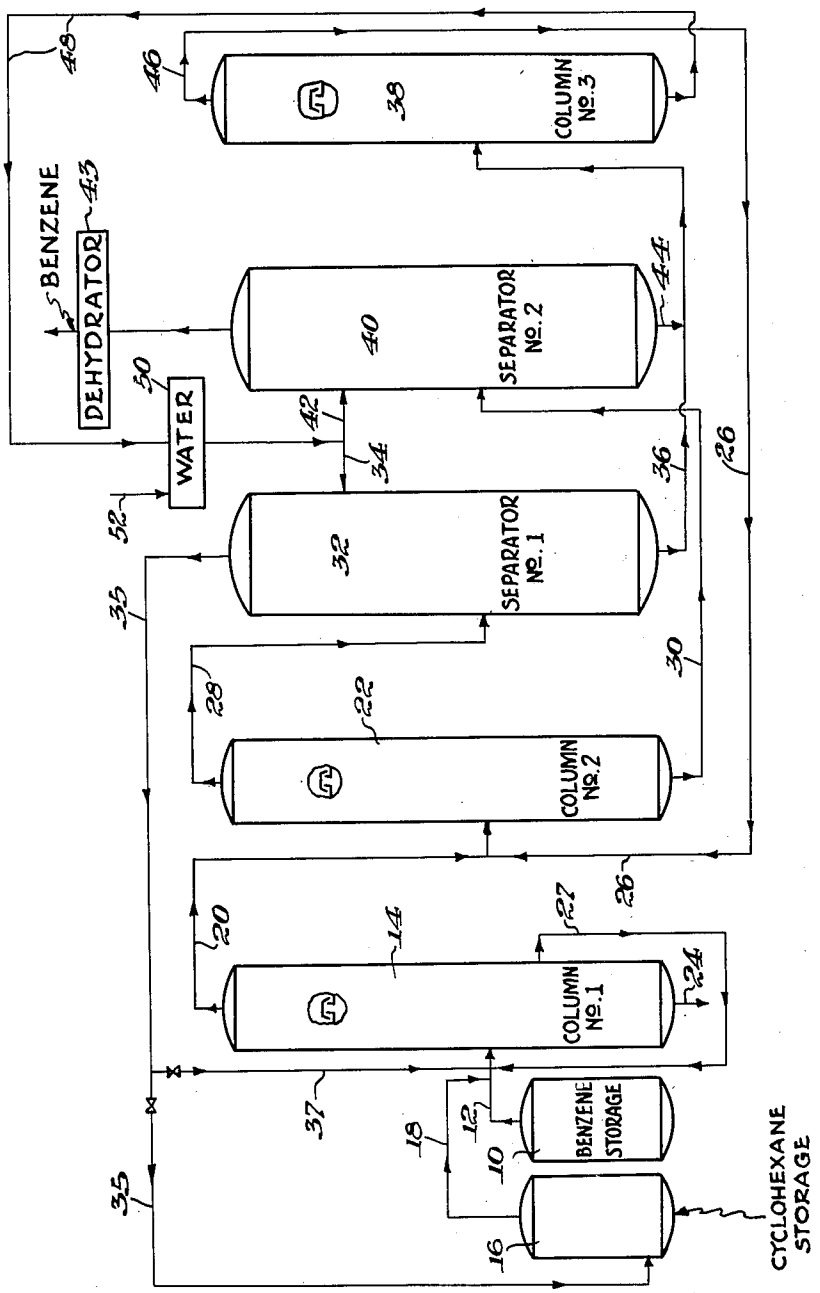
Jan. 8, 1952 J. R. ANDERSON 2,581,344

PURIFICATION OF DISTILLED COKE-OVEN BENZENE BY DISTILLATION WITH ADDED HYDROCARBON MATERIAL

Filed June 4, 1948



INVENTOR.
JOHN R. ANDERSON.
BY Edmund G. Borders
his ATTORNEY.

Patented Jan. 8, 1952 2,581,344

UNITED STATES PATENT OFFICE

2,581,344

PURIFICATION OF DISTILLED COKE-OVEN BENZENE BY DISTILLATION WITH ADDED HYDROCARBON MATERIAL

John R. Anderson, Pittsburgh, Pa., assignor to Koppers Company, Inc, Pittsburgh, Pa., a corporation of Delaware

Application June 4, 1948, Serial No. 31,098

7 Claims. (Cl. 202—42)

1

This invention relates to the purification of aromatic hydrocarbons. More particularly this invention relates to a two-stage azeotropic distillation method for separating pure aromatic hydrocarbons from mixtures that contain normally higher-boiling non-aromatic hydrocarbons which form azeotropes or non-ideal systems with the aromatic hydrocarbon component of the mixture.

It is well known that non-aromatic hydrocarbons may be separated from aromatic hydrocarbons by one appropriate azeotropic distillation in an efficient rectifying column, in mixtures in which the non-aromatic hydrocarbons normally have boiling points not significantly higher than the aromatic components in the given mixture. For example, cyclohexane, a non-aromatic hydrocarbon normally boiling at 80.7° C., may be separated from benzene, an aromatic hydrocarbon normally boiling at 80.1° C., by azeotropic distillation with acetone. Adequate azeotropic distillation methods whereby non-aromatic hydrocarbons may be separated from mixtures in which the aromatic component or components have normal boiling points significantly lower than the normal boiling points of the non-aromatic components, however, have heretofore not been discovered. For example, benzene, an aromatic hydrocarbon normally boiling at 80.1° C., forms a binary azeotrope with normal heptane, a non-aromatic hydrocarbon normally boiling at 98.4° C., and the separation of normal heptane from benzene by azeotropic distillation with, for example, acetone, is a matter of considerable difficulty where the problem is that of obtaining pure benzene from such mixtures.

An important source of aromatic hydrocarbons is light oil which is obtained as a byproduct of the coking of coal. Benzene derived from light oil, by sulfuric acid-washing and efficient rectification, contains non-aromatic hydrocarbons having normal boiling points and azeotropic properties with benzene such that pure benzene cannot be obtained by efficient rectification. The principal impurities in coke oven benzene normally boil above 90° C., and among the most troublesome impurities so far as purification is concerned are normal heptane having a normal boiling point of 98.4° C., 2,2,4-trimethylpentane having a normal boiling point of 99.3° C., and methylcyclohexane having a normal boiling point of 100.9° C., while benzene has a normal boiling point of 80.1° C.

It has been found that benzene and the paraffins and naphthenes which are usually associated with coke oven benzene form non-ideal

2

and in most cases azeotropic systems with benzene. Further it has been found that these non-ideal or azeotropic systems boil very close to the normal boiling point of benzene and that they are composed almost entirely of benzene. Therefore, coke oven benzene which contains the normally high-boiling impurities which form azeotropes with benzene cannot be purified satisfactorily by ordinary rectification, no matter how efficient the rectifying equipment employed. In the most efficient fractionations, the overhead distillate will consist of an azeotrope or a mixture of azeotropes.

It has become a common practice to submit coke oven benzene to azeotropic distillation with polar azeotropic agents. A commonly used polar azeotropic agent for separating paraffin and naphthene impurities from coke oven benzene is acetone which is commonly known as a selective azeotropic agent in that it does not form an azeotrope with benzene. I have found, however, that acetone either does not form azeotropes with some of the higher-boiling non-aromatic hydrocarbons present in coke oven benzene or that if acetone forms such azeotropes their compositions are such that no less than 100 volumes of acetone are required to remove one volume of the higher-boiling contaminants. As a result, the purification of coke oven benzene by azeotropic distillation with acetone is unsatisfactory; the benzene remains somewhat impure, and the recovery of acetone for recycling is an expensive operation because a considerable proportion of water must be added to the acetone distillate so that the small proportion of non-aromatic hydrocarbons can be made to separate as a second liquid phase.

Another commonly used polar azeotropic agent for the purification of coke oven benzene is methanol. This alcohol forms azeotropes with all of the non-aromatic hydrocarbon contaminants usually associated with coke oven benzene, but it is non-selective in its azeotropic behavior in that it also forms an azeotrope with benzene. Many tests have shown that the benzene azeotrope of methanol interferes in the separation of the non-aromatic hydrocarbons usually associated with coke oven benzene. For example, where coke oven benzene, which contains normal heptane and methylcyclohexane, is azeotropically distilled with methanol, normal heptane is concentrated somewhat in the lower-boiling distillates but methylcyclohexane is concentrated somewhat in the higher-boiling distillates. In fact, both normal heptane and methylcyclohexane are found in all of the distillates derived from such an azeotropic distillation of coke oven benzene with

methanol, presumably because of the closely related boiling points of the methylcyclohexane-methanol, normal heptane-methanol and benzene-methanol azeotropes.

Repeated tests have shown that the difficulties encountered in the use of methanol as an azeotropic agent for the purification of coke oven benzene are also found where employing other alcohols as azeotropic agents, for example ethanol, normal propanol, iso-propanol and butanol. The same difficulties are also encountered where employing methyl ethyl ketone as an azeotropic agent for the purification of coke oven benzene. Hence, methanol, ethanol, normal propanol, iso-propanol, butanol, anad methyl ethyl ketone, which are water soluble polar azeotropic agents, are not suitable for removing the non-aromatic hydrocarbons which are usually associated with coke oven benzene.

Other aromatic hydrocarbons, such as toluene, ethylbenzene, orthoxylene, metaxylene, paraxylene, and naphthalene, form azeotropes with non-aromatic hydrocarbons, and I have found that where these aromatic hydrocarbons are associated with normally somewhat higher-boiling non-aromatic hydrocarbons, such as in aromatic fractions derived from coke oven light oil, they also cannot be separated from their non-aromatic contaminants by ordinary distillation or by azeotropic distillation with polar azeotropic agents. Non-aromatic hydrocarbons and an aromatic hydrocarbon with a closely related boiling point form azeotropes with a common non-selective azeotropic agent which do not differ much in their azeotropic boiling temperatures. Hence the normal boiling point of the aromatic hydrocarbon cannot be substantially lower than the normal boiling points of the non-aromatic hydrocarbon contaminants in a given mixture without the non-aromatic hydrocarbon azeotropes having substantially the same azeotropic boiling temperature as the aromatic hydrocarbon azeotrope of the given non-selective azeotropic agent. Where using selective azeotropic agents, the non-aromatic hydrocarbon contaminants cannot normally boil substantially above the aromatic hydrocarbon without either the non-aromatic hydrocarbon azeotropes failing to be formed or without their compositions being exceedingly rich in the azeotropic agent.

I have discovered that aromatic hydrocarbons which contain significantly higher-boiling non-aromatic hydrocarbon contaminants which form non-ideal or azeotropic systems with the aromatic component or components may be purified by a method which consists first in distilling the impure aromatic hydrocarbon so contaminated with a sufficient amount of a selected non-aromatic hydrocarbon or mixture of non-aromatic hydrocarbons. In that way, I can obtain aromatic-non-aromatic azeotropes or azeotropic mixtures which do not contain non-aromatic hydrocarbons which normally boil significantly above the normal boiling temperature of the aromatic hydrocarbon component, and after separation from the non-aromatic hydrocarbon contaminants, these azeotropes may be distilled with a polar azeotropic agent to obtain the pure aromatic hydrocarbons.

The primary object of the present invention is to provide a method of purifying aromatic hydrocarbons by a two-stage azeotropic distillation in which the first distillation will remove the aromatic hydrocarbons overhead from the non-aromatic hydrocarbons which boil significantly above

the boiling point of the aromatic hydrocarbons.

Another object of the invention is to provide a method of purifying aromatic hydrocarbons by a two-stage azeotropic distillation in which the aromatic hydrocarbons are separated from high-boiling point non-aromatic hydrocarbons by distillation with a non-aromatic hydrocarbon azeotropic agent and the non-aromatic hydrocarbon azeotropic agent used in the first distillation is separated from the aromatic hydrocarbon in a second distillation.

I prefer to use in the first distillation, a non-aromatic hydrocarbon which will form an azeotrope with the aromatic hydrocarbon in which the non-aromatic hydrocarbon comprises from forty percent to sixty percent of the azeotrope, although non-aromatic hydrocarbons which form azeotropes with the aromatic hydrocarbon in which the non-aromatic hydrocarbon comprises as little as five per cent or as much as eighty per cent of the azeotrope, or mixtures of non-aromatic hydrocarbons which form azeotropes with the aromatic hydrocarbon in which the non-aromatic hydrocarbons comprise from five per cent to eighty per cent of the azeotropes may be used effectively.

An essential condition in the first distillation is to employ from one per cent to five per cent more of the added non-aromatic hydrocarbon or mixture of non-aromatic hydrocarbons than is required to form the azeotrope or azeotropes with all of the aromatic hydrocarbon. This excess of non-aromatic hydrocarbon ensures that substantially all of the aromatic hydrocarbon will be recovered and further it ensures that substantially none of the normally higher-boiling contaminants originally associated with the aromatic hydrocarbon will contaminate the overhead distillate.

In the preferred form of my invention for separating pure benzene from mixtures that contain non-aromatic hydrocarbons which normally boil substantially above the normal boiling point of benzene but which when present with benzene form azeotropes or non-ideal systems with benzene, I first distill the mixture with an amount of an added non-aromatic hydrocarbon, which forms an azeotrope with benzene in which the non-aromatic component comprises from forty per cent to sixty per cent of the azeotrope which is taken overhead by distillation. Then I distill the product of this distillation with an amount of added polar azeotropic agent which is sufficient so that the non-aromatic hydrocarbon, which was added in the first distillation and which now must be separated in a second distillation, is taken overhead by distillation, leaving pure benzene as the bottom product. The polar azeotropic agent forms an azeotrope with the non-aromatic hydrocarbon or hydrocarbons added in the first distillation, and the polar agent azeotrope of the non-aromatic hydrocarbon or hydrocarbons may be effectively resolved, for example by water extraction and decantation, so that both the non-aromatic hydrocarbon or non-aromatic hydrocarbon mixture used in the first distillation and the polar azeotropic agent used in the second distillation may be recovered and recycled to the process.

Cyclohexane forms an azeotrope with benzene which contains approximately equal parts of benzene and cyclohexane. The boiling point of cyclohexane is only sligthly higher than the boiling point of benzene and the cyclohexane-benzene

azeotrope is a distillate which can be readily separated from the normally higher-boiling contaminants associated with coke oven benzene. Further, mixtures of cyclohexane and benzene, such as the azeotropic distillate formed by cyclohexane and benzene, may be readily separated by azeotropic distillation with acetone or with methanol. Still further, the cyclohexane-acetone azeotrope or the cyclohexane-methanol azeotrope may be readily separated by extraction and decantation with water. Therefore, for the purification of coke oven benzene, cyclohexane is a suitable hydrocarbon to be used in the first distillation and either acetone or methanol are suitable polar azeotropic agents to be used in the second distillation. I have found, however, that any non-aromatic hydrocarbon or any mixture of non-aromatic hydrocarbons boiling above 68.° C. and below 90.5° C. can be used effectively in the first distillation of the impure benzene. The hydrocarbons boiling in this range include n-hexane, methylcyclopentane, 2,2-dimethylpentane, 2,4-dimethylpentane, cyclohexane, 2,2,3-trimethylbutane, 3,3-dimethylpentane and 2-methylhexane. Further, I have found that other aliphatic alcohols, such as ethanol and isopropanol, and another ketone, methyl ethyl ketone, may be used effectively in the second distillation.

Cyclohexane generally is not found in appreciable amounts in coke oven benzene but is present in petroleum distillates, in admixture with other non-aromatic hydrocarbons, and is produced as a hydrogenation product of benzene.

A further object of the invention is to provide a two-stage distillation in which aromatic hydrocarbons are separated in the first distillation by a non-aromatic hydrocarbon or mixture of non-aromatic hydrocarbons which form azeotropes with the aromatic hydrocarbons in substantially equal parts, and thereafter separating the aromatic hydrocarbons from the non-aromatic hydrocarbon azeotropic agent by distillation with a polar azeotropic agent which forms an azeotrope with the non-aromatic hydrocarbon so that the agent may be extracted from the non-aromatic hydrocarbons with water and decantation.

Another object of the invention is to provide a method of purifying aromatic hydrocarbons by a two-stage azeotropic distillation in which the first stage azeotropic agent will form an azeotrope with the aromatic hydrocarbon which is composed of approximately equal parts of the desired aromatic hydrocarbon and the non-aromatic hydrocarbon to permit the desired hydrocarbon to be readily separated from the non-aromatic hydrocarbon in a second distillation.

A further object of the invention is to provide a distillation process for the separation of non-aromatic hydrocarbon contaminants which boil above the boiling point of the desired aromatic hydrocarbon with an azeotropic agent which forms an azeotrope with the desired aromatic hydrocarbon and using the azeotropic agent in sufficient excess to ensure that the high boiling contaminants will not distill overhead with the desired aromatic hydrocarbon.

Another object of the invention is to provide a method of separating non-aromatic hydrocarbons having a boiling point higher than benzene from benzene by a two-stage distillation using cyclohexane to azeotrope with the benzene and carry the benzene-cyclohexane overhead in the first distillation and separating the benzene from the cyclohexane by azeotropic distillation with a polar azeotrope in the second distillation.

For the purification of toluene, ethylbenzene, orthoxylene, metaxylene, paraxylene, naphthalene and for other aromatic hydrocarbons, I prefer to use a mixture of non-aromatic hydrocarbons derived from petroleum as the azeotropic agent for the first distillation. For example, in the purification of toluene, I prefer to employ in the first distillation a petroleum distillate which contains paraffins and naphthenes and which has a boiling point range of from approximately 105° C. to approximately 112° C. Pure toluene may be obtained from the mixture of toluene azeotropes obtained in the first distillation by azeotropic distillation with polar azeotropic agents, for example, methyl ethyl ketone or methyl cyanide. As a general rule, I prefer that the mixture of non-aromatic hydrocarbons used as azeotropic agent in the first distillation have a boiling range of from not more than 5° C. below to not more than 5° C. above the boiling temperature of the aromatic hydrocarbon which is to be purified, although mixtures having somewhat wider boiling ranges, for example, from 10° C. below to 5° C. above the boiling point of the aromatic hydrocarbon to be purified may be used effectively.

A still further object of the invention is to provide a two-stage distillation process for purifying aromatic hydrocarbons having a boiling point higher than the boiling point of the aromatic hydrocarbon by an azeotropic distillation in the first stage using a mixture of non-aromatic hydrocarbons having a boiling point not more than 5° above or 5° below the boiling point of the desired aromatic hydrocarbon as the azeotropic agent for the first distillation and using a polar azeotropic agent for the second distillation.

With these and other objects and features in view the invention consists in the improved method of purifying aromatic hydrocarbons as hereinafter described and particularly defined in the claims.

The various features of the invention are illustrated in the accompanying drawing in which

The figure is a diagrammatic flow diagram of an apparatus in which the preferred method of invention may be carried out while using a non-aromatic hydrocarbon azeotropic agent for the first distillation, a polar azeotropic agent for the second distillation, and water for separating the aromatic hydrocarbon and non-aromatic azeotropic agent from the distillation products.

The preferred form of the invention will be described more particularly with reference to the purification of coke oven benzene although the process is well adapted to the purification of other aromatic hydrocarbons, such as toluene, ethyl benzene, orthoxylene, metaxylene, paraxylene, and naphthalene.

Referring to the figure, impure benzene is taken from a storage tank 10 and passed through line 12 into the mid-portion of a distilling column 14. To the impure benzene entering the column is added cyclohexane from storage tank 16 through line 18. In the distillation in column 14 a benzene-cyclohexane azeotrope is taken overhead through a line 20 and introduced into the mid-portion of a second distilling column 22. The overhead distillate from column 14 contains substantially all of the benzene introduced into the column, and the impurities in the benzene are removed from the base of the column through line 24, with a very small amount of cyclohexane therein. Cyclohexane containing a much smaller

amount of benzene that is present in the cyclohexane-benzene azeotrope is taken as a side cut from the lower part of column 14 through line 27 and returned to feed line 12. The side cut which is rich in cyclohexane ensures that no non-aromatic hydrocarbons normally boiling significantly higher than cyclohexane will contaminate the cyclohexane-benzene azeotrope removed from column 14 through line 20, and that no benzene will be lost through line 24. To the benzene-cyclohexane azeotrope is introduced through a line 26, a polar azeotropic agent such as methanol which forms an azeotrope with the cyclohexane as well as the benzene, or a polar azeotropic agent such as acetone which forms an azeotrope with the cyclohexane but not with the benzene. The cyclohexane azeotrope of the polar azeotropic agent is taken overhead from column 22 through line 28 and the benzene along with any excess of polar azeotropic agent over that required to form the cyclohexane azeotrope is removed from the column 22 through a line 30. The cyclohexane azeotrope of the polar azeotropic agent is passed through line 28 into the mid-portion of a decanting separator 32 and water is introduced into the separator through a line 34. A sufficient amount of water is introduced into the separator 32 to separate the cyclohexane from the polar azeotropic agent, the cyclohexane being the supernatant liquid and passes through a line 35 back to the storage tank 16 for recycling through the column 14. If desired cyclohexane may be returned directly to the line 12 by line 37 which is connected between lines 12 and 35. The solution of the polar azeotropic agent and water flows out of the bottom of the decanter 32 through a line 36 and is introduced into the mid-portion of a distilling column 38. The polar azeotropic agent-benzene mixture flows from the bottom of column 22 through the line 30 into the mid-portion of a decanting separator 40. Water is introduced into the separator 40 through a line 42 and the water and polar azeotropic agent are separated by decantation in the separator 40. The benzene is the supernatant liquid which passes through a dehydrator 43 and is recovered as pure benzene. The water-polar azeotropic agent mixture flows from the bottom of the separator 40 through a line 44 into the line 36 where it, along with the water-polar azeotropic agent mixture flowing from the bottom of separator 32 is passed into the column 38. The mixture of water and the polar azeotropic agent is distilled in the column 38 to separate the polar azeotropic agent from water. The polar azeotropic agent passes overhead through a line 46 back to the line 20 and is recirculated through the second distillation column 22. Water flows from the bottom of column 38 through a line 48 to a water reservoir 50. The reservoir 50 supplies water that passes through the line 34 and 42 to the separators or decanters 32 and 40. If desired, water may be introduced into the reservoir 50 through a line 52.

In the continuous process illustrated in the drawing, azeotropic agents, such as cyclohexane and methanol, and water, are added to the mixture being continuously distilled and these agents are then recovered and returned to their respective stages in the process. It is to be understood that a portion of these agents will be lost in the operation and, therefore, it is necessary to add to the various stages the agents to maintain the desired volume of agents for carrying on the separation operations.

The preferred form of the invention has been described with perticular reference to the purification of benzene where using cyclohexane as the azeotropic agent in the first distillation and methanol or acetone as the azeotropic agent in the second distillation. It is to be understood, that other non-aromatic hydrocarbons such as 2,4-dimethylpentane, 2,2-dimethylpentane and 2,2,3-trimethylbutane or mixtures of non-aromatic hydrocarbons having a boiling point range from approximately 75° C. to approximately 90° C. may be used in the first distillation for the purification of benzene, and that other azeotropic agents which are polar and water-soluble, for example ethanol or isopropanol may be used in the second distillation. Further, it is to be understood that other aromatic hydrocarbons, for example, toluene, ethylbenzene, orthoxylene, metaxylene, paraxylene and naphthalene may be purified by the method diagrammatically illustrated in the drawing but other appropriate non-aromatic hydrocarbons are to be used in the first distillation and appropriate polar azeotropic agents are to be used to the second distillation. For example, in the purification of toluene, 2,2,3-trimethylpentane or 3,3-dimethylhexane, or a mixture of non-aromatic hydrocarbons with a boiling point range of from approximately 105° C. to 112° C. may be used in the first distillation, and methyl ethyl ketone or methanol may be used in the second distillation. For other aromatic hydrocarbons, mixtures of non-aromatic hydrocarbons with a boiling point range of not more than 10° C. below and not more than 5° C. above the boiling point of the aromatic hydrocarbon or any single non-aromatic hydrocarbon boiling within that range may be used in the first distillation, and any polar and water-soluble azeotropic agent which will form a minimum-boiling azeotrope with the agent added in the first distillation may be used in the second distillation.

The preferred form of the invention having been thus described, what is claimed as new is:

I claim:

1. A method for the purification of distilled coke-oven benzene which contains a small amount of non-aromatic hydrocarbon impurities higher boiling than benzene and substantially inseparable therefrom by simple distillation, which comprises distilling in a fractional distillation zone a mixture of said benzene and added non-aromatic hydrocarbon material, said hydrocarbon material boiling normally between 75° and 85° C. and being present in said mixture in an amount in excess of that required to form an azeotrope with said benzene, removing from said distillation zone the azeotrope of benzene and said hydrocarbon material as an overhead fraction, removing at least a portion of the excess hydrocarbon material and said high boiling impurities from said distillation zone as a distillation residue, separating benzene and said hydrocarbon material by distillation with a polar azeotropic agent in a second fractional distillation zone and recovering purified benzene freed of high boiling non-aromatic impurities from a fraction from said second distillation zone.

2. The method of claim 1 in which the polar azeotropic agent is acetone.

3. The method of claim 1 in which the polar azeotropic agent is methanol.

4. A method for the purification of distilled coke-oven benzene which contains a small amount of non-aromatic hydrocarbon impurities

higher boiling than benzene and substantially inseparable therefrom by simple distillation, which comprises distilling in a fractional distillation zone a mixture of said benzene and added petroleum distillate boiling normally between 75° and 85° C., said petroleum distillate being present in said mixture and containing paraffins and naphthenes in an amount in excess of that required to form azeotropes between the paraffins and naphthenes and the benzene in said mixture, removing from said distillation zone said azeotropes as an overhead fraction, removing at least a portion of the excess paraffins and naphthenes and said high boiling impurities from said distillation zone as a distillation residue, separating benzene and said paraffins and naphthenes by distillation with a polar azeotropic agent in a second fractional distillation zone, and recovering purified benzene freed of high boiling non-aromatic impurities from a fraction from said second distillation zone.

5. A method for the purification of distilled coke-oven benzene which contains a small amount of non-aromatic hydrocarbon impurities higher boiling than benzene and substantially inseparable therefrom by simple distillation, which comprises distilling in a fractional distillation zone a mixture of said benzene and at least one added hydrocarbon selected from the group consisting of 2,2-dimethylpentane, 2,4-dimethylpentane, cyclohexane, and 2,2,3-trimethylbutane, said hydrocarbon being present in said mixture in an amount in excess of that required to form an azeotrope with said benzene, removing from said distillation zone the azeotrope of benzene and said hydrocarbon as an overhead fraction, removing at least a portion of the excess hydrocarbon and said high boiling impurities from said distillation zone as a distillation residue, separating benzene and said hydrocarbon by distillation with a polar azeotropic agent in a second fractional distillation zone, and recovering purified benzene freed of high boiling non-aromatic impurities from a fraction from said second distillation zone.

6. A method for the purification of distilled coke-oven benzene which contains a small amount of non-aromatic hydrocarbon impurities higher boiling than benzene and substantially inseparable therefrom by simple distillation, which comprises distilling in a fractional distillation zone a mixture of said benzene and added cyclohexane, the cyclohexane being present in said mixture in an amount in excess of that required to form an azeotrope with said benzene, removing from said distillation zone the azeotrope of benzene and cyclohexane as an overhead fraction, removing at least a portion of the excess cyclohexane and said high boiling impurities from said distillation zone as a distillation residue, separating benzene and cyclohexane in said azeotrope by distillation with a polar azeotropic agent in a second fractional distillation zone, and recovering purified benzene freed of high boiling non-aromatic impurities from a fraction from said second distillation zone.

7. A method for the purification of distilled coke-oven benzene which contains a small amount of non-aromatic hydrocarbon impurities higher boiling than said benzene and substantially inseparable therefrom by simple distillation, which comprises distilling in a fractional distillation zone a mixture of said benzene and added cyclohexane, said cyclohexane being present in said mixture in an amount in excess of that required to form an azeotrope with benzene, removing from said distillation zone the azeotrope of benzene and cyclohexane as an overhead fraction, removing at least a portion of the excess cyclohexane and said high boiling impurities from said distillation zone as a distillation residue, separating benzene and cyclohexane by distillation with acetone in a second fractional distillation zone, and recovering purified benzene freed of high boiling non-aromatic impurities from the bottoms fraction from said second distillation zone.

JOHN R. ANDERSON.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,212,810	Field	Aug. 27, 1940
2,356,240	Hamlin	Aug. 22, 1944
2,440,414	Oblad	Apr. 27, 1948
2,477,303	Lake	July 26, 1949