July 26, 1966 P. L. GIROTTI ETAL 3,262,875

PROCESS FOR EXTRACTING HIGH-PURITY AROMATIC HYDROCARBONS FROM A HYDROCARBONACEOUS MIXTURE

Filed May 31, 1962 3 Sheets-Sheet 1

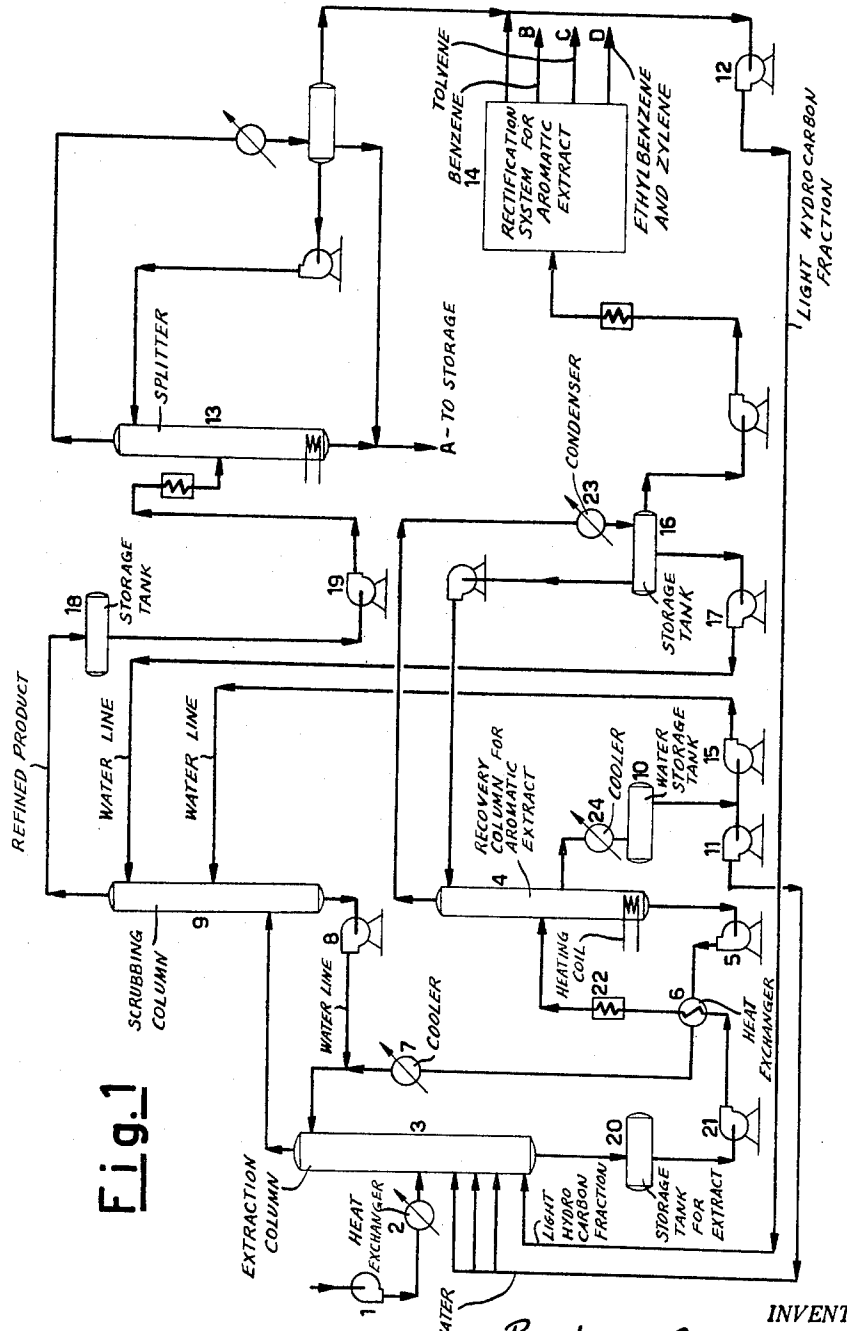



Fig. 1

INVENTORS
Pier Leone Girotti and
BY Ermanno Cinelli
Attorney

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Filed May 31, 1962 3 Sheets-Sheet 2

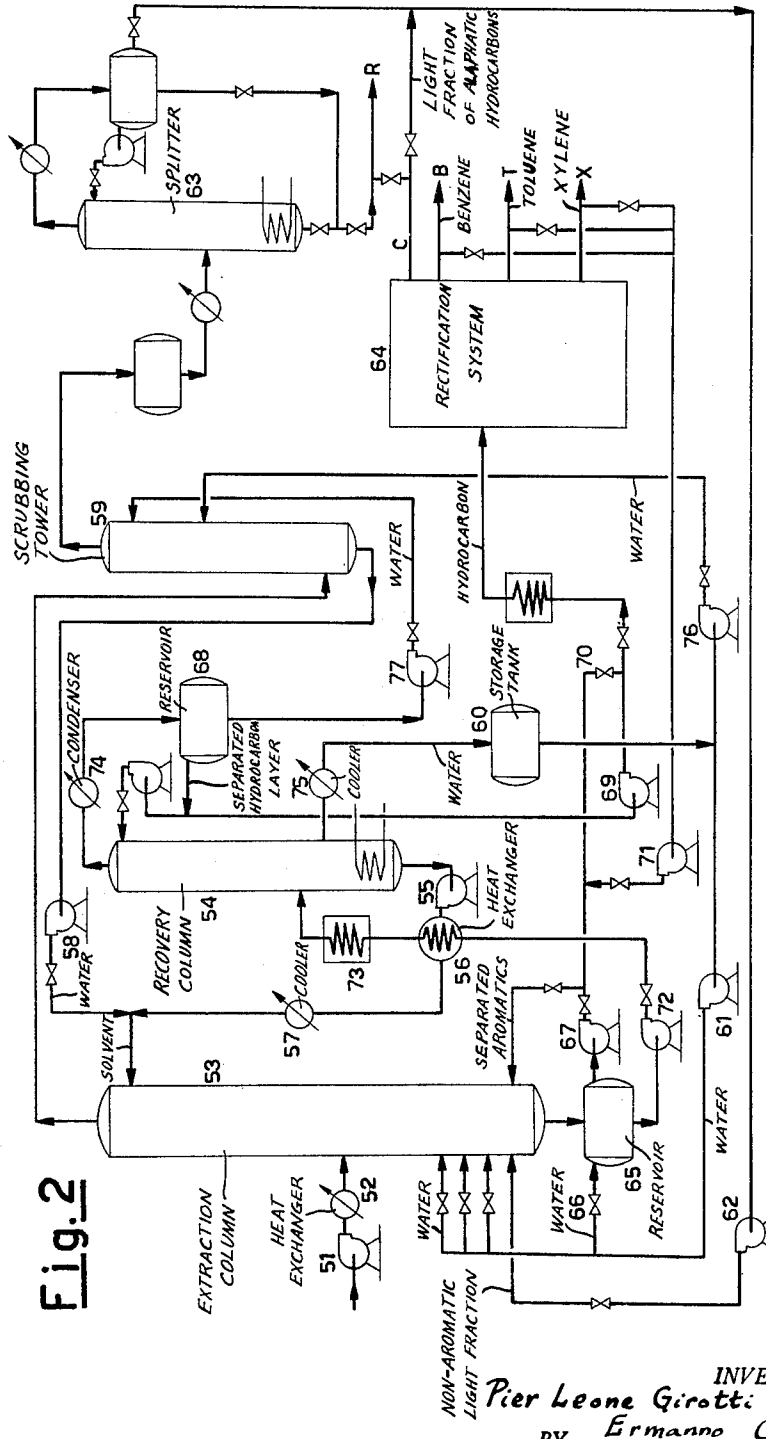



Fig. 2

INVENTORS
Pier Leone Girotti and
Ermanno Cinelli
BY
Attorney

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Filed May 31, 1962 3 Sheets-Sheet 3

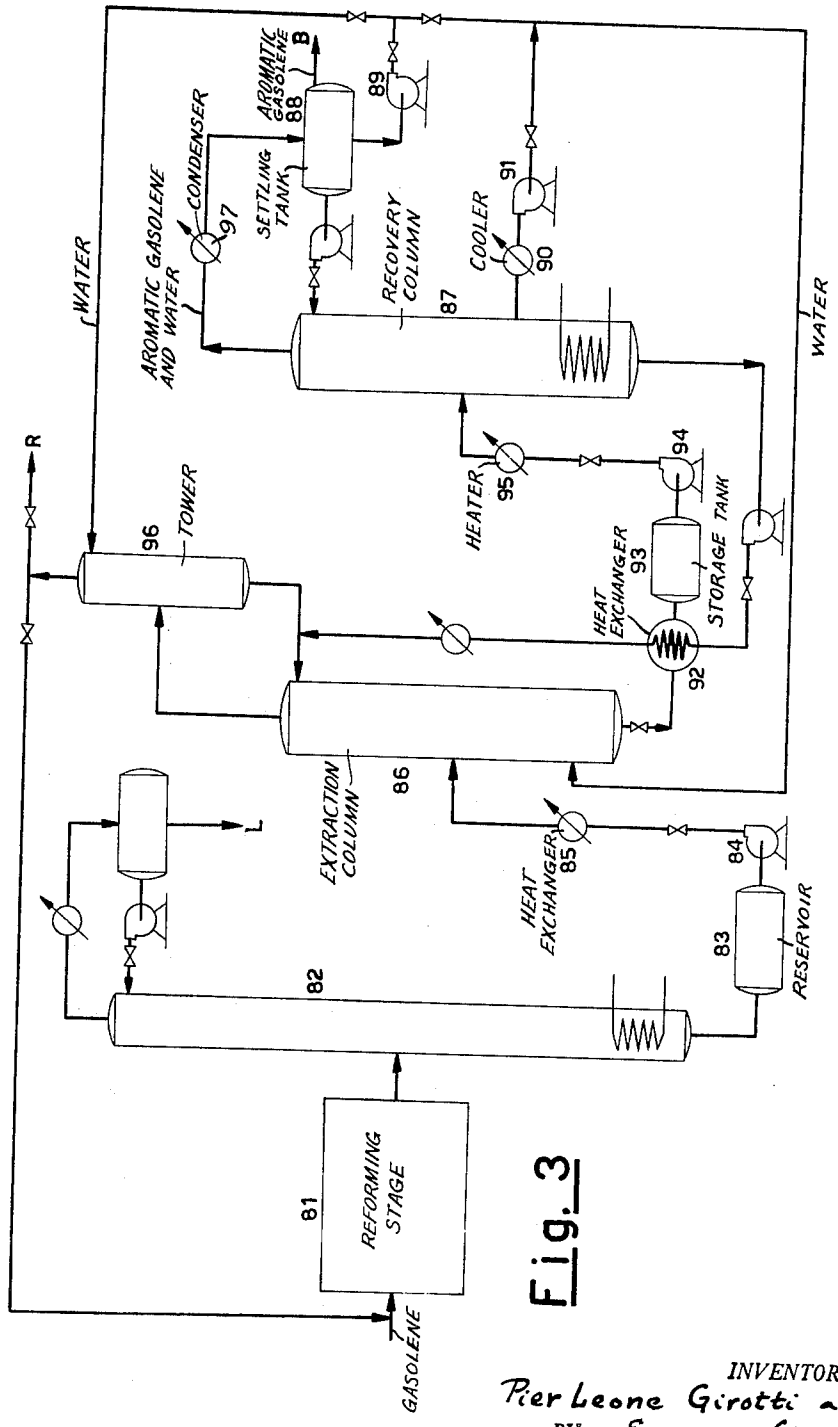



Fig. 3

INVENTOR.
Pier Leone Girotti and
BY Ermanno Cinelli
Attorney

United States Patent Office 3,262,875 Patented July 26, 1966



3,262,875

# PROCESS FOR EXTRACTING HIGH-PURITY AROMATIC HYDROCARBONS FROM A HYDROCARBONACEOUS MIXTURE

Pier Leone Girotti and Ermanno Cinelli, San Donato Milanese, Italy, assignors to SNAM-Società per Azioni

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3 Claims. (Cl. 208—324)

This invention relates to a process for recovering benzene, toluene, ethylbenzene, xylenes and higher homologs from mixtures containing said aromatic hydrocarbons in admixture with other, non-aromatic hydrocarbons.

The use of counterflow extraction processes and selective solvents is generally known in the art, for extracting the aromatic hydrocarbons from mixtures in which they are contained.

An example of such mixture is the re-formed gasoline obtained from the catalytic reforming of the straight-run gasolines.

Among the selective solvents or extractants known in the art, there can be cited diethylene glycol, oxidipropionitrile, thiodipropionitrile, sulpholane and others: these are miscible more with aromatic hydrocarbons than with other, non-aromatic hydrocarbons.

According to the present invention, the extraction of aromatic hydrocarbons from mixtures in which they are contained, is effected with a selective composite solvent mixture consisting of from about 99.5% to about 50% by volume of morpholine and of from about 0.5% to about 50% of water.

In the selective composite solvent mixture according to this invention, morpholine (tetrahydro-1,4-oxazine) can also be replaced, totally or in part, by a morpholine derivative in which one or more of the hydrogen atoms is replaced by an alkyl radical having from 1 to 4 carbon atoms.

The presence of water in the selective composite solvent mixture according to the present invention is highly critical: it is known, in fact, that morpholine alone is a solvent for the majority of the aliphatic and aromatic hydrocarbons.

The selective activity of the composite solvent mixture according to the invention is due to the presence of water in the volume range specified above: the other critical or preferential conditions will be specified as the present disclosure goes on.

The extraction process employing the composite solvent mixture according to the present invention is carried out at any temperature comprised in the range 0° C. to 95° C.; the pressure is not, as itself, a critical parameter. It is preferred to work, whenever practicable, in the neighborhood of the atmospherical pressure: in practice, however, the working pressure is a function of the temperature selected for the extraction process, the composition of the mixture to be subjected to extraction and, only subordinately, of the vapor pressure of the solvent.

In the solvent mixture according to the present invention, water plays the role of a selectivity-adjusting agent: as a matter of fact the hydrous morpholine employed in this invention, owing to the great miscibility of morpholine and its derivatives in general with water, has a great carrying-capacity towards the aromatic fractions even when the percentage of water in the morpholine-water system is relatively high. The invention thus affords the combined advantages of higher aromatics yield than heretofore practicable, along with a high purity of the extracted product.

Water can be added to morpholine either all at a time, or by increments: for example, water can be added to morpholine before entering the extraction system, at properly selected points of said system and, more preferably, in the vicinity of the point at which the extract is drawn. Also, a paraffinic hydrocarbonaceous fraction may be introduced in a properly selected point of the extraction system: this fraction should consist of aliphatic hydrocarbons having a boiling point lower than the boiling point of the aromatic hydrocarbon to be recovered which has the lowest boiling point.



Also a recycling stage of aromatic hydrocarbonaceous components can be adopted with advantage in certain cases, in the neighborhood of the point at which the final aromatic extract is drawn.

The practical performance of the process according to the invention will be explained and exemplified, without implying any limitation, the ensuing disclosure being aided by the accompanying drawings, in which:

FIG. 1 is a flow diagram of an extraction process according to one embodiment of the invention,

FIG. 2 is another flow diagram of an extraction process according to another embodiment of the invention, and

FIG. 3 is another flow diagram of an extraction process according to a still further embodiment of the invention.

The following examples will give a clear picture of the best way for putting the present invention into practice.

*Example 1*

Having reference, for this example, to the flow-sheet of FIG. 1, it will be readily apparent that the hydrocarbon charge, fed by a pump 1, is brought to the selected temperature in the combination cooler-heater 2, after which it enters the extraction system proper, 3: the latter can be a filled column, a bubble-cap column, a rotating plate column, a column having mixing and settling compartment, or any other suitable extraction column.

The solvent, coming from the bottom of the column 4 (in which the aromatic extract is recovered), is fed through the heat exchanger 6 and the cooler 7, and enters the extraction column 3 near the top portion thereof after having been admixed with water: the latter comes, via the pump 8, from the washing system 9 for the washing of the refined product. The rate of flow of water is adjusted so as to have the correct proportion of water in the system.

The volume ratio solvent/raw hydrocarbon mixture can be varied from 1:1 to 30:1. In general high solvent ratios are adopted when the mixture to be extracted is relatively rich in aromatics, and vice versa.

A further addition of water (from recovery) is effected, via the pump 11, from the storage tank 10, in one or more different point in the neighborhood of the lowermost portion of the extraction column 3.

In a zone intermediate between the water feed and the point at which the extracted product is withdrawn, there is introduced, via the pump 12, the light hydrocarbon fraction taken from the top of the splitter 13 and from the rectification system 14 of the aromatic extract. This fraction ordinarily contains non-aromatics, with a small aliquot of benzene.

The refined product is taken from the top of the column 3 and is fed to the lowermost portion of the scrubbing column 9; here the non-aromatics are freed from the solvent, by counterflowing it with water. This water is taken, partially, from the water recovered from the storage tank 10 which is forwarded, via the pump 15, to a point intermediate in the washing system; the balance of water is taken from the storage tank 16, via the pump 17, and sent to a point near the top of the washing system.

The refined product, stripped of the solvent, is now sent to the storage tank 18 wherefrom, via the pump 19, it is sent to the splitting column 13; here a portion of the light fraction is stripped and recycled, with the pump 12,

to the bottom of the extraction system. The refined product, A, is sent to storage.

According to a variant of the procedure, the light hydrocarbonaceous fraction can be supplied by any external source; if so, it would be possible to dispense with the splitter, whenever this is desired or deemed expedient.

From the bottom of the extraction column, the extract is sent to the storage tank **20** wherefrom it is taken by the pump **21** and sent, via the heat exchanger **6** and the oven **22**, to the recovery column for the aromatic extract. The bottom of this recovery column is equipped with a heating coil.

The "head" fraction, consisting of light non-aromatics, aromatic hydrocarbon, and water, is fed through the condenser **23** and comes to the tank **16** wherefrom a portion of the condensate is taken and fed again, as a reflux, to the recovery column; the remainder is decanted and the hydrocarbons, split up from water, are sent to the rectification, whereas, the water is fed to the processing installation as already explained. In the intermediate zone of the recovery column of the aromatic extract, a further aliquot of water is condensed; this latter goes, via the cooler **24**, to the storage tank **10**. This is made in order to concentrate the solvent so as to adjust its water content to the proper value before recycling it.

A prominent feature of the method of recovering the aromatics according to the present invention lies in the fact that the stripping of the desired aromatics from the extract is effected through an azeotropic distillation; this is carried out at temperatures below 100° C. This procedure is such as to permit that the solvent mixture be obtained, at the bottom of the recovery column, virtually free of hydrocarbons. This is of outstanding usefulness on taking into account that morpholine, whose boiling point at atmospheric pressures is about 128° C., would otherwise boil at a temperature lower than the boiling points of ethylbenzenes and the xylenes.

The aromatic hydrocarbons, from the storage-settler **16** are sent to rectification, so as to obtain, individually:

(1) Light aliphatic hydrocarbons, containing an aliquot of benzene: they are recycled, via the pump **12**, to the bottom of the extraction column;

(2) Benzene, indicated by B in the drawing FIG. 1;

(3) Toluene, C;

(4) Ethylbenzene and xylenes, D.

As already pointed out, the ratio (by volume) of the solvent mixture to the hydrocarbon mixture to be subjected to extraction, can be varied, in this invention, between 1:1 to 30:1.

The morpholine to be employed in the method according to the invention need not be of extreme purity since the impurities which are normally present in the technical product are not such as to impair the extraction process in the slightest.

A paramount advantage, and critical factor, of the present invention is that, by adding water to morpholine (up to 50% of the total volume of the mixture) it is possible to adjust both the solubility and the selective sensitivity of the solvent mixture. Preferably, the water content in the mixture is from 5% to 35% by volume.

A part of the water (from 5% to 15% of the total volume of water) is fed into the extraction column directly admixed with the morpholine; the balance is fed in at one or more points near the bottom of the extraction column.

The advantage of the water is that the water itself, having a great solubility in morpholine, rejects the aliphatic constituents of the raw mixture towards the top of the extraction column, and thus the purity of the aromatic extract is greatly improved.

In order to obtain products of very high purity it is advisable to introduce, in the extraction system, an aliquot of light hydrocarbons, preferably aliphatic, or at least prevailingly aliphatic.

These replace the last fractions of non-aromatics which, ordinarily, are separable by distillation with difficulty. This expedient is not essential, however, since purities as high as 98% are attained altogether, also without recycling.

The flow diagram FIG. 1 is sufficient to perform the process; a minimum of 5 theoretical stages in the extraction system proper is recommended. By employing the process of the present invention, it is possible to obtain an extract having a purity of 99.5% and over and the yields exceed 98% by volume with respect to the aromatics present in the starting hydrocarbonaceous mixture.

It has been further ascertained, in the practical operation of the present invention, that a recycling stage, effected with aromatics, is of advantage since it is conducive to aromatics of high purity.

The beneficial effect of the recycling with aromatics displays itself rather towards the purity of the final product than towards the purity of the aromatic extract.

The aromatic recycling can be effected by taking these aromatics from different points of the installation and the recycling mixture can consist of a single aromatic hydrocarbon or, of an aromatic mixture. Other components, such as water, can also be added. The following Example 2, to be read in connection with the flow-sheet FIG. 2, is illustrative of the recovery of benzene, toluene and xylenes from a reformed gasoline.

*Example 2*

The charge, fed by the pump **51** and brought to the desired temperature in the heat exchanger **52**, enters the extraction system proper **53**, which can be any suitable extraction column.

The solvent, coming from the recovery column **54**, is pumped by the pump **55** and passes through the heat exchanger **56** and the cooler **57** and enters near the top of the extraction column **53** after having been admixed with water coming, via the pump **58**, from the washing system **59** of the refined product.

The amount of water is properly adjusted and an additional quantity of recovery water is taken from the tank **60** via the pump **61** and fed to one or more points in the neighborhood of the bottom of the extraction column.

Intermediate between the points of water feed and the bottom of the column, a non-aromatic light fraction taken from the head of the splitter **63** by the pump **62** is introduced. This fraction could also come from the rectifying system **64** for the aromatic extract. The aromatic recycling is effected below the point at which the light non-aromatic fraction is introduced.

Water is injected in the reservoir **65** through the inlet **66** and thus the separation of a part of the aromatics of extract takes place since, owing to the addition of water, the selectivity of the composite solvent is increased while the solvent power is decreased. The separated aromatics are fed again at the foot of the column **53**, via the pump **67**.

Another method consists of taking, as a whole or in part, the products coming from the rectification system **64** or from the system **54** and sending them to the column **53** again, via the pump **71**. Other variant methods can also be adapted.

The extract from the first extraction column is taken from the reservoir **65** via the pump **72** and sent to the recovery column **54** for the aromatic extract, after having been passed through the heat exchanger **56** and the oven **73**.

The "head," consisting of non-aromatic light hydrocarbons, aromatics and water, are passed through the condenser **74** and come to the reservoir **68** wherefrom a part of the condensate is taken and refluxed in the column. The remainder splits up into two layers: the supernatant layer is hydrocarbons and the underlying layer is water.

From an intermediate zone of the recovery column **54** is condensed a further aliquote of water which, through the cooler **75**, goes to the storage tank **60**.

From the tank **60**, the water is partly taken by the pump **61** to be sent to the bottom of extraction column **53** and partly by the pump **76** to be sent to the scrubbing tower **59**. From the storage-settler **68** the underlying water is sent, via the pump **77**, to the washing system **59** of the refined product.

The supernatant hydrocarbon layer goes to the rectification stage **64** via pump **69**, to give:

(1) Light aliphatic hydrocarbons, which possibly contain an aliquote of benzene; they are recycled to the bottom of column **53**;

(2) Benzene, B (FIG. 2);

(3) Toluene, T;

(4) Xylenes, X.

*Example 3*

A counterflow extraction system having seven mixing and settling stages was charged with:

A fraction 75° C.–160° C. of reformed gasoline having a sp. gr. of 0.8010 at 20° C., consisting of 63.2% by wt. aromatics and 36.8% by wt. of aliphatics, in the seventh stage;

A solvent mixture composed by 90% by wt. of morpholine and 10% by wt. of water, corresponding to a ratio solvent charge 4.75 to 1 by weight (3.8:1 by volume) in the first stage;

Water according to a ratio 4:1 (charge/water) by vol. in the seventh stage.

By keeping the temperature at 25° C. and under atmospherical pressure an extract, containing in the solvent phase aliphatic hydrocarbons (13.82% by wt.) and aromatic hydrocarbons (86.18%) was obtained. The yield of aromatics was 98.6%.

*Example 4*

A nine-stage counterflow extraction system was fed:

At the fifth stage with a reformed gasoline similar to that of Example 3;

At the first stage with a solvent mixture formed by 89% by wt. morpholine and 11% by wt. water with solvent charge ratio of 8.72 to 1 by wt. (7 to 1 by vol.);

At the seventh stage with water, corresponding to a ratio charge water 2:1 by volume, and pentane corresponding to a charge/pentane ratio of 12.5 to 1 vol.;

At the eighth stage with water, corresponding to a ratio charge/water of 2:1 by vol. and pentane corresponding to a ratio charge/pentane of 12.5 to 1, by vol.

By maintaining the same operational conditions as in Example 3, there were obtained:

A refined product having 96.3% by wt. of paraffins and 3.7% aromatics, the paraffins yield being 99.4%;

An extract containing 99.6% by wt. of aromatics and 0.4% paraffin, the aromatics yield being 98.1%.

If the starting mixture contains a single aromatic compound, the rectification system **64** can be dispensed with since the settler **68** already give a substantially pure aromatic compound.

An interesting case is that in which the expected product is a high-octane gasoline.

When it is desired to obtain products with special characteristics, e.g., the so-called "nitration grade," rectification is essential, unless the starting "cut" comes from a very careful rectifying operation.

*Example 5*

A nine-stage counterflow extraction system is charged:

At the first stage with a charge formed by a 65° C.–95° C. gasoline fraction having a sp. gr. of 0.7264 at 20° C. containing 32.28% by wt. of aromatics. These are formed by benzene (95% by wt.) and toluene (5%);

At the first stage with a solvent mixture formed by 14% water and 86% morpholine (by weight) with a weight ratio solvent/charge of 4.14/1 corresponding to a volume ratio 3:1;

At the seventh stage with water for a weight ratio charge/water of 3:1;



At the eighth stage with water as at the seventh stage;

At the ninth stage pentane, for a weight ratio charge/pentane of 14.5 to 1.

By keeping the system at 25° C. and at atmospheric pressure there are obtained:

(1) an extract which, freed from water and solvent by distilling the azeotropes aromatics-water and splitting them up by condensation and decantation, is rectified to give:

(a) 7.63% by wt. of a low-boiling fraction having a sp. gr. of 0.6904 at 20° C., consisting, on a weight basis, of 32.43% of benzene, and of 67.57 pentane: this fraction is admixed with the refined fraction to be rectified, so as to recover the pentane;

(b) 89.61% by weight of a fraction, having a sp. gr. of 0.8772 at 20° C., formed by "nitration grade" benzene, consisting of 99.32% benzene, by weight;

(c) 2.76% by weight of a fraction, having a sp. gr. of 0.8772 at 20° C., which, after rectification gives a "head" product containing 85.45% by weight of abs. benzene: this is added to the "nitration grade" benzene of (b) above. The balance 14.55% by weight is a "tail" consisting of slightly impure toluene.

(2) A refined product which, combined with the "heads" of the rectification of the extract, is rectified, in turn, to recover the pentane and to give, as a pentane-free product, a paraffinic fraction having a sp. gr. of 0.6708 at 20° C. Said fraction contains, on a weight basis, 3.49% of aromatics, and 96.51% of aliphatic hydrocarbons.

A "nitration grade" benzene is eventually obtained, having a purity of 99.37% (weight basis).

The 92.87% by weight of the benzene present in the starting mixture has thus been recovered.

*Example 6*

A ten stage counterflow extraction system is charged:

At the seventh stage with a gasoline fraction (65° C.–95° C.) having a sp. gr. of 0.7264 at 20° C., which contains, on a weight basis, 32.28% aromatics and 67.72% aliphatics. The aromatic fraction consists of 95% benzene of 5% toluene (by weight) and with a recycle (taken from the "heads" of the rectification column of the aromatics) which is formed by 67.55% benzene and 32.45% aliphatic hydrocarbons. This recycle represents 1.3% of the total charge;

At the first stage with a solvent mixture consisting of morpholine (86% by weight) and water (14% by wt.) with a solvent/charge volume ratio of 3:1 corresponding to a weight ratio of 4.14 to 1;

At the eighth stage with water in an amount corresponding to a charge/water ratio of 4.54 to 1;

At the ninth stage with water, as for the eighth stage;

At the tenth stage with water in an amount corresponding to a charge/water ratio of 1.82:1 by weight, and with recycled benzene in an amount corresponding to a charge/benzene weight ratio of 8.3 to 1.

By keeping the extraction system at 25° C., under atmospherical pressure, there are obtained:

(1) a refined product which, stripped of the entrained solvent and water, is formed by 99.13% by weight of aliphatic hydrocarbons and 0.87% by weight of aromatic hydrocarbons. These latter consist of 17.72% by weight toluene and 82.28% by weight benzene;

(2) an extract which, stripped of water and solvent and after having recycled aromatic hydrocarbons at the foot of the extraction column, is rectified to give:

(a) 4.32% by weight of a fraction, having a specific gravity of 0.8257 consisting, on a weight basis, of 67.55% of aromatics, and 32.45% of aliphatic hydrocarbons: the fraction is recycled to the extraction column;

(b) 94.13% of "nitration grade" benzene: this has an abs. benzene content, by weight, of 99.57% and a sp. gr. of 0.878 at 20° C.;

(c) 1.62%, by weight, of a high-boiling fraction having a sp. gr. of 0.8765 at 20° C.: this fraction, after further fractional distillation, gives 75.8% of abs. benzene,

by weight, and 24.2% toluene, along with a small percentage of benzene as an impurity.

The "heads" of the latter fractional distillation are combined with the "nitration grade" benzene obtained from the rectification of the extract: there is obtained, in total, a "nitration grade" benzene having purity of 99.57%. The yields of benzene is 98.5%.

Instead of effecting a reforming under extremely severe conditions to obtain a high content of aromatics in the produced gasoline, which procedure would entail, of necessity, a short life of the catalyst and low yields of liquid products, it is possible to combine, with advantage, the reforming process with the extraction process according to this invention.

If the reforming is effected under not too severe conditions, it is possible, in fact, to obtain a high gasoline output, with slight losses due to gasification, along with a longer life of the catalyst. By adopting the process of this invention, with low solvent/charge ratios, it is possible to obtain a gasoline enriched in aromatics: as a matter of fact the process of this invention is such as to permit to extract, from a given mixed hydrocarbonaceous charge, the maximum possible yield of aromatics with the minimum possible expenditure.

An example of the procedure outlined above will now be given, and illustrated by the flow diagram of FIG. 3.

*Example 7*

Having now specific reference to the flow-sheet FIG. 3, it will be seen that the charge, consisting of straight-run gasoline, is sent to the reforming stage **81**: the products thus obtained is sent to the rectification column **82**, in which the gaseous products are stripped and the heavy fraction, consisting of reformed gasoline, is sent to the extraction system. Said reformed gasoline is stored in the reservoir **83** and, via the pump **84**, is sent to the heat-exchanger **85** wherein it is brought to the proper temperature and then introduced in the extraction apparatus **86** in counterflow relationship with the solvent mixture introduced from the discharge end for the refined product. In a point below the point of introduction of the gasoline charge, there is introduced water coming from the recovery column **87** of the extract. Water can be fed either through the settling tank **88** and the pump **89**, or through the cooler **90** and the pump **91**, to adjust the purity of the extract.

The extract, via the heat-exchanger **92**, the storage tank **93**, the pump **94** and the heater **95**, is introduced in the extract-recovery column **87**.

The refined product, washed beforehand with water in the tower **96** in order to recover possibly entrained solvent, is sent to the points of use or sent back by reforming. From the top of the extract-recovery column **87** there are obtained the aromatic gasoline and water: these, condensed in the condenser **97**, are split up in the storage-settler **88**, wherefrom the two layers are separately drawn. One layer is the highly aromatic gasoline, B, and the other consists of water: the latter is brought back to the tower **96** and the extraction system proper **86**.

By maintaining the reforming run under not too severe conditions it is thus possible to obtain a gasoline having a very high octane rating: it would be virtually impossible to achieve such a favorable result with the reforming run only, unless working under very severe conditions as regards the yields and the service life of the catalyst.

What we claim is:

**1.** A process for recovering aromatic hydrocarbons from mixtures which contain said aromatic hydrocarbons admixed with other, non-aromatic hydrocarbons, comprising the steps of contacting said hydrocarbonaceous mixtures with a composite solvent mixture consisting of from about 95% to about 50%, by volume, of morpholine and from about 5% to about 50%, by volume, of water at a temperature in the range 0° C. to 95° C., increasing the water content of the solvent during the operation, and continuing the operation until substantially all the aromatic fractions of said hydrocarbonaceous mixture have been extracted therefrom, subjecting the extract thus obtained to azeotropic distillation at a temperature below about 100° C., contacting said extract with a recycled fraction consisting of aliphatic hydrocarbons having a boiling point lower than the boiling point of that aromatic hydrocarbon to be recovered which has the lowest boiling point of such aromatic hydrocarbons, and separating the individual aromatic hydrocarbons thus obtained.

**2.** A process for recovering aromatic hydrocarbons from mixtures which contain said aromatic hydrocarbons admixed with other, non-aromatic hydrocarbons, comprising the steps of contacting said hydrocarbonaceous mixtures with a composite solvent mixture consisting of from about 95% to about 50%, by volume, of morpholine and from about 5% to about 50%, by volume, of water at a temperature in the range 0° C. to 95° C., increasing the water content of the solvent during the operation, and continuing the operation until substantially all the aromatic fractions of said hydrocarbonaceous mixture have been extracted therefrom, subjecting the extract thus obtained to azeotropic distillation at a temperature below 100° C., contacting said extract with a recycled fraction consisting of aromatic hydrocarbons having a boiling point lower than the boiling point of that aromatic hydrocarbon to be recovered which has the lowest boiling point of such aromatic hydrocarbons, and separating the individual aromatic hydrocarbons thus obtained.

**3.** A process for recovering aromatic hydrocarbons from a hydrocarbonaceous mixture which contains said aromatic hydrocarbons mixed with other, non-aromatic hydrocarbons, comprising the steps of adding to the mixture a composite solvent consisting of from about 95% to about 50%, by volume, of morpholine and from about 5% to about 50%, by volume, of water at a temperature of about 25° C. and at approximately atmospheric pressure, the ratio of solvent to hydrocarbonaceous mixture being from about 1:1 to about 30:1, and extracting the aromatic fractions from the mixture of solvent and hydrocarbonaceous mixture while adding to the mixture of the solvent and hydrocarbonaceous mixture a non-aromatic hydrocarbon having a boiling point lower than the boiling point of the lowest-boiling-point aromatic hydrocarbon to be recovered, freeing the non-aromatic hydrocarbon from the solvent by counterflowing with water, stripping the aromatic hydrocarbons from the extract by azeotropic distillation at a temperature below 100° C., rectifying the stripped aromatic hydrocarbons to separate them into individual aromatic hydrocarbons, and feeding the water obtained during distillation and rectification back to the mixture of solvent and hydrocarbonaceous mixture, a portion of the aromatic hydrocarbonaceous fraction being recycled in the neighborhood of the point at which the aromatic fractions are extracted from the mixture of solvent and hydrocarbonaceous mixture.

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DELBERT E. GANTZ, *Primary Examiner.*

ALPHONSO D. SULLIVAN, *Examiner.*

P. P. GARVIN, H. LEVINE, *Assistant Examiners.*