflux at the bottom of the extractor.

While purities have been improved and heating requirements lowered using this technique, optimization has not been achieved.

SUMMARY OF THE INVENTION

An object of this invention, therefore, is to improve the reflux in aqueous solvent single extractor-single distillation column systems whereby heating requirements are reduced and the purity of the aromatics is raised to previously unattained levels.

Other objects and advantages will become apparent hereinafter.

According to the present invention, high purity aromatic hydrocarbons are effectively recovered using minimal heat in a continuous solvent extraction-distillation process for the recovery of aromatic hydrocarbons having boiling points in the range of about 80°C. to about 175°C. from a feedstock containing aliphatic hydrocarbons and at least about 40 percent by weight, based on the weight of the feedstock, of aromatic hydrocarbons wherein the feedstock contains no more than about 4 percent by weight, based on the weight of the feedstock, of light aliphatics consisting essentially of aliphatic and cycloaliphatic hydrocarbons, each having no more than 5 carbon atoms, a boiling point no higher than about 50°C., and being condensable at a pressure of nc more than about 3 atmospheres and at a temperature of no less than about 50°C.; a single extraction column having about 3 to about 25 theoretical stages is used for the solvent extraction to provide an extract; and a single distillation column is used to distill the extract to provide a mixture of the aromatic hydrocarbons comprising the following steps:

a. introducing the feedstock into the extraction column at the middle theoretical stage thereof;

b. contacting the feedstock in the extraction column with a mixture of water and a solvent, said solvent being a water-miscible organic liquid having a boiling point of at least about 200°C. and having a decomposition temperature of at least about 225°C., and with reflux hydrocarbons introduced into the extraction column below the bottom theoretical stage thereof to provide an extract comprising aromatic hydrocarbons, reflux aliphatic hydrocarbons, solvent, and water and a raffinate comprising essentially aliphatic hydrocarbons;

c. introducing the extract into the distillation column to separate the aromatic hydrocarbons and the reflux hydrocarbons from the extract;

d. recycling the reflux hydrocarbons from step (c) to the extraction column as provided in step (b); and

e. recovering the aromatic hydrocarbons of step (d);

the improvement comprising introducing a sufficient amount of the light aliphatics defined above into the extraction column at the middle theoretical stage thereof to provide a total percent by weight of said light aliphatics, based on the weight of the feedstock, in the range of about 5 percent to about 12 percent.

BRIEF DESCRIPTION OF THE DRAWING

The sole FIGURE is a schematic flow diagram of an illustrative embodiment of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

As noted, there is an industrial need for BTX, which is available in high proportion,e.g., at least about 40 percent by weight, in a wide variety of hydrocarbon feedstocks such as reformed gasolines; coke oven light oils; cracked gasolines; and dripolenes, which, after hydrogenation, can contain as much as 70 to 98 percent BTX. These feedstocks also contain both aliphatic and cycloaliphatic hydrocarbons (herein sometimes referred to collectively as aliphatic hydrocarbons). Since the individual hydrocarbon compounds which make up these feedstocks are well known, they will not be discussed extensively; however, it can be pointed out that the major components of the feedstocks used herein are hydrocarbons with boiling points ranging from 25°C. to 175°C. including straight-chain and branched-chain paraffins and naphthenes, such as n-heptane, isooctane, and methyl cyclohexane, and aromatics such as BTX.

The BTX fraction can include benzene, toluene, the C_8 aromatics including ortho-xylene, meta-xylene, para-xylene, and ethyl benzene, and C_9 aromatics, which, if present at all, usually appear in the smallest proportion in relation to the other components.

It is also important to point out that conventional feedstocks used in extraction-distillation systems of the type discussed herein for the recovery of BTX contain about 1 to 3 percent and, in some cases, up to about 4 percent by weight based on the weight of the feedstock, of light aliphatics which are defined herein as aliphatic and cycloaliphatic hydrocarbons having no more than 5 carbon atoms in each compound, said compound having a boiling point no higher than about 50°C. and being condensable at a pressure of no more than 3 atmospheres and a temperature of no less than 50°C. (hereinafter referred to as the "defined light aliphatics").

These defined light aliphatics provide the crux of subject improvement since it has been found that when at a particular percentage level and when introduced at a particular point in the extraction column, they enhance the reflux to a point where there is a noticeable and advantageous drop in heating requirement together with a higher level of purity.

The use of this improvement is found to be advantageous in any process falling within the process definition set out heretofore under the summary of the invention; however, it is found to be particularly advantageous when applied to the preferred embodiment described hereinafter.

The process described here, exclusive of subject improvement, is the subject of our copending application Ser. No. 180,996, filed on Sept. 16, 1971, now U.S. Pat. No. 3,714,033. This application bears the same title as the instant application and is incorporated by reference herein.

A typical breakdown of the defined light aliphatic fraction, in percent by weight based on the total weight of the fraction, is about 0 percent to about 95 percent of the full range of C_5 aliphatics, about 0 percent to about 5 percent of C_4 aliphatics, and a small proportion, about 0 percent to about 1 percent, of C_1 to C_3 aliphatics.

The solvents used in the subject process are, as described above, water-miscible organic liquids, (at process temperatures) having a boiling point of at least about 200°C. and having a decomposition temperature of at least about 225°C. The term "water-miscible" includes those solvents which are completely miscible over a wide range of temperatures and those solvents which have a high partial miscibility at room temperature since the latter are usually completely miscible at process temperatures. The solvents are also polar and are generally comprised of carbon, hydrogen and oxygen with some exceptions. Examples of solvents which may be used in the process of this invention are dipropylene glycol, tripropylene glycol, dibutylene glycol, tributylene glycol, ethylene glycol, diethylene glycol, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, diethylene glycol monomethyl ether, diethylene glycol monoethyl ether, sulfolane, N-methyl pyrrolidone, triethylene glycol, tetraethylene glycol, ethylene glycol diethyl ether, propylene glycol monoethyl ether, pentaethylene glycol, hexaethylene glycol, and mixtures thereof. The preferred group of solvents is the polyalkylene glycols and the preferred solvent is tetraethylene glycol.

Additional solvents, which may be used alone or together, or with the aforementioned solvents are amides such as formamide, acetamide, dimethylformamide, diethylformamide, and dimethylacetamide; amines such as diethylenetriamine and triethylenetetramine; alkanolamines such as monoethanolamine, diethanolamine, and triethanolamine; nitriles such as beta,beta^1-oxydipropionitrile and beta,beta^1-thiodipropionitrile; phenol and the cresols; the methyl sulfolanes; sulfoxides such as dimethyl sulfoxide and diethyl sulfoxides; lactones such as gamma-propiolactone and gamma-butyrolactone.

The apparatus used in the process both for the main extraction and distillation is conventional, e.g., an extraction column of the multistage reciprocating type containing a plurality of perforated plates centrally mounted on a vertical shaft driven by a motor in an oscillatory manner can be used as well as columns containing pumps with settling zones, sieve trays with upcomers, or even a hollow tube while the distillation can be conducted in a packed or bubble plate fractionating column. Countercurrent flows are generally utilized in both extraction and distillation columns.

Heat exchangers, decanters, reservoir, solvent regenerator, and raffinate still are also conventional as well as various extractors other than the main extractor. These other extractors are preferably single stage mixer-settlers, but can be any of the well known types.

The number of theoretical stages in the extraction column can be about three to about 25 stages and is preferably about five to about 12 stages. The "middle theoretical stage" of the column is defined to include the theoretical stages running from 0.25 times the theoretical stages in the column plus one to 0.75 times the theoretical stages in the same column, e.g., in a column having four theoretical stages, the "middle theoretical stage" would include, as defined herein, the second and third theoretical stages and in a column having 24 theoretical stages, the "middle theoretical stage" would include the seventh to the 18th theoretical stages, inclusive. It will be recognized by those skilled in the art that the number of theoretical stages is to be considered as based on functioning theoretical stages and that the bottom theoretical stage is located just above the reflux inlet no matter what the actual physical layout of the column is. The feed and the defined light aliphatics can be introduced anywhere in the middle theoretical stage; however, it is preferred that both be introduced at the same point in the middle theoretical stage to obtain the full benefits of the process.

The solvent is used as an aqueous solution thereof containing water in an amount of about 1 percent to about 8 percent by weight based on the weight of the solvent and preferably containing water in an amount of about 2 percent to about 5 percent by weight. This aqueous solution is referred to hereafter in some instances as a solvent-water mixture.

Generally, to accomplish the extraction, the ratio of solvent (exclusive of water) to feedstock in the extractor is in the range of about 4 to about 8 parts by weight of solvent to one part by weight of feedstock. This broad range can be expanded upon where non-preferred solvents are used. A broad range of about 3 to about 12 parts by weight of solvent to one part by weight of feedstock and a preferred range of about 5 parts to about 7 parts of solvent per part of feedstock can be used successfully for the solvent of preference and other like solvents. In final analysis, however, the ratio is selected by the technician based on experience with the particular feedstock and depends in part upon whether high recovery or high purity is being emphasized, although the instant process will improve purity in any case.

The reflux to the extraction zone is generally made up of about 20 percent to about 50 percent by weight aliphatics having from five to seven carbon atoms and about 50 percent to about 80 percent by weight aromatics, both based on the total weight of the reflux. The reflux, percentages, and ratios discussed in this paragraph are not considered to include and do not apply to the defined light aliphatics, although the latter might be referred to as an auxiliary reflux. There is some overlap in composition, however, in that some of the components which make up the reflux are the same as some of the defined light aliphatics. These similar components in the reflux should not be included when determining whether the percentage of defined light aliphatics meets the stated requirements. In any event, the sources, other than the feedstock, are different for both in that the major source of the reflux is the distillate from the main distillation column while the source of the defined light aliphatics is from raffinate distillation or from outside the system. The ratio of reflux to feed-stock in the extraction zone is, generally, maintained in the range of about 0.5 to about 1.5 parts by weight of reflux to one part by weight of feedstock and preferably about 0.6 to about 1.25 parts by weight of reflux to one part by weight of feedstock, but, again, is selected by the technician just as the ratio of solvent to feedstock. The reflux aliphatics pass into the extract rather than being taken overhead with the raffinate and are recycled to the extractor from the reflux decanter as will be seen hereinafter.

The temperature in the extraction zone is maintained in the range of about 100°C. to about 200°C. and is preferably in the range of about 125°C. to about 150°C., especially for the solvent of preference.

The pressure in the extraction zone is maintained in the range of about 75 psig. to about 200 psig. As is well known in the art, however, one selected pressure is not maintained throughout the extraction zone, but, rather, a high pressure within the stated range is present at the bottom of the zone and a low pressure again within the stated range is present at the top of the zone with an intermediate pressure in the middle of the zone. The pressures in the zone depend on the design of the equipment and the temperature, both of which are adjusted to maintain the pressure within the stated range.

The temperature at the top of the distillation zone, which, in terms of the apparatus used, may be referred to as a distillation column or stripper, is at the boiling point of the mixture of aromatics present in the zone while the temperature at the bottom of the stripper is generally in the range of about 135°C. to about 200°C.

The pressure at the top of the stripper, an upper flash zone in this case, is in the range of about 20 psig to about 35 psig. In a lower flash zone just beneath the upper flash zone and connected thereto, the pressure is in the range of about 10 psig to about 20 psig and is about 10 or 15 psig lower than the pressure in the upper flash zone. The pressure in the rest of the distillation zone is maintained in the range of about 15 psig to about 25 psig with some variation throughout the zone.

The steam brought into the bottom of the distillation zone enters at a temperature of about 100°C. to about 150°C. and is under a pressure of about 15 psig to about 25 psig. The total water present in the distillation column is essentially in vapor form and is generally in the range of about 0.1 parts to about 0.5 parts by weight of water to one part by weight of aromatics in the zone and preferably in the range of about 0.1 parts to about 0.3 parts by weight of water to one part by weight of aromatics. The water used for the steam may be called stripping water. A small amount of water is present in liquid form in the distillation zone dissolved in the solvent.

Referring to the drawing:

The feedstock and defined light aliphatics are introduced through line **1** into heat exchanger **2** where they are preheated to a temperature in the range of about 50°C. to about 150°C. The mixture then continues through line **1** to enter extractor **3** at the middle tray thereof, which is the equivalent of the middle theoretical stage. An aqueous solvent solution having a temperature in the range of about 125°C. to about 175°C. enters at the top tray of extractor **3** through line **4** and percolates down the column removing aromatics from the feedstock.

The raffinate, essentially free of aromatics, leaves the top of the column and passes through heat exchanger **2** where it is used to preheat the feedstock and is cooled in turn to a temperature in the range of 75°C. to about 125°C. The raffinate comprises about 95 percent to about 98 percent by weight aliphatics, about 1 percent to about 3 percent by weight dissolved and entrained solvent, and about 0 percent to about 3 percent by weight aromatics. The raffinate then passes through cooler **6** where it is further cooled to about 25°C. to about 50°C. and proceeds along line **5** to decanter **7** where it separates into two phases, an aliphatic hydrocarbons phase and a solvent phase, the solvent being contaminated with aliphatics.

It should be noted that the "phase" is named after its main component, which is present in the phase in an amount of at least 50 percent by weight and, in most cases, in an amount of at least 90 percent by weight.

The aliphatic hydrocarbons phase, which can still be referred to as the raffinate, now contains about 96 percent to about 99 percent by weight aliphatics, about 0 percent to about 1 percent by weight dissolved and entrained solvent, and about 0 percent to about 3 percent by weight aromatics. The solvent phase, on the other hand, contains about 90 percent to about 96 percent by weight solvent, about 2 percent to about 5 percent by weight water, and about 2 percent to about 4 percent by weight aliphatics.

The raffinate continues overhead through line **5** into raffinate extractor **8**, which can be a single stage mixer-settler or other conventional type of extractor.

The solvent phase passes through line **10** to join line **12** referred to hereinafter or it can be optionally recycled to the top of extractor **3** along line **20** (connection to extractor not shown).

The raffinate is washed with a portion of the water phase from reflux decanter **29** and separated in raffinate extractor **8** into an aliphatic hydrocarbons phase (still called the raffinate) which is essentially free of solvent and water and contains about 97 percent to about 100 percent by weight aliphatics and about 0 percent to about 3 percent by weight aromatics, and a raffinate water phase as bottoms which contains about 75 percent to about 90 percent by weight water, about 10 percent to about 25 percent by weight solvent, and about 0.1 percent to about 1 percent by weight aliphatics.

The raffinate proceeds along line **55** to raffinate still **56** where the defined light aliphatics are removed as a vapor overhead and pass through condenser **58.** A portion of the condensate is returned to the top tray of raffinate still **56** as a reflux which aids in purifying the lights by knocking down the high boilers. The heavier aliphatics are removed as bottoms and pass through line **62** where a portion is diverted through reboiler **63** and returns to raffinate still **56** below the bottom tray as a vapor to provide most of the heating requirement. The balance of the heavy aliphatics proceeds along line **62** to storage (not shown).

The balance of the condensate proceeds along line **60** where it joins line **1** thus supplying a high proportion of the defined light aliphatics for the process. This raffinate distillation is the preferred and most satisfactory mode for supplying the bulk of the defined light aliphatics for the process. About 50 percent to about 100 percent by weight of the defined light aliphatics can be supplied in this manner although initially the necessary defined light aliphatics must be provided from an outside source (not shown) to line **1** and make-up defined light aliphatics may be added from time to time to maintain the required level. The raffinate still is similar to stripper **23** except that it can be half the height and half the diameter thus reducing its cost and heating requirements. The temperature in the bottom of the raffinate still is maintained in the range of about 45 °C. to about 200 °C., the pressure is maintained in the range of about 5 psig to about 40 psig, and the reflux ratio is about 0.5:1 to about 3.0:1.

Part of the raffinate water phase can optionally be recirculated through extractor **8** via line **11**, line **9** and line **5** as shown. This recirculation is conventional with a mixer-settler arrangement, but may not be advantageous with other types of extractors. As noted, this water phase still contains, along with the water and solvent, a small amount of aliphatics. All of the balance of the water phase is, therefore, directed from line **11** along line **12** to extractor **13**, which can again be a single-stage mixer-settler.

Feeding into line **12** via line **50** is an aromatics slipstream, which at its source (see line **14**) is an essentially pure stream of aromatics, i.e., having a purity of at least 95 percent by weight, or in other words, at least 95 percent by weight of the slipstream is aromatic hydrocarbons. The purity of the slipstream is preferably about 98 percent and for optimum performance, i.e., to obtain the highest purity product, about 99 percent or even higher. It is called a slipstream or sidestream because the amount of aromatics fed into the water phase passing through line **12** is very small. The amount of slipstream aromatic hydrocarbons used in the process is in the range of about 0.1 percent to about 5 percent by weight of the aromatic hydrocarbons in the feedstock and is preferably in the range of about 0.5 percent to about 2.0 percent by weight of such aromatic hydrocarbons. The slipstream washes the water in extractor **13** to remove the small amount of aliphatics, which is so detrimental to the efficiency of the process. This aromatics slipstream can be recycled along line **15** through extractor **13** to further wash the water phase where a mixer settler extractor is used and it is then, preferably, sent along line **16** to line **1** where it is reintroduced into the feedstock and passes into the system once more. The water, which is essentially devoid of aliphatics, but contains solvent, then passes as bottoms from extractor **13** through line **17** and into water reservoir **51** via line **37**.

Returning to extractor **3**, it has been noted above that the aqueous solvent percolates down the column carrying with it the aromatics. In the middle of the column, the aqueous solvent comes into contact with both the feedstock and the defined light aliphatics. Although the mechanism involved is not clear it is believed that the defined light aliphatics initially build up in the extract until a saturation point is reached and then they pass into the raffinate. The effect of the defined light aliphatics once the saturation point is reached is to reduce the amount of aliphatics which pass into the extract or, in other words, to improve the selectivity of the solvent solution and enhance the activity of the reflux. In the lower half of extractor **3**, the solvent solution of aromatics comes into countercurrent contact with a reflux liquid, which enters extractor **3** below the bottom tray (or theoretical stage) along line **18**. The reflux percolates up the lower half of extractor **3** progressively dissolving in and purifying the solvent solution of aromatics. The solution which is formed, i.e., the extract, comprises about 10 percent to about 20 percent by weight feedstock aromatics, about 2 percent to about 5 percent by weight water, about 65 percent to about 75 percent by weight solvent, about 4 percent to about 8 percent by weight reflux aromatics, and about 3 percent to about 6 percent by weight reflux aliphatics, all based on the total weight of the extract.

The extract leaves the bottom of extractor **3** through line **19** and passes through heat exchanger **22** where it is cooled to a temperature in the range of about 100°C. to about 125°C. The extract proceeds along line **19** and enters stripper **23**, the distillation zone, at upper flash chamber **24**, which, as noted heretofore, is at a lower pressure than the extractor. Part of the extract flashes on entering the flash chamber and is taken overhead through line **18** in vapor form. Another part of the extract passes as a liquid into lower flash chamber **21**, which is operated at an even lower pressure and further flashing occurs. These flashed vapors join the fractionated vapors and pass through line **30** to join the vapors passing through line **18**. The balance of the extract (at least about 80 percent by weight) percolates down the column into the fractionation zone where it comes into countercurrent contact with the stripping vapors, i.e., steam, and more vapors are generated. A part of the vapor rises to the top of the column and mixes with the flashed vapors in flash chamber **21** as noted. The overhead distillate comprises about 40 to about 75 percent by weight aromatics, about 20 to about 40 percent aliphatics, about 2 percent to about 10 percent by weight water, and about 0 percent to about 5 percent by weight solvent, all based on the total weight of the overhead distillate.

After the aqueous solvent descends about halfway down the column, it becomes essentially free of aliphatics. At this point, a vapor side-stream distillate is removed through line **26**. The side-stream distillate is comprised of about 65 to about 90 percent by weight aromatics, about 10 to about 30 percent by weight water, and about 1 percent to about 10 percent by weight of solvent, based on the total weight of the side-stream distillate.

The bulk of the solvent and water solution, an amount equal to over 98 percent by weight of the solvent and water entering stripper **23** through line **19**, leaves the bottom of stripper **23** through line **4**. A portion of this solution is diverted into reboiler **28** and returns as a vapor to a point below the bottom tray of stripper **23** to provide most of the stripper's heating requirements. The balance of the water and solvent extraction is recycled to the top tray of extractor **3** through line **4.** Recycled stripping water containing some dissolved solvent enters stripper **23** through line **27** from water reservoir **51** after essentially all of it is converted in heat exchanger **22** to steam.

Returning to the overhead distillate mentioned heretofore, such overhead distillate is a combination of flashed vapors and fractionated vapors having the aforementioned composition. This overhead distillate is also known as a reflux distillate. The vapor is first condensed and cooled to between about 38°C. and 94°C. in reflux condenser **25.** The condensate then passes into reflux decanter **29** where a reflux hydrocarbons phase is decanted from a water phase. The reflux hydrocarbons phase comprises about 20 to 50 percent by weight aliphatics having from five to seven carbon atoms, and about 50 to about 80 percent by weight aromatics and is recycled as reflux through line **18** to extractor **3** as previously described.

The water phase contains about 95 to about 99 percent by weight water, about 0 to about 5 percent by weight solvent, and about 0.1 to about 0.5 percent by weight aliphatics. It passes through line **31** and is split in two streams, lines **32** and **33**, a raffinate wash stream and an aromatics wash stream, respectively. These washes can take place as shown by splitting the stream or the entire stream can be used to wash the raffinate first and then the aromatics providing that the water is treated with an aromatics slipstream before the aromatics wash.

As noted heretofore, the side-stream distillate is withdrawn in vapor form from stripper **23** through line **26** and condensed in aromatics condenser **34** and further cooled to a temperature in the range of about 25°C. to about 50°C. in cooler **35**, which can be a heat exchanger or other type of cooling device. The condensate then passes into aromatics decanter **36** where an aromatic hydrocarbons phase containing about 99.8 to about 99.9 percent by weight aromatics, and about 0.1 to about 0.2 percent by weight solvent and a water phase containing about 90 percent to about 98 percent by weight water, about 2 percent to about 10 percent by weight solvent, and about 0.1 percent to about 0.5 percent by weight aromatics are formed. The water phase passes through line **37** to water reservoir **51.** Optionally, all of part of the water phase can be directed through valved line **38** to join line **32** for use as raffinate wash.

The aromatic hydrocarbons phase proceeds from decanter **36** through line **26** along which an aromatics slipstream is taken through line **14** to wash water coming from reflux decanter **29** along line **33.** As noted, this slipstream can be in the range of about 0.10 percent to about 5.0 percent of the total aromatics in the feedstock and is preferably in the range of about 0.50 percent to about 2.0 percent of the total aromatics in the feedstock. These percentages are by weight.

In practice, the weight of the total aromatics is determined by analysis of a sample portion of the feedstock. Aromatics added, e.g., as slipstream, during the process cycle are included in the determination.

The slipstream can, alternatively, be obtained from another source such as the overhead product of a benzene fractionating column, which is not shown in the drawing, or from a source completely removed from the system. As long as the slipstream has the previously noted high aromatics content, it will be satisfactory in this process.

The combined streams of lines **33** and **14** proceed into wash extractor **39,** which can be a single stage mixer-settler or other form of extractor. Where a mixer-settler is used, it is advantageous to use an aromatics recycle which passes along line **42** and joins lines **33** and **14** returning to wash extractor **39.** The slipstream, now containing a small amount of aliphatics, passes overhead from wash extractor **39** into line **42** and along line **50** to join lines **12** and **15** and proceeds into wash extractor **13** as discussed previously.

Reflux water, now essentially free of aliphatics, is withdrawn from wash extractor **39** and proceeds along line **43,** which joins line **26,** and passes into aromatics extractor **44,** which can be a single stage mixer-settler or other type of extractor. This reflux water, along with water recycled from the settling zone in the case of a mixer-settler via line **45,** which joins line **43,** and process makeup water from line **46** (source not shown) contacts the aromatic product proceeding along line **26** into aromatics extractor **44** and recovers essentially all of the small amount of solvent remaining in the aromatics. This water with solvent then proceeds along line **47** to join line **17,** which joins line **37** and enters water reservoir **51.** High purity aromatic product is withdrawn from the process through line **26.**

Removal of certain impurities, which may include some aliphatics of a type which can build up in the system and affect it in a deleterious manner, is accomplished by taking a small purge of the water circuit. To accomplish this purge, water is withdrawn from any of the decanters and discarded periodically or continuously. One such purge can be accomplished through line **48.** It is found that only a small proportion of the solvent is lost by such a purge; however, this solvent can be recovered if desired. The water purge stream can be in the range of about 0.25 percent to about 2.0 percent by weight of the total water in the system and is preferably in the range of about 0.5 percent to about 1.0 percent by weight of the water in the system.

The total water in the system can be determined easily because the amount of water introduced can be controlled. Allowances must be made for water losses through leakage, entrainment and upsets, however.

Solvent can be recovered from this purge by directing the water through line **49** to join line **53** and enter solvent regenerator **52** where the solvent is separated from low boiling and high boiling impurities by steam distillation under vacuum. The solvent is recovered and recycled along line **54** to extractor **3** (connection not shown) and the water and impurities discarded.

It will be noted that in the preferred embodiment of subject process the slipstream taken through line **14** is first used to wash the water phase from reflux decanter **29** (i.e., one stream) and then the water phases from raffinate decanter **7** (optional) and raffinate extractor **8.** This procedure can be varied so that a different slipstream from a different source is used for each wash or, as previously mentioned, a single slipstream is used to wash one water phase where stream **31** is not split, but is first used to wash raffinate.

In the preferred embodiment, it was stated heretofore that the slipstream picks up some aliphatics in extractor **39** before proceeding to extractor **13.** It should be pointed out that the purity of this slipstream containing the small amount of aliphatics is only reduced by about one percent and that it still has a purity of at least about 95 percent by weight and preferably about 98 percent so that the definition of the slipstream with respect to purity is fulfilled.

The description of the invention is in terms of a continuous process which has already been initiated. In order to initiate the process, it is necessary to supply to the system from an outside source, sufficient of the defined light aliphatics to first saturate the extract or the reflux loop and then a sufficient amount to bring the defined light aliphatics up to the prescribed level. This is also done for the solvent and water. Once the process is initiated, these components are recycled with make-up being added when necessary.

Subject process is found to be particularly advantageous for feedstock containing at least about 80 percent by weight aromatics and even more so for those containing about 90 percent or more aromatics. Optimum performance is achieved when the benzene content is also high, i.e., at least about 25 percent by weight of the feedstock. The process is found to be beneficial for feedstocks having an aromatics content of at least about 40 percent by weight, however.

What is claimed is:

1. In a continuous solvent extraction distillation process for the recovery of aromatic hydrocarbons having boiling points in the range of about 80°C. to about 175°C. from a feedstock containing aliphatic hydrocarbons and at least about 40 percent by weight, based on the weight of the feedstock, of said aromatic hydrocarbons wherein the feedstock contains no more than about 4 percent by weight, based on the weight of the feedstock, of light aliphatics consisting essentially of aliphatic and cycloaliphatic hydrocarbons, each having no more than 5 carbon atoms, a boiling point no higher than about 50°C. and being condensable at a pressure of no more than about 3 atmospheres and at a temperature of no less than about 50°C.; a single extraction column having three to 25 theoretical stages is used for the solvent extraction to provide an extract; and a single distillation column is used to distill the extract to provide a mixture of the aromatic hydrocarbons comprising the following steps:

a. introducing the feedstock into the extraction column at the middle theoretical stage thereof;

b. contacting the feedstock in the extraction column with a mixture of water and a solvent, said solvent being a water-miscible organic liquid having a boiling point of at least about 200°C. and having a decomposition temperature of at least about 225°C., and with reflux hydrocarbons introduced into the extraction column below the bottom theoretical stage thereof to provide the extract comprising aromatic hydrocarbons, reflux aliphatic hydrocarbons, solvent, and water and a raffinate comprising essentially aliphatic hydrocarbons;

c. introducing the extract into the distillation column to separate the aromatic hydrocarbons and the reflux hydrocarbons from the extract;

d. recycling the reflux hydrocarbons from step (c) to the extraction column as provided in step (b); and

e. recovering the aromatic hydrocarbons of step (c);

the improvement comprising introducing a sufficient amount of the light aliphatics defined above into the extraction column at the middle theoretical stage thereof to provide a total percent by weight of said light aliphatics, based on the weight of the feedstock, in the range of about 5 percent to about 12 percent.

2. The process of claim **1** comprising the following additional steps:

f. introducing the raffinate formed in step (b) into a raffinate distillation zone to separate the light aliphatics defined in claim **1** therefrom;

g. recycling the defined light aliphatics from step (f) to the middle theoretical stage of the extraction column; and

h. recovering the balance of the raffinate from step (f).

3. The process of claim **2** wherein the feedstock and the defined light aliphatics enter the extraction column at about the same point.

4. The process of claim **3** wherein the temperature in the extraction column is in the range of about 100°C. to about 200°C., the pressure in the extraction column is in the range of about 75 psig to about 200 psig, the temperature in the distillation column is in the range of about 135°C. to about 200°C., and the pressure in the distillation column is in the range of about 10 psig to about 35 psig.

5. The process of claim **4** wherein the feedstock contains at least about 80 percent by weight, based on the weight of the feedstock, of aromatic hydrocarbons.

6. The process of claim **4** wherein

i. the ratio of solvent to feedstock in the extraction column is in the range of about 3 to about 12 parts by weight of solvent to one part by weight of feedstock;

ii. the amount of water in the extraction column is about 1 percent to about 8 percent by weight based on the weight of the solvent in said column;

iii. the ratio of reflux to feedstock in the extraction column is in the range of about 0.5 to about 1.5 parts by weight of reflux to one part by weight of feedstock; and

iv. the ratio of water to aromatic hydrocarbons in the distillation column is in the range of about 0.1 to about 0.5 part by weight of water to one part by weight of aromatic hydrocarbons in said column.

7. The process of claim **6** wherein the solvent is a polyalkylene glycol.

8. The process of claim **7** wherein the solvent is tetraethylene glycol.

9. The process of claim **8** wherein the feedstock contains at least about 80 percent by weight, based on the weight of the feedstock, of aromatic hydrocarbons.

10. The process of claim **8** wherein the extraction column has five to 12 theoretical stages.

11. In a continuous solvent extraction-distillation process for the recovery of aromatic hydrocarbons having boiling points in the range of about 80°C. to about 175°C. from a feedstock containing aliphatic hydrocarbons and at least about 40 percent by weight, based on the weight of the feedstock, of said aromatic hydrocarbons wherein the feedstock contains no more than about 4 percent by weight, based on the weight of the feedstock, of light aliphatics consisting essentially of aliphatic and cycloaliphatic hydrocarbons, each having no more than five carbon atoms, a boiling point no higher than about 50°C. and being condensable at a pressure of no more than about 3 atmospheres and at a temperature of no less than about **50**°C.; a single extraction column having three to 25 theoretical stages is used for the solvent extraction to provide an extract;

and a single distillation column is used to distill the extract to provide a mixture of the aromatic hydrocarbons comprising the following steps:

a. introducing the feedstock into the extraction column at the middle theoretical stage thereof;

b. contacting the feedstock in the extraction column with a mixture of water and a solvent, said solvent being a water-miscible organic liquid having a boiling point of at least about 200°C. and having a decomposition temperature of at least about 225°C., and with reflux hydrocarbons introduced into the extraction column below the bottom theoretical stage thereof to provide the extract comprising aromatic hydrocarbons, reflux aliphatic hydrocarbons, solvent, and water and a raffinate comprising essentially aliphatic hydrocarbons;

c. contacting the extract with steam in the distillation column to provide an overhead distillate comprising a reflux hydrocarbons phase and a water phase, a side cut distillate comprising an aromatic hydrocarbons phase and a water phase, and bottoms comprising a mixture of solvent and water;

d. dividing the water phase of the overhead distillate into first and second streams;

e. contacting the raffinate with the first stream to provide an aliphatic hydrocarbons phase and a water phase;

f. contacting the second stream with an aromatic hydrocarbons stream containing at least 95 percent aromatic hydrocarbons, the amount of said stream being in the range of about 0.1 percent to about 5 percent by weight of the total aromatic hydrocarbons in the feedstock, to form an aromatic hydrocarbons phase and a water phase;

g. contacting the aromatic hydrocarbons phase of the side-cut distillate with the water phase of (f) to form an aromatic hydrocarbons phase and a water phase;

h. contacting the water phase of step (e) with an aromatic hydrocarbons stream containing at least 95 percent aromatic hydrocarbons, the amount of said stream being in the range of about 0.1 percent to about 5 percent by weight of the total aromatic hydrocarbons in the feedstock, to form an aromatic hydrocarbons phase and a water phase;

i. recycling the water phases of steps (g) and (h) to the distillation zone where said water phases are essentially converted to steam;

j. recycling the reflux hydrocarbons phase of the overhead distillate and the bottoms of step (c) to the extraction zone to provide reflux hydrocarbons and mixture of water and solvent, respectively, for step (b); and

k. recovering the aromatic hydrocarbons phase of step (g) and the aliphatic hydrocarbons phase of step (e);

the improvement comprising introducing a sufficient amount of the light aliphatics defined above into the extraction column at the middle theoretical stage thereof to provide a total percent by weight of said light aliphatics, based on the weight of the feedstock, in the range of about 5 percent to about 12 percent.

12. In a continuous solvent extraction-distillation process for the recovery of aromatic hydrocarbons having boiling points in the range of about 80°C. to about 175°C. from a feed-stock containing aliphatic hydrocarbons and at least about 40 percent by weight, based on the weight of the feedstock, of said aromatic hydrocarbons wherein the feedstock contains no more than about 4 percent by weight, based on the weight of the feedstock, of light aliphatics consisting essentially of aliphatic and cycloaliphatic hydrocarbons, each having no more than five carbon atoms, a boiling point no higher than about 50°C. and being condensable at a pressure of no more than about 3 atmospheres and at a temperature of no less than about 50°C.; a single extraction column having three to 25 theoretical stages is used for the solvent extraction to provide an extract; and a single distillation column is used to distill the extract to provide a mixture of the aromatic hydrocarbons comprising the following steps:

a. introducing the feedstock into the extraction column at the middle theoretical stage thereof;

b. contacting the feedstock in the extraction column with a mixture of water and a solvent, said solvent being a water-miscible organic liquid having a boiling point of at least about 200°C. and having a decomposition temperature of at least about 225°C., and with reflux hydrocarbons introduced into the extraction column below the bottom theoretical stage thereof to provide the extract comprising aromatic hydrocarbons, reflux aliphatic hydrocarbons, solvent, and water and a raffinate comprising essentially aliphatic hydrocarbons;

c. contacting the extract with steam in the distillation column to provide an overhead distillate comprising a reflux hydrocarbons phase and a water phase, a side cut distillate comprising an aromatic hydrocarbons phase and a water phase, and bottoms comprising a mixture of solvent and water;

d. contacting the raffinate with the water phase of the overhead distillate to provide an aliphatic hydrocarbons phase and a water phase;

e. contacting the water phase of step (d) with an aromatic hydrocarbons stream containing at least 95 percent aromatic hydrocarbons, the amount of said stream being in the range of about 0.1 percent to about 5 percent by weight of the total aromatic hydrocarbons in the feedstock, to form an aromatic hydrocarbons phase and a water phase;

f. contacting the aromatic hydrocarbons phase of the side-cut distillate with the water phase of (e) to form an aromatic hydrocarbons phase and a water phase;

g. recycling the water phase of step (f) to the distillate zone where said water phase is essentially converted to steam;

h. recycling the reflux hydrocarbons phase of the overhead distillate and the bottoms of step (c) to the extraction zone to provide reflux hydrocarbons and mixture of water and solvent, respectively, for step (b); and

i. recovering the aromatic hydrocarbons phase of step (f) and the aliphatic hydrocarbons phase of step (d);

the improvement comprising introducing a sufficient amount of the light aliphatics defined above into the extraction column at the middle theoretical stage thereof to provide a total percent by weight of said light aliphatics, based on the weight of the feedstock, in the range of about 5 percent to about 12 percent.

13. The process of claim **11** wherein the solvent is a polyalkylene glycol.

14. The process of claim **13** wherein the solvent is tetraethylene glycol.

15. The process of claim **12** wherein the solvent is a polyalkylene glycol.

16. The process of claim **15** wherein the solvent is tetraethylene glycol.

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