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RECOVERY OF INDOLES

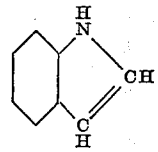
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This invention relates to a continuous method for separating indole and its homologs from other substances, particularly from the industrial products in which they occur. More specifically, the invention relates to a continuous method whereby the indoles in industrial products or similar mixtures are first concentrated by use of a selective solvent and then removed from a concentrated mixture by azeotropic distillation with paraffin hydrocarbons such as kerosene.

Indole is used in the production of perfumes and in the synthesis of tryptophane, an essential amino acid. More widespread industrial use would be likely except for its scarcity and high cost. It occurs naturally in organic sources, such as oil of jasmine, clove oil and the products of putrefaction of amino acids. The major commercial source at present is the coke industry, where indole is recovered from coal tar oil. A new source of supply, however, is now being found with the commercial development of coal hydrogenation processes. Indole is found in the so-called neutral light oil fraction derived from the coal by hydrogenation. The neutral light oil fraction is composed principally of aromatic and paraffinic and cycloparaffinic hydrocarbons; the tar acids and nitrogen bases have been separated from it. Although indole, which has the formula:



is a nitrogen containing compound similar in structure to the nitrogen bases, it is neutral to weakly acid in nature and resembles the aromatic hydrocarbons in many of its properties, which accounts for its being found in the neutral light oil fraction rather than with the nitrogen bases as might be expected.

In the past indole has been recovered from the appropriate fraction of coal tar by taking advantage of the weakly acidic properties of its N—H group. This involves precipitating the potassium or sodium salt of indole, separating the salt from the oil mixture, and then hydrolyzing the salt to give indole and sodium or potassium hydroxide. A number of German patents, including Nos. 223,304, 238,138 and 454,696, disclose this method, which is unsatisfactory in that multiple treatment of the indole fraction is necessary to obtain indoles of sufficiently high quality, and each step must be carried out batchwise.

Working with the light oil product of coal hydrogenation, we have found that indoles can be recovered from indole-bearing hydrocarbon mixtures, such as the light oil, by removing the indoles as azeotropes with suitable boiling range fractions of paraffin hydrocarbon such as kerosene. We have further found, as will be seen, that it is preferable, though not essential, to first concentrate the indoles as a solution in the aromatic hydrocarbons.

To accomplish this concentration, we have found that an alkylene carbonate may be used effectively to extract the indoles and aromatic hydrocarbons from the indole-bearing oil prior to the azeotropic distillation. Carbonate extraction could also be used to concentrate the indoles prior to employing a final separation process other than the formation of azeotropes.

We have found that indoles form azeotropes with a great number of hydrocarbons, both aromatic and aliphatic, having a wide range of boiling points. Hydrocarbons which form azeotropes with indole include n-tridecane, n-tetradecane, decahydroacenaphthene, 1-methylnaphthalene, tetrahydroacenaphthene, hexahydrofluorene, 6-methyltetralin, and paraffin cuts from the neutral light oil, and distillation tests were made with mixtures of these and indole. Azeotrope formation was observed in every case, with the greatest reduction of the azeotrope boiling point below the indole boiling point being obtained with the paraffins. We have found that indole can form azeotropes with parffin hydrocarbons boiling above 180° C. The vast number of compounds with which indoles will distill as azeotropes, and the wide range of the boiling points of these compounds cause indoles to be distributed widely throughout the boiling point range of the neutral light oil, as well as the other oils in which they occur in most cases, and complicates the problem of their removal by requiring treatment of a large quantity of oil to obtain a relatively small amount of indoles. Indole, with a normal boiling point of 254° C., has been found in relatively small amounts in neutral light oil boiling between 210° and 245° C., while methylindoles, with normal boiling points between 266° and 273° C., are found in neutral light oils having boiling points between 245° and 260° C., and the higher homologs of indole are found in the higher boiling point ranges of the neutral light oil. It is quite possible, however, that in other types of neutral light oil, the indoles might be found over a broader or narrower range of boiling points, and the invention is not restricted in any way in this regard.

We have discovered a method whereby the tendencies of indoles to form azeotropes can be utilized for their separation from admixture with hydrocarbons. Because azeotropes of the paraffin hydrocarbons with indoles boil at sharply reduced temperatures as compared to the indoles themselves, it has been found that paraffin hydrocarbons can be used most efficiently to remove the indole. First, however, it is desirable to concentrate the indole and its homologs in a predominantly aromatic fraction of the neutral light oil. Aromatic hydrocarbons do not decrease the boiling point of azeotropes to nearly so great a degree as do the paraffins, and the presence of aromatic hydrocarbons with the indoles does not interfere with their azeotropic separation with parffins. Substantially all the aliphatic hydrocarbons should first be removed for best results so that the mixture with the indoles is essentially aromatic. Moderate amounts of partially hydrogenated polynuclear compounds, such as the alkyltetralins, may be tolerated, but if the degree of hydrogenation of the polynuclear hydrocarbon is too great, it behaves substantially as an aliphatic and hence is undesirable.

In the practice of our invention, therefore, the indoles which are present in indole-bearing oil, which in the case of neutral light oil from coal hydrogenation includes all liquid product boiling from 100° C. to 300° C., can first be concentrated into a much smaller fraction containing only indoles and aromatic hydrocarbons. This concentration can be accomplished by the use of a selective solvent which will extract only indoles and aromatic hydrocarbons from the indole-bearing oil. We have found that 1,2-alkylene carbonates containing from three to four car-

bon atoms are particularly useful, though other alkylene carbonates may also be effective. Trimethylene carbonate can also be used. The volume ratio of carbonate to indole-bearing oil may be between 1 to 20 and 20 to 1. Atmospheric pressure and a temperature up to about 10 degrees centrigrade above the freezing point of ethylene carbonate, or room temperature in the case of propylene carbonate, are preferred, but temperatures up to the temperature where appreciable decomposition of the carbonate occurs are operable. The selectivity and capacity of the carbonate solvent may be changed if desired by the addition to the carbonate of minor amounts of other compounds, for example, water.

The carbonate extraction may be run as a single stage batch extraction but preferably a multistage continuous extraction is employed. Mechanical aids such as mixers, packing, baffles and the like may be used in the extraction apparatus. This carbonate extraction removes as extract substantially all of the indoles present together with the more highly aromatic hydrocarbons such as naphthalene. Thus this extract represents a more concentrated indole mixture than the indole-bearing oil such as coal hydrogenation light oil from which it is derived. After this extraction is complete, the alkylene carbonate solvent is separated from the mixture by one of several methods, including freezing out and water-washing. Multiple washings with water have proved satisfactory.

While indoles can be recovered directly from an indole-bearing oil, and such removal is within the scope of the invention, when the indoles have first been concentrated in an aromatic fraction, they can then be more readily removed by azeotropic distillation with a paraffin hydrocarbon mixture for the reasons given earlier. A pure paraffin or mixture of pure paraffins having a suitable boiling point can be used, but we have found kerosene fractions, which are more desirable from an economic viewpoint, to be entirely suitable in the invention. The choice of the particular boiling range of the kerosene will of course vary with the boiling range of the particular aromatic fraction being treated. A kerosene fraction is used which boils over a range of four or five degrees, with the highest temperature of the kerosene fraction boiling range being about coincident with the lowest temperature of the boiling range of the aromatic fraction. If the indole is found to be concentrated in a narrower boiling range of components within the aromatic fraction, it will be more efficient to first remove by distillation that portion of the aromatic fraction boiling below and/or above the boiling range of that portion of the aromatic fraction which contains substantially all the indole. A suitable kerosene is then employed as before to remove the indole as an azeotrope.

The recovery of both indole and methylindoles necessitates one additional step. Indole ordinarily appears in the lower boiling portion of the aromatic fraction while methylindoles and the higher homologs of indole normally are found in the upper boiling portion, with little or no overlapping between the two. Thus, where it is desired to recover both indole and methylindole, it is necessary only to find by simple test the boiling point within the boiling range of the aromatic fraction below which boiling point the indole is found and above which boiling point the methylindoles are present. The aromatic fraction is then separated into two sub-fractions at this point, as by distillation, one sub-fraction below and one sub-fraction above the critical boiling point. The lower boiling sub-fraction is then treated exactly as above for the recovery of indole alone. The higher boiling sub-fraction is treated in analogous manner except, of course, that the kerosene fraction chosen has a higher boiling range, with the high temperature of this kerosene's four or five degree boiling range being about coincident with the critical temperature at which the two sub-fractions were separated. This critical temperature has become the lowest temperature of the boiling range of the upper sub-fraction of the aromatic fraction, that is, the sub-fraction containing the methylindoles. In our work with coal hydrogenation neutral light oil, we have found this critical temperature to be about 252° C. when substantially all of the aliphatic hydrocarbons have been removed, with the major portion of the indole being present in that sub-fraction of the aromatic fraction boiling between 236° and 252° C. The majority of the methylindoles are present in that sub-fraction of the aromatic fraction boiling between 252° and 271° C. The invention, of course, is not limited to neutral light oils having this particular distribution of indole and would be just as useful with different types of hydrocarbons mixtures containing indoles.

The final step in the method of our invention is the removal of the indole from the kerosene azeotropes. Unless the percentage of indole is extremely low, indole itself will usually precipitate out of the azeotrope when it is cooled and can then be recovered by filtering. Methylindoles and higher homologs are more apt to separate as an oil rather than precipitate, particularly if a mixture of isomers is present. In this case the indole compound is separated from the azeotrope by extraction with a solvent which will extract the indoles from the azeotrope and be immiscible with the hydrocarbons. The indoles-solvent layer is removed and the indoles separated therefrom by distilling the solvent out. We have found methanol to be an excellent solvent for this purpose, though any compound having the desired properties would be suitable. The indole products in any case can be further purified by recrystallizing the crude product from water, aqueous methanol or a low-boiling paraffin.

By the continuous process of our invention, relatively pure indoles of high quality are produced in quantity from the industrial products in which they occur, such as neutral light oil from coal hydrogenation, coke oven oil or coal tar oil, or from any hydrocarbon mixture containing indoles. Indole-bearing oils also frequently contain phenolic compounds and nitrogen bases. Compounds of these types are not separated from the indoles by the process and must be removed by established techniques.

The following examples illustrate the various phases of the invention. Example I shows that an alkylene carbonate, in this case ethylene carbonate, will extract indoles and aromatic hydrocarbons from an indole-bearing oil, and thus concentrate the indoles in admixture with aromatic hydrocarbons. Examples II, III, and IV show that indoles may be removed by prolonged azeotroping directly from an indole-bearing oil regardless of its hydrocarbon composition although the yields are relatively low compared to the percentage of indole originally present. Example V shows the greater efficiency and better results of a combined method wherein the indoles present in an indoles-bearing oil are first concentrated into an indoles-aromatic hydrocarbon fraction by the use of an alkylene carbonate solvent, and then removed from this concentrated mixture by azeotroping with a paraffin hydrocarbon.

Example I

Coal hydrogenation neutral light oil having a nominal boiling point range of 100° to 260° C. and containing about one percent by weight of indoles and about one percent by weight of phenolic compounds was subjected to a plurality of single stage extractions with successive portions of ethylene carbonate. Each portion of carbonate amounted to five percent of the weight of the neutral light oil and was circulated through the oil for a period of 24 hours by means of a pump, after which it was withdrawn with its dissolved content of indoles and aromatic hydrocarbons, and this extract was then washed with water to remove the ethylene carbonate. Successive five percent by weight portions of ethylene carbonate were circulated through the neutral light oil and the ex-

tracts withdrawn and wished in this manner. The maximum content of indoles in any one sample of extracted oil was 13.2 percent by weight. Forty-one percent by weight of the contained indoles had been extracted when 3.7 percent by weight of the neutral light oil had been extracted, and 71 percent by weight of the contained indoles had been extracted when 8.6 percent by weight of the neutral light oil had been extracted. The concentration of the indoles in the extracts at this point was 8.3 percent by weight. All of the indoles originally present in the neutral light oil were removed when 25 percent by weight of the neutral light oil had been extracted with ethylene carbonate. The concentrated solutions of indoles in aromatic hydrocarbons thus obtained were suitable for azeotropic distillation according to the process of the invention.

Example II

Seventy-eight grams of neutral light oil produced by the hydrogenation of coal and having a boiling temperture range of 240° to 242° C. and containing 3.6 percent indole by weight by infra-red analysis, was distilled with 156 ml. of kerosene having a boiling temperature range of 210° to 220° C., using a column having about five theoretical plates. Nine cuts weighing 17 grams each and a residue were obtained. Analysis of typical cuts and the residue was as follows:

Cut Number	Boiling Temperature Range, ° C.	Weight Percent Indole
1	200–206	1.8
2	206–207	2.1
5	209–210	1.0
7	210–211	1.3
8	211–212	0.5
9	212–230	0.2
Residue	230 plus	0.1

Example III

One hundred grams of neutral light oil produced by the hydrogenation of coal and having a boiling temperature range of 240° to 242° C. and containing 2.6 percent indole by weight of infra-red analysis, was distilled with 200 ml. of kerosene having a boiling temperature range of 220° to 230° C., using a column having about thirty theoretical plates. Eleven cuts weighing 17 grams each and a residue were obtained. Solid indole was obtained from the first two cuts upon cooling the distillate to room temperature. Analysis of typical cuts was as follows:

Cut Number	Boiling Temperature Range, ° C.	Weight Percent Indole
1–2	213–217	5.2
3	217–218	0.9
5	219–221	0.1
11	230–235	0.0
Residue	235 plus	0.1

Example IV

Forty-seven ml. of neutral light oil produced by the hydrogenation of coal and having a boiling temperature range of 230° to 234° C. and containing 7.0 percent indole by infra-red analysis, was distilled with 122 ml. of kerosene having a boiling temperature range of 220° to 222° C., using a column having about 30 theoretical plates. Nineteen cuts of five ml. each were obtained and solid indole was obtained from the first five cuts upon

cooling the distillate to room temperature. Analysis of typical cuts was as follows:

Cut Number	Boiling Temperature Range, ° C.	Weight Percent Indole
1	217–218	6.9
3	218–218	9.3
5	218–219	6.3
8	220–220	2.7
11	222–222	0.5
17	222–222	0.02

Example V

A neutral light oil sample obtained by the hydrogenation of coal was extracted with ethylene carbonate to give an extract of aromatic hydrocarbons substantially free of paraffins and partially hydrogenated polynuclear compounds. The aromatic fraction thus extracted constituted 15 percent by weight of the neutral light oil sample and was found by test to contain 95 percent of the indoles originally present in the total neutral light oil sample. The ethylene carbonate extraction solvent was removed by water-washing and the aromatic fraction was then distilled into two sub-fractions. The first lower boiling subfraction had a boiling point range of approximately 236° to 252° C. and contained substantially all the indole present in the aromatic fraction. The second or higher boiling sub-fraction had a boiling point range of approximately 252° to 271° C. and contained substantially all the methylindoles present in the aromatic fraction.

The lower boiling sub-fraction was distilled as an azeotrope with kerosene having a boiling range of 233° to 237° C. In this manner 98 percent by weight of the indole present in the sub-fraction (6.9 percent by weight of the sub-fraction by test) was recovered in azeotropes boiling at temperatures between 219° and 233° C. The residue from the azeotropic distillation, boiling above 237° C., containing only 0.06 percent by weight of indole, showing excellent recovery of the indole from the sub-fraction. The amount of kerosene required to form an azeotrope with the indole was about one-half the weight of the sub-fraction. The indole was recovered from the kerosene by cooling and filtration and the crude product was purified by recrystallization from methanol.

The higher boiling sub-fraction was distilled as an azeotrope with an equal weight of kerosene having a boiling range of 247° to 252° C. In this manner 90 percent by weight of the methylindoles in the sub-fraction (10 percent by weight of the sub-fraction by test) was recovered in azeotropes boiling at temperatures between 240° and 252° C. The residue from the azeotropic distillation, boiling above 252° C., contained only 0.8 percent by weight of methylindoles, showing excellent recovery of the methylindoles from the sub-fraction. The kerosene-methylindoles azeotrope was cooled, whereupon the methylindoles separated out as an oil. They were recovered by extraction with methanol followed by stripping of the methanol.

We claim:

1. A process for recoviring indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with a lower alkylene carbonate; separating said extract to remove said carbonate and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon.

2. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with a lower alkylene carbonate; washing said extract with water to remove said carbonate

in water solution and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon by cooling and filtering.

3. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with a lower alkylene carbonate; washing said extract with water to remove said carbonate in water solution and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; employing a hydrocarbon-immiscible solvent to remove said indoles from said azeotrope as an extract of indoles; and distilling said extract of indoles to distill off said solvent and leave said indoles as product.

4. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with trimethylene carbonate; separating said extract to remove said carbonate and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon.

5. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with a 1,2-alkylene carbonate containing from 3 to 4 carbon atoms; separating said extract to remove said carbonate and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon.

6. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with ethylene carbonate; separating said extract to remove said carbonate and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon.

7. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with propylene carbonate; separating said extract to remove said carbonate and leave an indoles-containing residue; adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon.

8. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with a lower alkylene carbonate; separating said extract to remove said carbonate and leave an indoles-containing residue; adding a mixture of paraffin hydrocarbons having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbons; and recovering said indoles from said paraffin hydrocarbons.

9. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises extracting an indoles-containing extract from said indoles-bearing oil with a lower alkylene carbonate; separating said extract to remove said carbonate and leave an indoles-containing residue; adding kerosene having a boiling temperature above 180° C. to said residue to form a second mixture; distilling said second mixture to remove said indoles as minimum-boiling azeotropes with said kerosene; and recovering said indoles from said kerosene.

10. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said oil to form a mixture; distilling said mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon.

11. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said oil to form a mixture; distilling said mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; and recovering said indoles from said paraffin hydrocarbon by cooling and filtering.

12. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises adding at least one paraffin hydrocarbon having a boiling temperature above 180° C. to said oil to form a mixture; distilling said mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbon; employing a hydrocarbon-immiscible solvent to remove said indoles from said azeotrope as an extract of indoles; and distilling said extract of indoles to distill out said solvent and leave said indoles as product.

13. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises adding a mixture of paraffin hydrocarbons having a boiling temperature above 180° C. to said oil to form a mixture; distilling said mixture to remove said indoles as minimum-boiling azeotropes with said paraffin hydrocarbons; and recovering said indoles from said paraffin hydrocarbons.

14. A process for recovering indoles from an indoles-bearing oil containing aromatic hydrocarbons which comprises adding a kerosene fraction having a boiling temperature above 180° C. to said oil to form a mixture; distilling said mixture to remove said indoles as minimum-boiling azeotropes with said kerosene; and recovering said indoles from said kerosene.

15. A process for removing a mixture of indoles and aromatic hydrocarbons from an indoles-bearing oil which comprises employing a lower alkylene carbonate to extract said mixture from said oil and then separating the extract thus obtained to remove said carbonate and leave a residue of indoles and aromatic hydrocarbons.

16. A process for removing a mixture of indoles and aromatic hydrocarbons from an indoles-bearing oil which comprises employing a lower alkylene carbonate to extract said mixture from said oil and then washing the extract thus obtained with water to remove said carbonate in water solution and leave a residue of indoles and aromatic hydrocarbons.

17. A process for removing a mixture of indoles and aromatic hydrocarbons from an indoles-bearing oil which comprises employing a 1,2-alkylene carbonate containing from 3 to 4 carbon atoms to extract said mixture from said oil and then separating the thus obtained extract to

remove said carbonate and leave a residue of indoles and aromatic hydrocarbons.

18. A process for removing a mixture of indoles and aromatic hydrocarbons from an indoles-bearing oil which comprises employing ethylene carbonate to extract said mixture from said oil and then separating the thus obtained extract to remove said carbonate and leave a residue of indoles and aromatic hydrocarbons.

19. A process for removing a mixture of indoles and aromatic hydrocarbons from an indoles-bearing oil which comprises employing propylene carbonate to extract said mixture from said oil and then separating the thus obtained extract to remove said carbonate and leave a residue of indoles and aromatic hydrocarbons.

20. A process for recovering separately indole and homologs of indole from an indoles-bearing oil containing aromatic hydrocarbons which comprises separating said indoles-bearing oil into two sub-fractions, one containing substantially all the indole and one containing substantially all the indole homologs; adding to the indole-containing sub-fraction at least one paraffin hydrocarbon having a boiling temperature above 180° C. to form a mixture; distilling said mixture to remove said indole as minimum-boiling azeotropes with said paraffin hydrocarbon; recovering indole from said paraffin hydrocarbon; adding to the indole homologs-containing sub-fraction a second paraffin hydrocarbon having a boiling temperature above 180° C. to form a second mixture; distilling said second mixture to remove said indole homologs as minimum-boiling azeotropes with said second paraffin hydrocarbon; and recovering indole homologs from said second paraffin hydrocarbon.

References Cited in the file of this patent

UNITED STATES PATENTS

2,688,645	Baderstcher et al.	Sept. 7, 1954

FOREIGN PATENTS

812,079	Germany	Aug. 27, 1951
695,479	Great Britain	Aug. 12, 1953