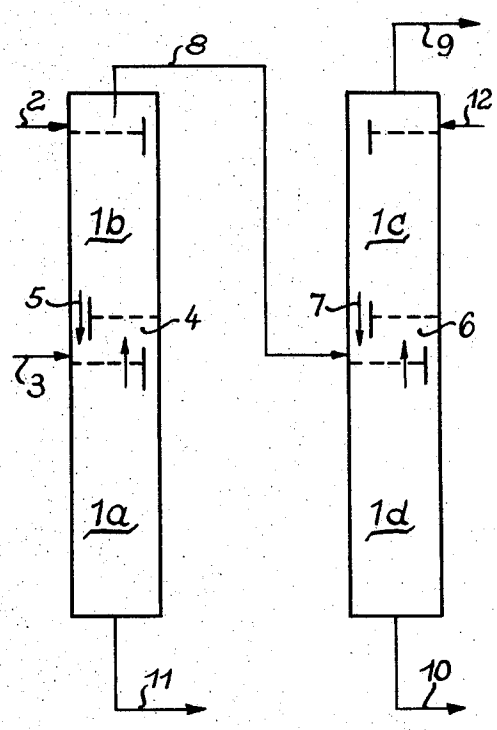
Jan. 12, 1971 G. LUTHER ET AL 3,554,873
SIMPLIFIED EXTRASTIVE DISTILLATION PROCESS FOR OBTAINING AROMATICS OF DIFFERING BOILING-POINT RANGES
Filed Oct. 11, 1967 2 Sheets-Sheet 1

Fig.1



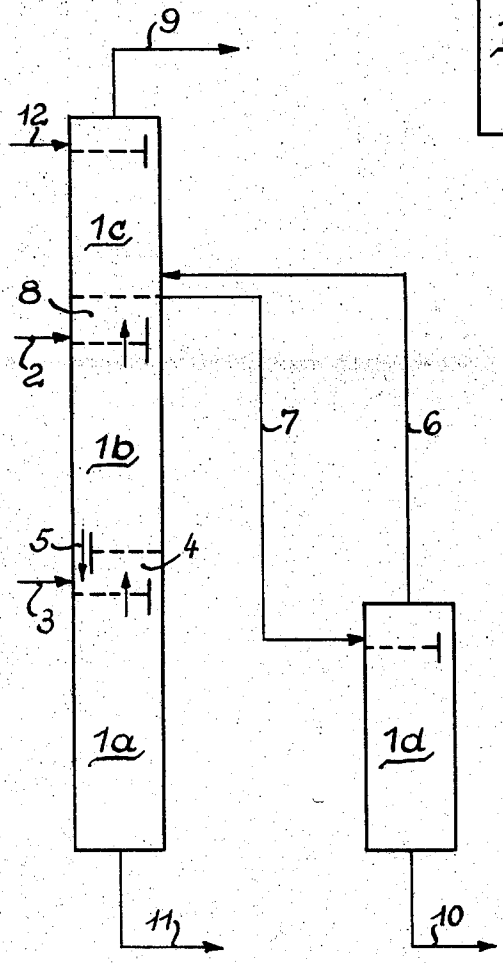


Fig.2

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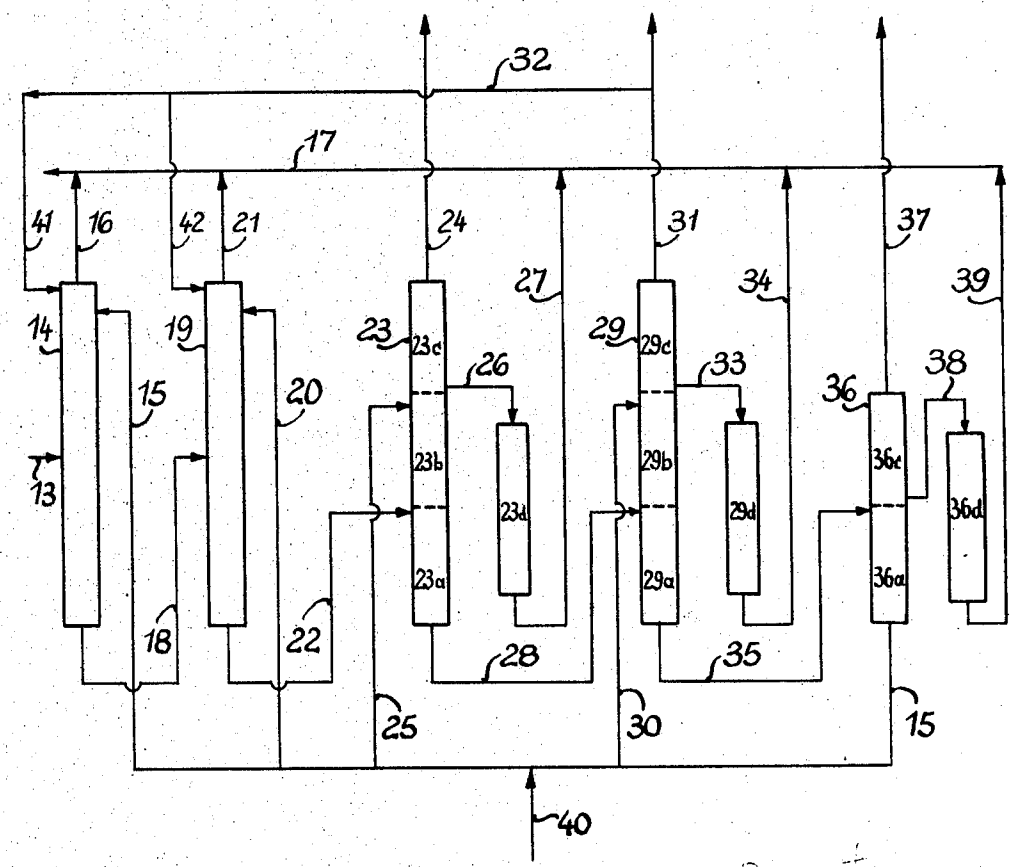
Fig. 3



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SIMPLIFIED EXTRACTIVE DISTILLATION PROCESS FOR OBTAINING AROMATICS OF DIFFERING BOILING-POINT RANGES

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14 Claims

ABSTRACT OF THE DISCLOSURE

An extractive distillation process for simultaneously obtaining aromatics with different boiling-point ranges from a feed stock containing varied amounts of non-aromatics along with the desired aromatics, said process being of the type wherein the feed stock is passed through at least one extractive distillation column and is contacted with a selectively active extraction agent to which aromatics other than the desired aromatics have been added, and wherein the mixture of aromatics and extraction agent is withdrawn from the bottom of the column while withdrawing the non-aromatics in vapor form from the top of the column, the improvement comprising passing the aromatics containing mass received from the said extractive distillation column through a plurality of in-series connected column systems, the number of said systems corresponding to the number of aromatics to be obtained and subjecting the mass in each of said column systems successively to an extractive distillation and to a normal distillation and recovering from each system the desired aromatic fraction.

CROSS-REFERENCES TO RELATED APPLICATIONS

Application Ser. No. 627,788, filed on Apr. 3, 1967, by the same inventors as the present application and patented May 20, 1969 as U.S. Pat. No. 3,445,537 and relating to "Extractive-Distillation Process for Obtaining Aromatics of Differing Boiling-Point Ranges" and assigned to the same assignee as the present application.

BACKGROUND OF THE INVENTION

The use of an extractive distillation process for obtaining pure aromatics from mixtures containing non-aromatic hydrocarbons together with the aromatics has been disclosed, for instance, in German published application No. 1,206,414. The feed stock in this process is introduced at about the vertical center of the extractive distillation column and is contacted with an extraction agent that flows from top to bottom through the column and is selectively active towards the aromatics. The aromatics are withdrawn in that process together with the extraction agent from the bottoms of the column and are separated by subsequent distillation. The nonaromatics are simultaneously caused to vaporize in the extractive distillation column and are withdrawn in vapor form through the top thereof.

This process, due to the simple arrangement, has many advantages over the liquid-liquid extraction. To name a few: An installation for recovery of the extraction agent from the raffinate is eliminated and, in general, complex mechanical installations are avoided, such as a multiplicity of revolving pumps and similar apparatus. The viscosity of the extracting agent is substantially reduced through the applied higher temperature, which

results in a material improvement of the product exchange between extraction agent and product to be extracted. One obtains, therefore, improved charge potentialities and can get along with smaller amounts of extraction agent at comparable performances. The dimensions of the extractor can also be reduced in most cases. These last two advantages result in cost-saving when the extractive distillation is compared with the liquid-liquid extraction.

The extractive distillation unfortunately has two shortcomings as against the liquid-liquid extraction. In the first place, there is a comparatively high portion of extraction liquid in the raffinate phase of the extractive distillation plant. In the second place, the problem of obtaining products with widely differing boiling-point ranges from the extractive distillation process is difficult to solve.

As stated in the copending case, it was found that aromatics appear along with the desired paraffins in the raffinate phase even when using extraction agents that are free of aromatics. This phenomenon is in particular noted in the raffinate phase of extractive distillation plants employed to separate aromatics from non-aromatics. For instance, working with a propylene carbonate as extraction agent there were found contents of benzene between 10% and 30% by weight in the raffinate phase, depending on working conditions. This high share of aromatics in the raffinate phase (paraffin fraction) amounts to an uneconomical and low yield, particularly where the feed material contains a low amount of aromatics. The extractive distillation method has therefore been used heretofore only for feed products with a content of non-aromatics up to about 20%, since in these cases the mentioned shortcomings were without significance, or otherwise the extractive distillation was carried out in combination with a liquid-liquid distillation.

The reason for the presence of aromatics in the raffinate phase (paraffin fraction) was found to be that two liquid layers are formed on the top plates of the extractive distillation column. In other words, there are concentration conditions which result in a system having a mixture gap.

In this copending process the non-aromatics containing mass drawn off from the extraction columns may be subjected to further treatment in separate auxiliary columns where any residues of lower boiling-point aromatics that may still be present are removed from the product, whereupon the bottom product of the auxiliary columns which substantially no longer contains any aromatics may be subjected to another extractive distillation. Preferably this is done by collecting the different masses of non-aromatics products from the several extractive distillation columns and subjecting them to another common extractive distillation. If desired, the final refining may also be effected in a normal distillation step.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide for a modification and improvement of the process of the copending application. In a more specific way, it is an object of the present application to dispense with the just-described after-treatment of the non-aromatics in a separate column or in several such columns. It is therefore also an object of the present invention to improve the economics of the earlier process in regard to necessary apparatus and operating costs by eliminating the separate column or columns for the after treatment.

The improvement consists in passing the mass to be processed after it comes from the extractive distillation column or columns through a plurality of in-series con-

nected column systems, the number of said systems corresponding to the number of aromatics to be obtained, and subjecting the mass in each of said column systems successively to an extractive distillation and to a normal distillation and recovering from each system the desired aromatic fraction.

The novel features which are considered as characteristic for the invention are set forth in particular in the appended claims .The invention itself, however, both as to its construction and its method of operation, together with additional objects and advantages thereof, will be best understood from the following description of specific embodiments when read in connection with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWING

FIG. 1 illustrates, in a diagrammatic form, one single column system for use in the process of the invention;

FIG. 2 illustrates a modified form of such column system; and

FIG. 3 is a flow sheet illustration of the extractive distillation process and apparatus of the invention. Secondary parts of apparatus such as evaporators, cooling-apparatus, heat exchangers, pumps and valves are omitted in the flow sheet to give a clear picture of the process.

BRIEF DESCRIPTION OF THE PREFERRED EMBODIMENTS

The method of the invention accordingly differs from that of the mentioned copending application in that further separation of the extraction product mixture enriched by the preceding extractive-distillations, into individual fractions, takes place with the aid of a column system whose column sections not only perform the function of extractive-distillation but also the function of normal distillation. At the top of the extractive-distillation portion composed of the extractive stripper section and the extractive rectifying section there are drawn off a lower boiling aromatic component, such as benzene, and a refined (non-aromatics) product, having a boiling point within the range of the next higher boiling aromatic component, such as toluene.

This top product of the extractive-distillation is subsequently split, by distilling, into benzene and the non-aromatics of the toluene boiling range.

Those aromatics which have a higher boiling point range than that obtained from the first column system may be similarly obtained from downstream connected column systems.

The concept of the column system used in the method of the invention is best explained by reference to FIG. 1. The column system shown therein consists of the four column sections 1*a*, 1*b*, 1*c* and 1*d* respectively. The column sections 1*a* and 1*b* both primarily operate in the mode of extractive distillation, section 1*a* being an extractive stripper section and section 1*b* being an extractive rectifying section. The column sections 1*c* and 1*d* both operate in the mode of normal distillation, section 1*c* being a distillative stripper section.

The extractive column sections 1*a* and 1*b* are interconnected by the product streams **4** and **5** in the same way as in a normal extractive-distillation column, both on the vapor side as well as on the liquid side.

The distillative column sections 1*c* and 1*d* are interconnected by the product streams **6** and **7** as in normal distillation, on the vapor side as well as on the liquid side.

The extractive column, consisting of sections 1*a* and 1*b*, is connected at its head by the product stream **8** to the distillative column consisting of sections 1*c* and 1*d*. This product stream **8**, which may be vapor or liquid, is fed to the uppermost plate of the distillative stripper section 1*d*.

In the operation of such a column system, extraction agent is supplied through a pipeline **2** to the uppermost plate of the extractive rectifying section 1*b*, a feed mixture obtained from the bottom of a preceding column being supplied through a pipeline **3** to the uppermost plate of the extractive stripper section 1*a*, whilst a reflux is supplied through a pipeline **12** to the uppermost plate of the distillative rectifying section 1*c*.

The extraction product (e.g. aromatic) fraction to be obtained from such system is tapped off through pipeline **9**, whilst the refined product (e.g. non-aromatic) fraction, having the boiling range of the extraction product fraction of the next column system, is tapped off through pipeline **10**.

The bottom of the extractive stripper section 1*a*, consisting substantially of extraction agent and residual aromatics is tapped off through pipeline **11**. Where appropriate, this bottom product may be supplied to a subsequent column system for further processing.

The column system shown in FIG. 1 may take various forms; the individual column sections may, for instance, be arranged as illustrated in FIG. 2, in which the reference numerals have the same significance as in FIG. 1. The embodiment illustrated in FIG. 2 differs from that of FIG. 1 in that the extractive rectifying section 1*b* and the distillative rectifying section 1*c* are both constructed as parts of a single column unit also including the extractive stripper section 1*a*. Other forms are also possible.

As regards the procedure (a) referred to hereinbefore, this can be accomplished by allowing to remain in the extraction agent those extraction product-like substances (e.g. aromatics) originating from the feedstock which are not to be extracted or are to be only partially extracted. It is of course also possible to add to the extraction agent other extratcion product-like substances (aromatics) not to be extracted and not present in the feedstock. For example, it is possible to add toluene to the extraction agent if benzene and xylene are to be obtained by extractive-distillation of a feedstock containing these aromatics mixed with paraffins. It is also possible to add to the extraction agent xylene or C_9 aromatics which are not to be extracted from such a feedstock in a plant in which only benzene is to be extracted. The amount of extraction product-like substances (e.g. aromatics) to be added to the extraction agent may be up to 20% by volume referred to the extraction agent.The amount of such an additive also depends on the pressure at which extractive-distillation is performed. This is subject to the rule that under condtions of elevated pressure (positive pressure) the amount of additive may be correspondingly reduced.

Increasing the working pressure of the first extractive-distillation column results in an increase of the solubility of the refined product (e.g. non-aromatics) in the extraction agent. This procedure leads to some deterioration in the selectivity of the extraction agent. The resultant disadvantages may however be compensated by freeing the extraction agent/refined product mixture, in a downstream connected extractive-distillation system operated at lower pressure (atmospheric or subatmospheric pressure), of refined product fractions which can be removed only with great difficulty. The method of operation according to the invention is however not confined to the use of positive pressure in the first extractive-distillation column.

According to a further feature of the invention, the extractive rectifying section 1*b* may be omitted if a yield of the next higher boiling extraction product (e.g. aromatic) fraction is not required. Moreover, it is possible to dispense with the distillative stripper section 1*d* if a yield and purification of the heads products of this column system (aromatic) are not required. Finally, an additional column section may be mounted upon the head of the extractive rectifying section 1*b*, the head of the aforementioned column section being fed with extraction product-like substances (e.g. aromatics) or a frac-

tion containing such substances and which serves for the additional extraction agent recovery.

The method of the invention may further include the step of returning to the extractive-distillation system a bottom product obtained from the last extractive stripper section and which consists of extraction agent feed of the required extraction products e.g. aromatics. The sensible heat of this bottom product as well as of the bottom product of the extractive-distillation columns operated at higher pressures may then be employed for heating the extractive-distillation columns operated at lower pressures and/or the distillative stripper sections.

At the head of the extractive stripper secion certain losses of extraction agent may occur. However, these losses may be easily avoided by arranging for a small part of the high boiling extraction products-like substances (e.g. aromatics) which are not to be extracted to be introduced as washing medium into the extraction column above the extraction agent feed, instead of adding these substances directly to the extraction agent. These substances will then wash the extraction agent from the vaporized refined product (e.g. non-aromatics.)

It should be understood that the method according to the invention is not confined to the use of a particular extraction agent, the criterion for suitability being an adequate selectivity for separating the extraction products from the refined products. The following substances are suitable extraction agents for separating aromatics from paraffins: propylene carbonate, N-oxyalkyl derivatives for 1,3-propane diamine and N-substituted morpholine. This enumeration does not, however, represent any limitation to the aforementioned compounds. Many other compounds may also be employed as extraction agents. The method of the invention can be employed for the separation of any desired kinds of substances with any desired extraction agents if the attainably purity of the heads product of extractive-distillation is limited by a miscibility gap.

Referring now to FIG. 3, this illustrates in block diagram-flowsheet form the principal plant components used, typically, to carry out the method of the invention for separating aromatics from paraffins: in this figure, all auxiliary devices which are of a lower order of significance for the method, such as heaters, coolers, heat exchangers, pumps, valves and the like, have been omitted in the interest of clarity. The process will be described in relation to its use for refining a feedback consisting of a hydrocarbon mixture having the following composition by weight (in. kg.):

Non-aromatics	460
Benzene	300
Toluene	150
Xylene	70
C_9 aromatics	20

For the purpose of this description, a feed rate of 1000 kg. per hour will be assumed and the quantities of materials are expressed on this hourly basis.

When using a feedstock of the above-mentioned composition, the weight ratio of extraction agent to feedstock should be equal to 3, that is 3000 kg. of extraction agent are employed. The extraction agent consists of approximately 2880 kg. propylene carbonate and approximately 120 kg. toluene. Approximately 80% of this quantity of toluene is fed through a line **41** to the head of a first extractive-distillation column **14** and the remaining 20% is fed through a line **42** to the head of a second extractive-distillation column **19**. The toluene employed in the extraction medium may at least partly originate in the feedstock being extracted therefrom in the process as will be described and fed by recycle line **32** to the pipelines **41** and **42** from the toluene output line **31**.

The feedstock is introduced through a line **13** to a middle plate of the first extractive-distillation column **14** which operates at a pressure of approximately 3.5 atm. The appropriate amount of the propylene carbonate constituent of the extraction agent is fed through a line **15** to one of the upper plates of the column **14**. A large proportion of the non-aromatics in the feed mixture escapes in vapour form through a line **16** at the head of the column and is fed into a manifold line **17**. The bottom product of the column **14** which contains the aromatics and the extraction agent in addition to the residue of non-aromatics passes through a line **18** to constitute the feed for the second extractive-distillation column **19** which operates at normal (atmospheric) pressure. The feed in this case is also introduced to one of the middle plates of the column **19**. Propylene carbonate is fed through the line **20** to one of the upper plates of the column **19**. In this column **19**, the non-aromatics escape in vapour form through a line **21** at the head of the column and, after condensation, are also fed into the manifold line **17**.

The bottoms product of the column **19** is conveyed through a line **22** into a first column system **23** of the form illustrated in FIG. 2 sections **23***a*, **23***b* and **23***c* corresponding with sections **1***a*, **1***b* and **1***c* of FIG. 2 and being combined in a single column. The side column **23***d* of the system **23** is a distillative stripper section corresponding with section **1***d* in FIG. 2. The line **22** feeds between the extractive rectifying section **23***b* and the extractive stripper section **23***a* of that system, which system serves for the stripping of benzene from the said bottom product, benzene being discharged via a line **24**. Propylene carbonate is fed through a line **25** to the uppermost plate of the extractive rectifying section **23***b*. A fraction containing the non-aromatics is drawn off from the bottom of the distillative rectifying section **23***c* and passes through a line **26** into the side column **23***d*, from which the non-aromatics are removed at the bottom and are fed into the manifold line **17** through a line **27**.

The bottoms product of the extracting stripper section **23***a*, which contains residual aromatics in addition to the extraction medium, is pumped through a line **28** into a second column system **29**, again arranged as described with reference to FIG. 2, which serves the purpose of stripping the toluene from said bottom product. Propylene carbonate is supplied through a line **30**. The extracted toluene is drawn off through a toluene output line **31**, some being recycled, as described, to the extractive distillation columns **14** and **19** through the recycle line **32**. The side column **29***d* serves as distillative stripper section and is connected to the bottom of section **29***c* by a line **33**. The non-aromatics are drawn off through a line **34** which extends into the manifold line **17**.

The bottoms product of the column system **29**, containing the xylene as well as the higher boiling aromatics and extraction agent is finally pumped through a line **35** into the final column system **36** which serves for extracting the xylene.

The column system **36** is again arranged as described in relation to FIG. 2 but differs in that, since the yield and purity of the C_9 aromatics to be obtained by extraction of xylene from the bottom product of the column system **29** is immaterial, the system **36** does not include an extractive rectifying section, nor does it have an extraction agent feed.

The xylene is drawn off through a line **37**. The side column **36***d* functions as distillative stripper section and is connected to the bottom of the column section **36***c* via a line **38**. The remaining non-aromatics and the residual aromatics with a higher boiling point than xylene are withdrawn via a line **39** and fed to the manifold line **17**. The recovered extraction agent from the bottom of section **36***a* (predominantly propylene carbonate since toluene has been stripped therefrom by the column system **29**) is delivered for reuse via the line **15** and the branch lines **20**, **25**, **30**. Fresh propylene carbonate is supplied through a line **40** when necessary to make good losses and to introduce this constituent of the extraction agent into the process when the plant is started up.

The following yields were obtained with a plant arranged and operated as described above and using the specified feedstock:

Benzene=287 kg.=96.7% of the feedstock benzene.
Toluene=115 kg.=76.6% of the feedstock toluene.
Xylene=69 kg.=98.6% of the feedstock xylene.

Substantially 100% of the non-aromatics of the feedstock were drawn off through the manifold line **17**. The method according to the invention is therefore characterised by good separation and a high yield for all required fractions. All aromatics obtained had a high degree of purity.

In many cases, it may be desirable for the higher boiling aromatics, for example C_9 aromatics, to be extracted in addition to benzene, toluene and xylene. If these aromatics are required to be extracted from the mixture obtained from the bottom of the column section **36***d*, such mixture may be processed in a further column system. If no quantitative separation of the C_9 aromatics is envisaged, such processing may be accomplished in a simple stripper column.

With respect to the first column system **23**, in which the stripping off of benzene is carried out, the following conditions may prevail:

The feed stock, after having been freed in extractive-distillation columns **14** and **19** of a large portion of the non-aromatic constituents, is withdrawn through conduit **22** and introduced into the first column system **23**, for stripping off of benzene.

The product introduced into column system **23** has the following composition:

	Kg.
Non-aromatics	119.32
Benzene	288.8
Toluene	262.96
Xylene	69.16
C_9-aromatics	19.76

With respect to the toluene content, it has to be considered that in addition to the toluene content of the feed stock (150 kg.) also about 120 kg. toluene had been introduced into the system with the extraction agent.

The benzene which has been stripped off in column system **23**, is withdrawn through conduit **24**, and has the following composition:

	Kg.
Benzene	286.8565
Toluene	0.0287
Non-aromatics	0.1148

Thus, the benzene is of a purity of 99.95%.

The non-aromatics which are separated in column system **23**, are withdrawn through conduit **27**. This fraction has the following composition:

	Kg.
Non-aromatics	109.032
Benzene	1.943
Toluene	19.932

The aromatics having boiling points higher than benzene accrue in the sump of extractive column **23***a* and are pumped through conduit **28** into the next following column system **29** in which toluene is stripped off.

This fraction has the following composition:

	Kg.
Non-aromatics	10.17
Toluene	242.92
Xylene	69.16
C_9-aromatics	19.76

The now following column systems for the stripping off of toluene and xylene operate in substantially the same manner as described above with respect to the stripping off of benzene.

Without further analysis, the foregoing will so fully reveal the gist of the present invention that others can by applying current knowledge readily adapt it for various applications without omitting features that, from the standpoint of prior art, fairly constitute essential characteristics of the generic or specific aspects of this invention and, therefore, such adaptations should and are intended to be comprehended within the meaning and range of equivalence of the following claims.

What is claimed as new and desired to be protected by Letters Patent is set forth in the appended claims:

1. Extractive distillation process for simultaneously obtaining different aromatics with different boiling point ranges from a feed stock containing varied amounts of non-aromatics along with the desired aromatics comprising passing the feed stock through at least one extractive distillation column while contacting it in said column with an at least partially non-aromatic selectively active extraction agent containing aromatics other than the aromatics to be recovered; withdrawing a liquid extract of aromatics and extraction agent from the bottom of said column while withdrawing the non-aromatics in vapor form at the top of said column; then passing said liquid extract of aromatics and extraction agent through a plurality of column systems arranged in series, one of said systems being provided for each desired type of aromatic; subjecting said liquid extract of aromatics and extraction agent in each of said systems to an extractive distillation followed by a conventional distillation, and withdrawing the aromatic to be recovered from the respective rectifying distillation, and also withdrawing non-aromatic from said stripping distillation.

2. Process according to claim **1** wherein the mixture of extraction agent, aromatics and non-aromatics in at least one of said column systems is passed in sequence through an extractive stripping section, an extractive rectifying section, a distillative rectifying section and a distillative stripping section.

3. Process according to claim **2** wherein the mixture of extraction agent, aromatics and non-aromatics in at least one of said column systems is passed from an extractive stripping section to a superposed extractive rectifying section and is then passed into a second column formed of two superposed distillation sections.

4. Process according to claim **2** wherein the mixture of extraction agent, aromatics and non-aromatics in at least one of said column systems is passed in succession through a first column composed of three sections comprising an extractive stripping section, an extractive rectifying section and a distillative rectifying section and then into a second column composed of a distillative stripping section.

5. Process according to claim **1**, wherein the amount of aromatics that is added to the extraction agent is up to 20% by volume relative to the volume of the extraction agent.

6. Process according to claim **1** wherein the mixture of extraction agent, aromatics and non-aromatics is passed through at least two successively arranged extractive distillation columns, each successive column being operated with reduced pressure relative to the preceding column.

7. Process according to claim **6** wherein the first of said extractive distillation columns is operated at a pressure above atmospheric.

8. Process according to claim **1** wherein in the last of said columns, said mixture of extraction agent, aromatic and non-aromatic is passed from an extractive stripper section directly to a distillative rectifying section and from there to a distillative stripper section, the desired aromatic fraction being withdrawn from the top of said distillative rectifying section and the refined non-aromatics product being withdrawn from the bottom of the distillative stripper section.

9. Process according to claim **1** wherein in at least one of said systems the mixture of extraction agent, aromatics and non-aromatics is passed from an extractive stripper section to an extractive rectifying section and then to a

distillative rectifying section, from which the desired aromatics fraction is withdrawn at the top.

10. Process according to claim 2 wherein the extraction agent that has been stripped from the aromatics to be recovered is withdrawn from the last extractive stripper section and is fed back into the distillation process.

11. Process according to claim 7 wherein the sensible heat of the bottoms product of the last extractive stripper column and of the high-pressure operated extractive distillation column is employed for heating the lower-pressure extractive distillation columns and the other stripper columns.

12. Process according to claim 1 wherein a portion of the aromatics used in the extraction agent is introduced into the extractive distillation columns separate from the extraction agents at a place above the inlet for the remainder of the extraction agent.

13. An extractive distillation apparatus, comprising at least two extractive distillation columns arranged in series and having connected to the last extractive distillation column a plurality of column systems arranged in series, at least one of said column systems being comprised of an extractive stripper section, an extractive rectifying section superposed on said extractive stripper section, and a distillative rectifying section superposed on said extractive rectifying section, and, connected with said distillative rectifying section, a separate distillative stripper section, means for introducing the mixture of extraction agent, aromatics and non-aromatics obtained from said extractive distillation columns at a point in proximity to the borderline between the extractive stripper section and the extractive rectifier section of each column system, outlet means at the top of said distillative rectifying section for the desired aromatics, conduit means for passing the extraction agent and non-aromatics from the bottom of the extractive stripper section to the next column system and conduit means for feeding back the extraction agent from the last extractive stripper section to the first extractive distillation column.

14. An extractive distillation apparatus, comprising at least two extractive distillation columns arranged in series and having connected to the last extractive distillation column a plurality of column systems arranged in series, at least one of said column systems being comprised of an extractive rectifying section superposed on said extractive stripper section, and a distillative rectifying section and a distillative stripper section, the last two sections being superposed on each other, means connecting the top of said extraction rectifying section and a point in proximity to the boundary between said two distillative sections, outlet means at the top of said distillative rectifying sections for the desired aromatics, conduit means for passing the extraction agent and non-aromatics from the bottom of the extractive stripper section to the next column system, and conduit means for feeding back the extraction agent from the last extractive stripper section to the first extractive distillation column.

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