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H. D. EVANS ETAL

3,222,416

EXTRACTION PROCESS

Filed Dec. 18, 1961

2 Sheets-Sheet 1

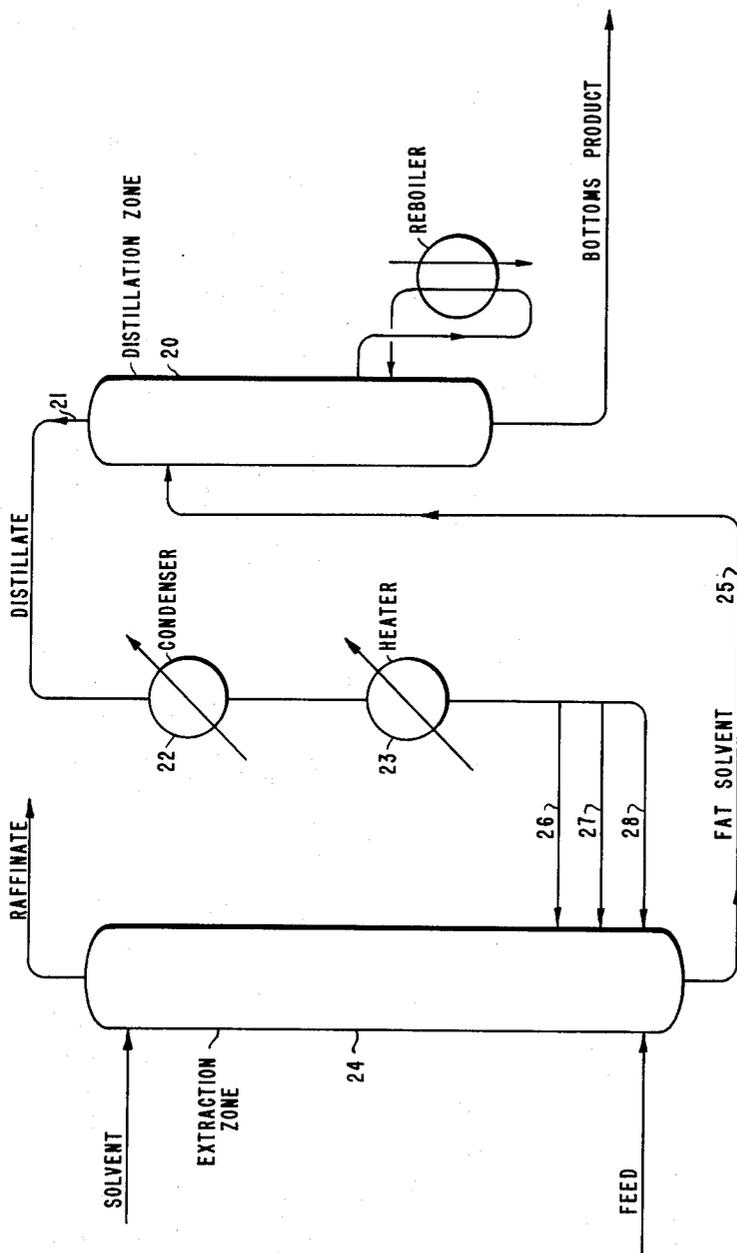


FIGURE I

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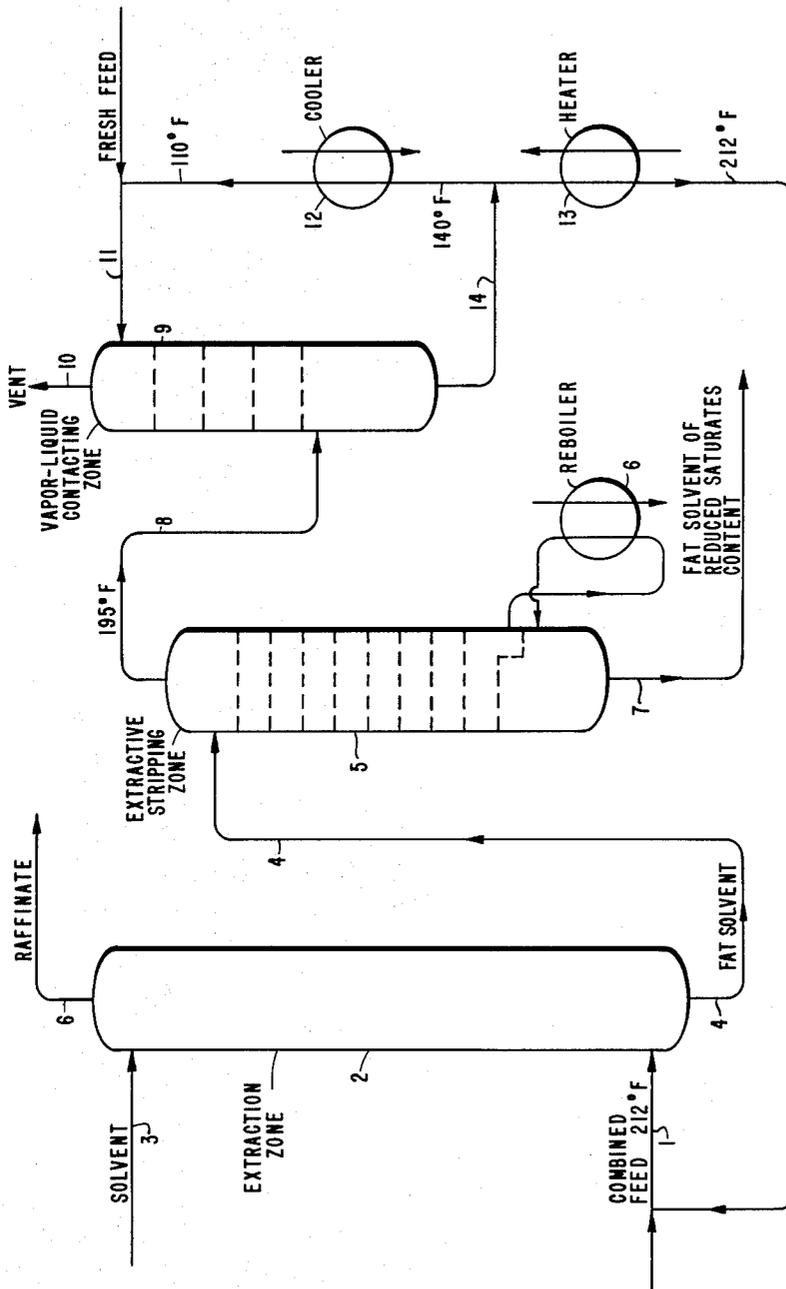


FIGURE II

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3,222,416

EXTRACTION PROCESS

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4 Claims. (Cl. 260-674)

This invention relates to an improved extraction process for recovering aromatic components from aromatic-containing feedstocks wherein sulfolane solvents are used as selective solvents. More specifically, this invention is directed to an improved extraction process for the recovery of high-purity aromatics wherein the saturates and aromatics contained in the overhead stream recovered from the fat sulfolane solvent extract by partial distillation is advantageously mixed with the fresh feed prior to introducing the latter into the extraction zone.

A conventional process for the recovery of high purity aromatics from various feedstocks is the extraction of such mixtures with selective solvents such as diethylene glycol. A general process of this type is disclosed in U.S. Patent 2,407,820, to Durrum issued September 17, 1946. FIGURE 1 is essentially a schematic diagram of pertinent aspects of the process disclosed in the above-referenced U.S. patent.

In the normal operation of such a conventional process (referring to FIGURE 1) the saturates and aromatics are removed from the fat solvent phase (withdrawn from extraction zone 24 through line 25) by means of extractive distillation in distillation zone 20. The aromatic/saturate distillate is withdrawn from zone 20 through line 21 and condensed in condenser 22 and reheated to the extraction temperature in heater 23 and reintroduced into extraction zone 24 for further recovery of the contained aromatics. The point of introduction of the condensed aromatic saturate distillate stream through lines 26, 27, and 28 into extraction zone 24 is a function of the aromatic content of the stream.

It has now been discovered that in some cases, for example, in the recovery of benzene from a light platformate fraction by sulfolane extraction, the above-referenced recycle stream can be advantageously mixed with a fresh feed stream prior to introduction of the latter into the extractor, thereby recovering a portion of the heat which would normally be lost to the cooling water if the distillation overhead stream were recycled directly to the extractor through a condenser as disclosed in FIGURE 1.

Accordingly, it is an object of this invention to provide an improved extraction process for the separation of high-purity aromatics from aromatic-containing feedstocks. It is a further object of this invention to provide an improved extraction process wherein the heat contained in the aromatic/saturate overhead stream obtained from the partial distillation of the extract phase is transferred to the fresh feed entering the extraction zone without adversely affecting the aromatic or saturate product purity. It is a further object of this invention to provide an improved extraction process for the recovery of high-purity aromatics wherein fresh feed is used as the coolant for the aromatic/saturate overhead product from a partial distillation of an extract phase, thereby avoiding the necessity of (1) cooling the overhead stream and (2) preheating the fresh feed. Other objects and advantage of this invention will become apparent in the description thereof which is made with reference to the accompanying draw-

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ing, which consists of FIGURES 1 and 2. FIGURE 1, as discussed above, is the schematic diagram of a conventional extraction process wherein the partial distillation overhead stream is condensed in a conventional condenser and reheated prior to introduction into the extraction zone. FIGURE 2 is a schematic diagram of a preferred embodiment of the invention wherein the overhead product from the partial distillation of the extract phase is contacted with the fresh feed prior to introduction into an extraction zone, thereby avoiding the need for condensing the overhead stream and preheating the fresh feed.

Accordingly, it has now been discovered that through a novel arrangement of equipment, significant improvements can be achieved in the conventional type extraction processes as discussed above with respect to heat loss and cooling water requirements.

According to the process of the instant invention, fresh feed is passed in counterflow to extractive stripping zone vapors in a vapor-liquid contacting zone. The heat of condensation and sensible heat of the vapors are adsorbed in the fresh feed, thereby eliminating cooling water requirements and reducing the amount of feed preheat required. In the event the amount of fresh feed is not sufficient to cool the vapors to the temperature required for condensation, part of the combined stream is recirculated through a cooler to remove the excess heat. By employing the particular arrangement of equipment which will be described in more detail below, the net saving in cooling required compared to conventional condensing was 33% whereas the net savings of preheating required was about 30%. A further advantage of the instant invention with respect to conventional condensing is that the vapors leaving the recycle condensing system by way of a venting line are cooled to a lower temperature, thereby reducing vapor losses.

In the extraction process of the invention, the conditions of temperature, pressure and concentration of solvent in the extraction zone are selected to insure optimum recovery of high-purity aromatics from the feed charge. A lean, relatively aromatic-free sulfolane solvent selective for aromatic hydrocarbons and which is relatively immiscible with non-aromatic hydrocarbons is admitted to one end of an extraction zone and in extractive contact with the aromatics contained in the feed charge. Such a solvent should have a relatively high selective solubility for aromatics at the extraction temperature. The liquid feed is introduced at the bottom of the extraction zone. The solvent flows through the zone countercurrent to the feed charge. The fat solvent containing the selectively dissolved aromatics is continuously withdrawn from the extraction zone at the end opposite the end where the solvent is introduced, and is then introduced into an extractive stripper.

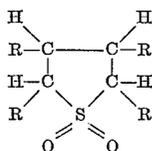
The raffinate from the extraction zone, containing some of the solvent, passes overhead out of the extraction zone. The distillate from the extractive stripper containing non-aromatic and some aromatic hydrocarbons is passed to a vapor-liquid contacting device where it is passed in counterflow to the portion of the fresh feed. The fresh feed/condensed extractive stripper overhead stream is then introduced into the extraction zone. The water-sulfolane-aromatic hydrocarbon extractive stripper bottoms are then introduced into a solvent stripper. The stripped lean solvent is then recycled to the extraction zone. The aromatics product stripped from the solvent is passed

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overhead along with the water. Subsequently, the aromatics are separated from the water as a high-purity aromatic product.

The selective solvents which can be used according to the invention should be selective to aromatics and must have some miscibility with water to insure that the solvent will pass out of the bottom of the extractive stripper along with the water. Further, the solvent must be stable at the extractive stripper and solvent stripper temperatures. That is, the solvent must be sufficiently heat-stable so that it may at least partially vaporize without decomposition in the presence of the components of the mixture to be selectively extracted. Among the preferred solvents are the various sulfolane solvents.

In a preferred embodiment of the invention, sulfolane solvents having the following general formula are used in the improved extraction process:



where R can be a hydrogen atom, an alkyl group having up to 10 carbon atoms, an alkoxy radical having up to 8 carbon atoms, an aryl alkyl radical having up to 12 carbon atoms or mixtures thereof. In a particularly preferred embodiment, all R's are hydrogen.

Many different aromatic/non-aromatic mixtures may be separated by the extraction process of the present invention. However, a preferred embodiment of the invention is the recovery of aromatics from hydrocarbon feed mixtures containing substantial concentrations of aromatics and a particularly preferred embodiment of the invention is the recovery of benzene from a light platformate fraction by sulfolane extraction.

The apparatus employed in this process may be any conventional or 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 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 temperature for the extraction zone may range within wide limits provided it is below the bubble temperature of the mixture and above the solidification temperature of the selective solvent under the pressure and temperature conditions maintained in the extraction zone. The most suitable operating temperatures vary with the pressure maintained during the operation and are generally a compromise, since in general the selectivity of the sulfolane solvents seems to decrease with an increase in temperature. In a preferred embodiment of the invention, substantially atmospheric pressures are maintained in the vapor-liquid contacting zone. The temperature of the vapor-liquid contacting zone can range from about 80° F. to about 180° F. And preferably the temperature at the top of the zone is from about 80° F. to 110° F., with the bottom temperature ranging from 150° F. to about 180° F. The temperature of the extractive stripper overhead stream ranges from about 180° F. to about 200° F., whereas, the temperature of the preheated fresh feed containing the recycled extractive stripper overhead ranges from about 150° F. to about 180° F.

The amount of sulfolane solvent used in the extraction zone should be at least sufficient to dissolve the constituent to be extracted. In some cases a considerable excess over this amount may be used, especially when it is desired to recover the last traces of the more soluble constituents from the raffinate. The most economic amount varies with the nature of the particular sulfolane solvent being

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used and the temperatures and pressures employed and the particular feed mixture. This amount can be determined experimentally. Useful solvent to feed ratios in the extraction zone may range from about ½:1 to about 20:1 by volume and preferably not more than about 5:1 by volume.

The optimum temperature to be employed in the extraction zone varies with the particular feedstock to be treated and the sulfolane solvent used, and may range up to about 350° F. or more, but in most instances temperatures below about 250° F. and particularly those in the range of from about 100 to about 200° F. are preferred.

Referring now to FIGURE 2 of the drawing, the feed stream having a temperature of about 212° F. and comprising an aromatic-containing feedstock and also containing the condensed extractive stripper overhead is introduced by means of line 1 into extraction zone 2. Extraction zone 2 can be a conventional liquid-liquid extraction column, such as a column containing a plurality of perforated plates horizontally positioned therein, or in a preferred embodiment of the invention, zone 2 is a rotating disc contactor, as described in U.S. Patent to Remain, 2,601,674, issued June 24, 1952.

A lean solvent stream is introduced through line 3 into the upper portion of extraction zone 2. The selective solvent flows down zone 2 countercurrent to the feed charge and selectively dissolves the aromatics therefrom with the fat solvent containing the selectively dissolved aromatics and some nonaromatics, passing out of extraction zone 2 via line 4. The fat solvent is then introduced into the top of extractive stripper 5. The raffinate from extraction zone 2 is passed overhead via line 6.

Extractive stripper 5 is equipped with reboiler 6 and is preferably provided with trays, preferably valve trays or trays fitted with one or more preferential discharge zones (see dotted lines). A fat solvent having a significantly reduced saturates content is withdrawn from extractive stripper 5 through line 7. The overhead product withdrawn from extractive stripper 5 through line 8 consists essentially of non-aromatic hydrocarbon and has a temperature of about 195° F. and is substantially free of selective solvent, however, it does contain some aromatic hydrocarbons. This extractive stripper overhead product is introduced into the lower portion of vapor-liquid contacting zone 9. Vapor liquid contacting zone 9 is equipped with vapor vent 10 and is preferably equipped with the type trays used in extractive stripper 5. The upper portion of contacting zone 9 is maintained at about 110° F. with the lower portion having a temperature of about 140° F. Fresh feed having a temperature of about 100° F. is introduced into the vapor liquid contacting zone through line 11. If the amount of fresh feed introduced into vapor liquid contacting zone 9 via line 11 is not sufficient to cool the extractive stripper vapors in the zone to the temperature required for condensation thereof, part of the combined stream (the bottom product withdrawn from zone 9 through line 14 containing the fresh feed and some of the condensed vapors) is recirculated to the zone after passing through cooler 12 to remove the excess heat. The bottoms product from zone 9 is withdrawn through line 14 having a temperature of about 140° F. and is passed through heater 13 and preheated to an acceptable extraction temperature of about 212° F. before introduction into extraction zone 2.

We claim as our invention:

1. A process for the recovery of benzene from a light hydrocarbon platformate fraction liquid feed mixture comprising:

(1) countercurrently contacting said feed mixture in a vapor-liquid contacting zone with a vapor fraction recovered from an extractive stripping zone whereby at least part of said vapor is directly condensed and wherein a combined feed fraction is formed;

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(2) passing said combined feed fraction comprising a mixture of feed and condensed vapor from said extractive stripping zone to a liquid-liquid extraction zone;

(3) countercurrently contacting said combined feed fraction in said liquid-liquid extraction zone with an aromatic-selective sulfolane solvent whereby a raffinate comprising substantially non-aromatic hydrocarbons is withdrawn overhead, and wherein a fat solvent stream comprising substantially aromatics, sulfolane solvent, and a minor amount of non-aromatics is recovered;

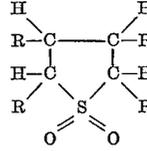
(4) passing said fat solvent stream to an extractive stripping zone wherein a bottoms product comprising substantially sulfolane solvent and aromatics of reduced saturates content is recovered and later used as the source of aromatics product and solvent for recycle;

(5) recovering overhead from said extractive stripping zone a vapor product comprising aromatics and saturates which are directly contacted with said feed fraction in said vapor contacting zone.

2. A process according to claim 1 wherein the extractive stripping zone distillate introduced in a vapor-liquid contacting zone has a temperature between about 150° F. to about 350° F. and the feed introduced into the vapor-liquid contacting zone has a temperature between about 80° F. and about 150° F., and the feed/condensed distillate mixture withdrawn from said vapor-liquid contacting zone has a temperature between about 120° F. to about 200° F.

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3. A process according to claim 1 wherein the selective solvent is a sulfolane solvent having the structural formula



wherein R is 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, an aryl alkyl radical having up to 12 carbon atoms and mixtures thereof.

4. A process according to claim 1 wherein the selective solvent is sulfolane.

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