

March 31, 1953

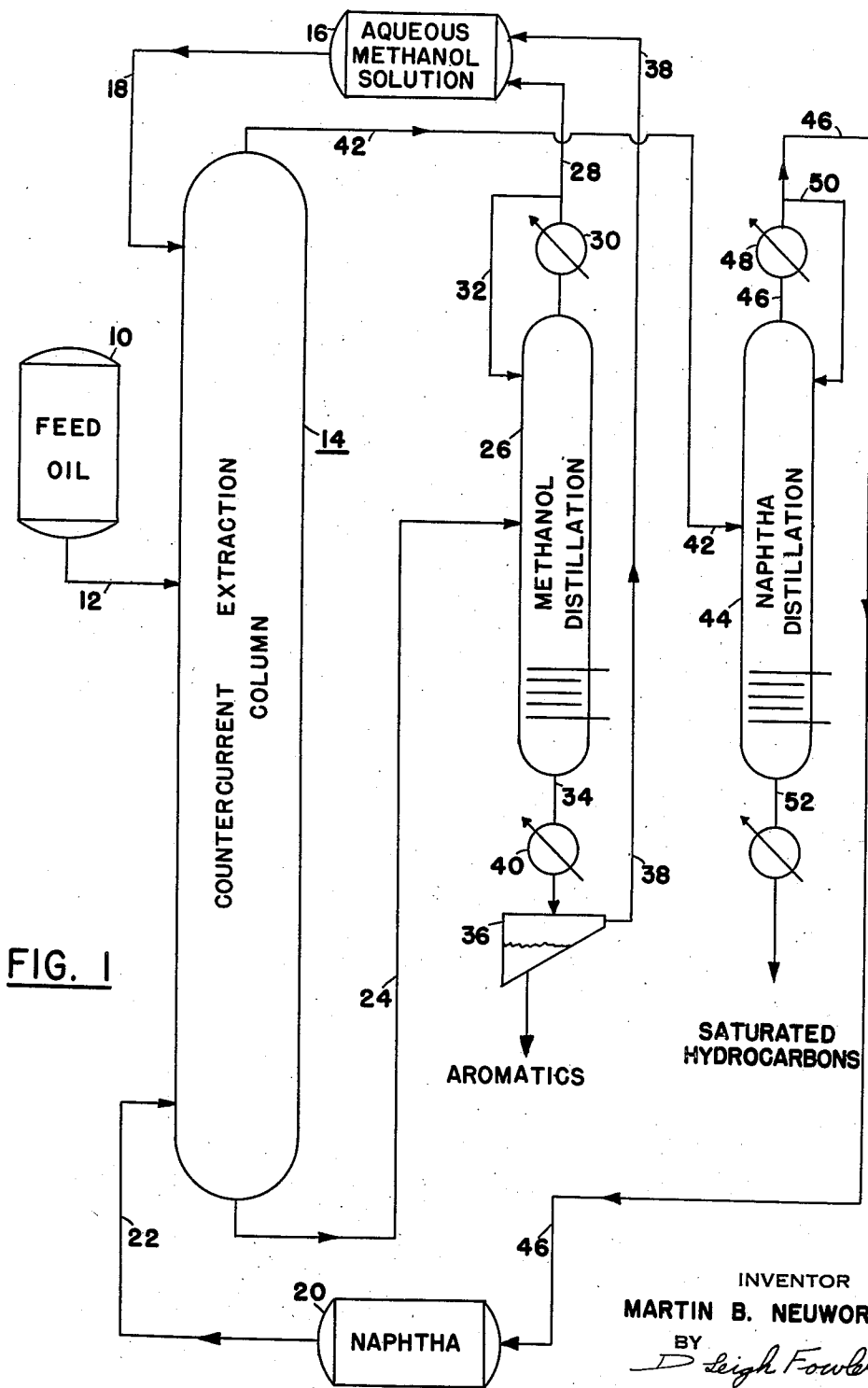
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2,633,448

DOUBLE SOLVENT EXTRACTION OF OILS

Filed Sept. 12, 1950

5 Sheets-Sheet 1



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DOUBLE SOLVENT EXTRACTION OF OILS

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5 Sheets-Sheet 2

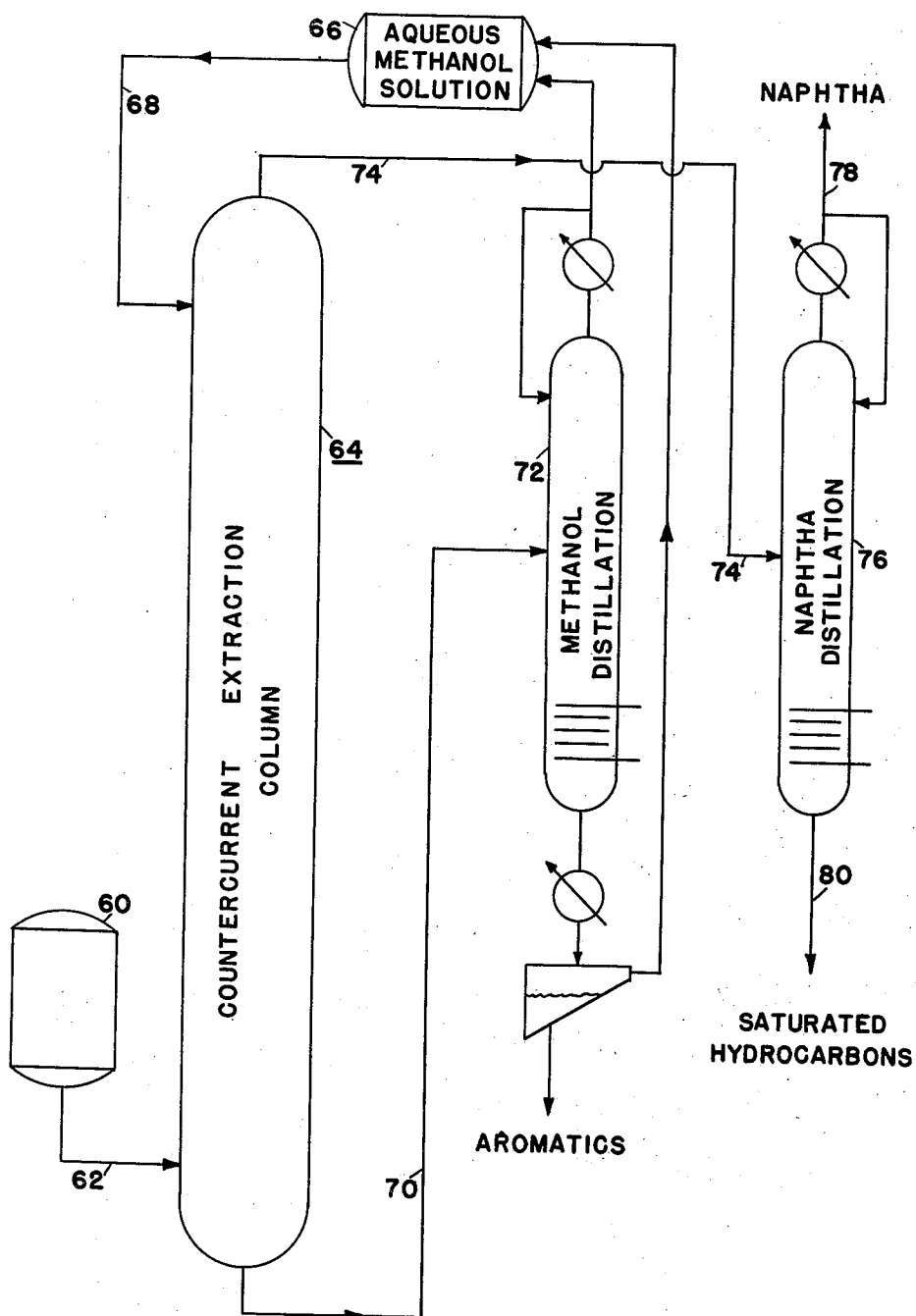


FIG. 2

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DOUBLE SOLVENT EXTRACTION OF OILS

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5 Sheets-Sheet 3

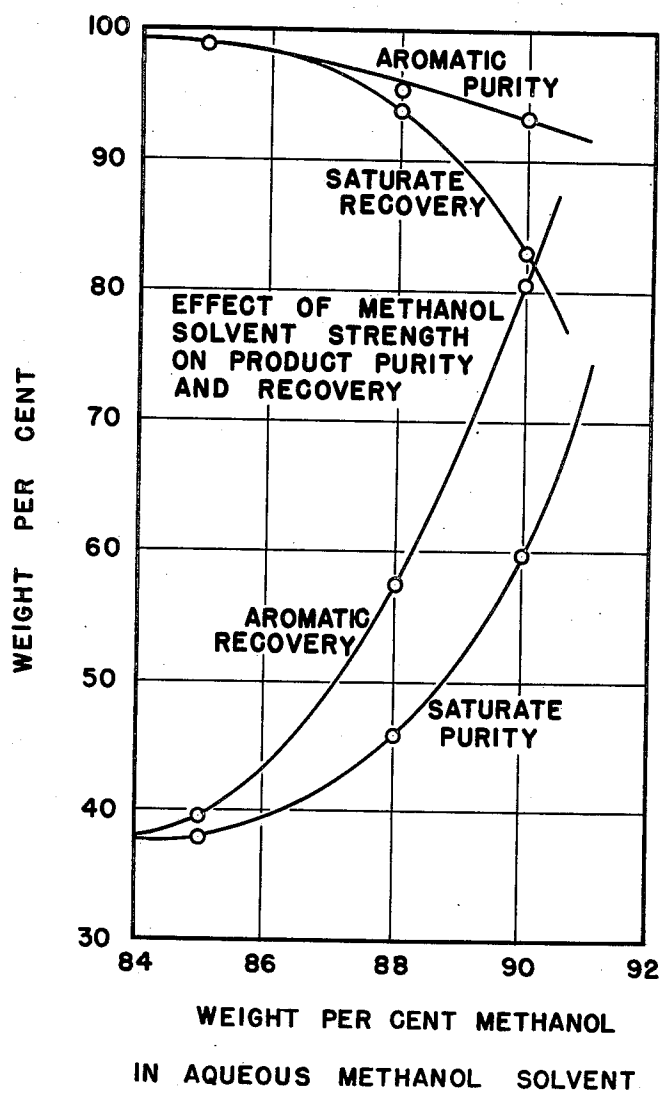


FIG. 3

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DOUBLE SOLVENT EXTRACTION OF OILS

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5 Sheets-Sheet 4

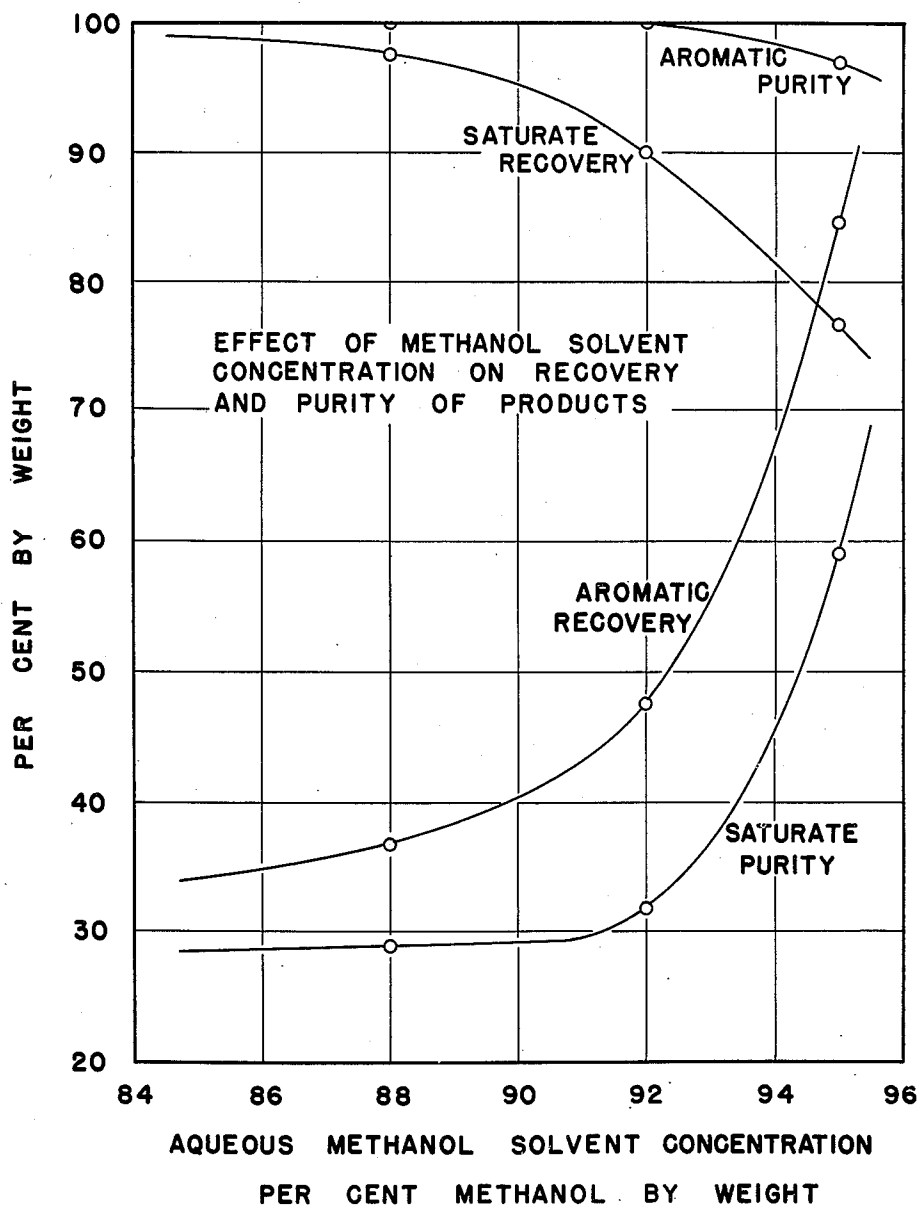


FIG. 4

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DOUBLE SOLVENT EXTRACTION OF OILS

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5 Sheets-Sheet 5

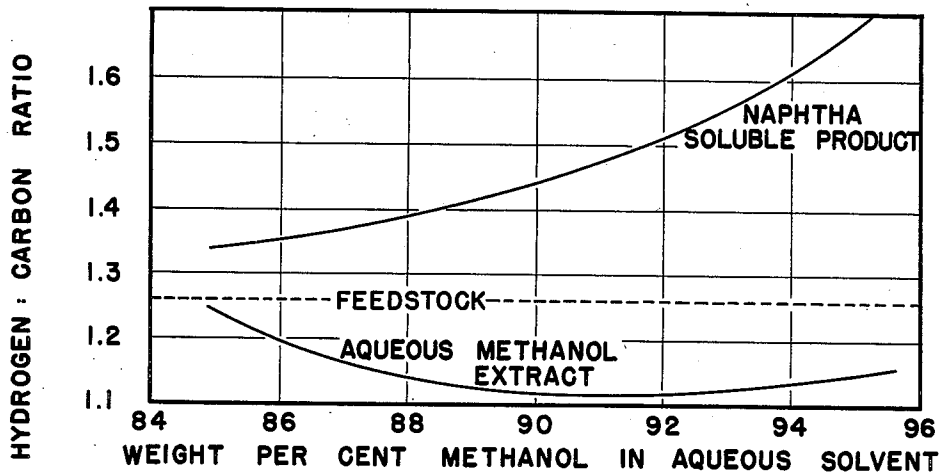


FIG. 5

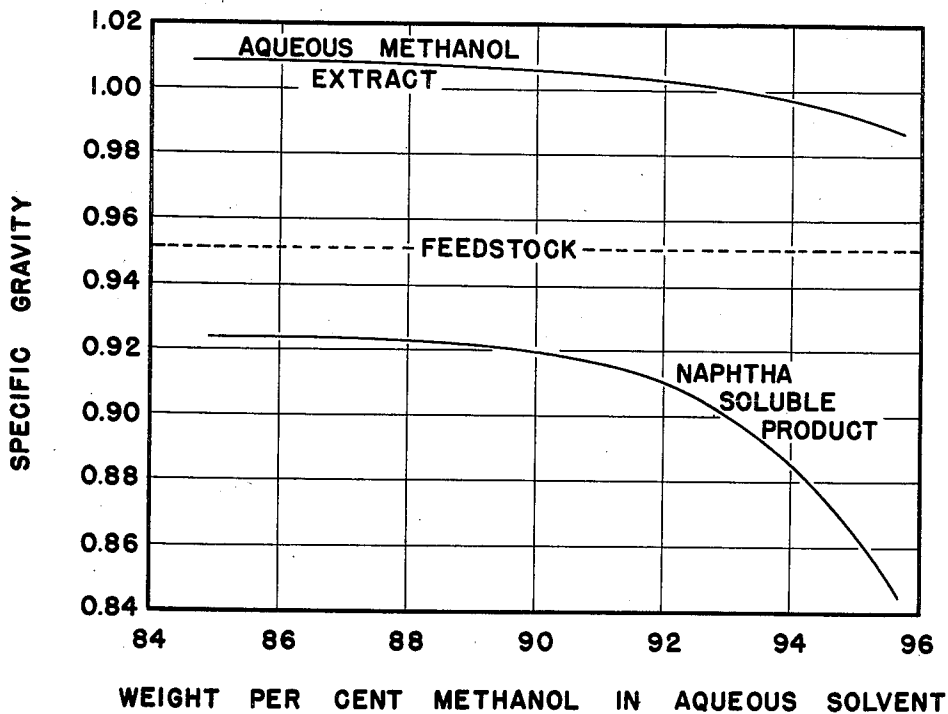


FIG. 6

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2,633,448

DOUBLE SOLVENT EXTRACTION OF OILS

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Application September 12, 1950, Serial No. 184,473

9 Claims. (Cl. 196—14.2)

1

The present invention relates to the separation of organic compounds and more particularly to a process for the double solvent extraction of aromatic constituents from a hydrocarbon distillate containing them in mixture with saturated hydrocarbons.

My new process is especially applicable to the neutral oil fraction of the tar produced by the low temperature carbonization of coal but also can be applied to any substantially tar acid free mixture containing aromatics and saturated hydrocarbons, such as coal hydrogenation oils, shale oil fractions, oils produced by synthesis processes and petroleum fractions.

The primary object of my invention is to remove the aromatic constituents from a mixture containing aromatic and saturated hydrocarbons and thereby to produce a high yield of substantially aromatic-free saturated hydrocarbons.

Another object of my invention is to separate and recover a high yield of substantially aromatic-free aromatic constituents from a mixture containing aromatic and saturated hydrocarbons.

A further object of the present invention is to produce a substantially aromatic-free saturated hydrocarbon fraction from a mixture containing aromatic and saturated hydrocarbons.

It is also an object of this invention to produce from a mixture containing aromatic and saturated hydrocarbons a saturated hydrocarbon fraction substantially free of aromatics, unsaturated hydrocarbons and corrosive contaminants.

In accordance with my invention, I have discovered that by contacting an oil containing both aromatic and saturated hydrocarbons but substantially free of tar acids with a low boiling naphtha solvent and an aqueous methanol solution, a separation of the aromatics from the saturated hydrocarbons can be effected.

The aromatic constituents of the oil along with certain unsaturated compounds which may be present are removed as extract in the aqueous methanol phase while the saturated hydrocarbons, such as naphthenes and paraffins, are recovered as solute in the naphtha phase.

More specifically, aqueous methanol is fed into the top portion of a vertical, double solvent, countercurrent extraction column. A low boiling naphtha solvent is fed into the bottom of the column. An oil boiling below 350° C. and containing aromatic and saturated hydrocarbons but substantially free of tar acids is fed into the column at a point located between the two solvent feed points and is contacted with the aqueous

2

methanol and naphtha solvents which pass through the column in countercurrent relation. Aromatics and certain unsaturated compounds from the feed are stripped by the aqueous methanol and removed from the bottom of the column dissolved in the aqueous methanol; the saturated hydrocarbons dissolve in the low boiling naphtha solvent and are removed from the top of the column.

The naphtha solvent employed in the process of this invention should have a low density i. e. below about 0.80, but preferably below 0.75, in order to insure rapid phase separation in the extraction column. Further, the naphtha must be a low boiling cut, boiling in the range of 60 to 130° C., but preferably 60 to 100° C., in order to simplify the subsequent separation of the naphtha from the recovered saturated hydrocarbons by distillation. A preferred solvent which possesses all these properties is the hexane cut of paraffinic naphtha.

The aqueous methanol solvent must contain from 75 to 96 per cent of methanol by weight. I have discovered that with aqueous methanol solutions containing 75 to 96 per cent by weight of methanol, a substantial recovery of aromatics from an oil containing them in mixture with saturated hydrocarbons can be effected. I have discovered further that the recovery of aromatics increases with increasing methanol concentration of the aqueous methanol solvent. Moreover, I have found that with aqueous methanol solvent weight concentrations in the range 86 to 96 per cent methanol, high yields of substantially pure aromatics can be effected. Expressing this alternatively, with aqueous methanol solvent weight concentrations within the range of 86 to 96 per cent methanol, my new process will yield substantially quantitative recovery of saturated hydrocarbons which are substantially free of aromatic constituents. In fact, where high yields of high purity aromatics are desired or where high yields of aromatic-free saturated hydrocarbons are desired, it is critical that the methanol concentration of the aqueous methanol solvent be maintained within the range of 86 to 96 per cent of methanol by weight.

However, it should be pointed out that there are applications where it is desirable to remove only a small portion of the aromatic constituents contained in a given feed oil. By the process of my invention, under the conditions hereinafter set forth, the purity or extent of recovery of aromatics—conversely, the purity or extent of recovery of saturated hydrocarbons—can be con-

trolled within wide ranges by regulating the concentration of the aqueous methanol solvent within the range of 75 to 96 weight per cent methanol. In general, within this range, as the methanol concentration is increased, the purity of the recovered aromatics decreases and the recovery of aromatics increases, whereas the purity of the saturated hydrocarbons increases and the recovery of saturates decreases with increasing methanol concentration.

Furthermore, in the particular application of the present invention to the neutral oil produced from low temperature coal carbonization tars, the critical range of aqueous methanol concentration for both high recovery and high purity is from 91 to 96 per cent by weight of methanol.

Since pure methanol is completely miscible with the naphtha solvent, it cannot be employed in this process because no phase separation results. At solvent concentrations approaching 100 per cent methanol, appreciable quantities of the naphtha solvent are dissolved in the aqueous methanol solution. Thus aqueous methanol concentrations exceeding 96 weight per cent of methanol are unsatisfactory.

The present process will operate effectively upon substantially tar acid free feed oils boiling below 350° C. and containing aromatic constituents and saturated hydrocarbons, such as naphthenes and paraffins. The lower boiling point of the feed stock must be sufficiently above the boiling range of the solvents so that subsequent separation of solvent from product can be accomplished by distillation, and preferably is about 160° C. Adverse effects on operability resulting from the increased viscosity which accompanies higher end point fractions also should be considered in determining the upper boiling point of the feed material. The presence of small amounts only, that is, up to about 5 per cent by weight of tar acids such as phenols, cresols, xylenols and higher alkylated phenols in the feed oil can be tolerated in the present process. Tar acids in excess of 5 per cent seriously affect the efficiency of extraction by my new process. Any tar acids in the feed oil will be recovered in the aqueous methanol extract.

My new process is particularly successful in recovering aromatics from an oil which is comprised predominantly of such aromatics in mixture with saturated hydrocarbons.

For a better understanding of my invention, its objects, advantages and methods of operation, reference should be had to the drawings in which:

Figure 1 is a diagrammatic illustration of apparatus adapted to carry out the process of the present invention;

Figure 2 is a diagrammatic illustration of apparatus adapted to carry out a modification of the process of the present invention;

Figure 3 is a graphical illustration of the effect of methanol concentration on recovery and purity of the products;

Figure 4 is a graphical illustration showing the effect of methanol concentration on recovery and purity of the products obtained from a neutral oil;

Figure 5 is a graphical illustration showing the effectiveness of separation by means of the present invention using the hydrogen-to-carbon ratio of the products as an indicative parameter;

Figure 6 is a graphical illustration showing the effectiveness of separation by means of the present invention using the specific gravity of the products as the indicative parameter.

Referring specifically to Figure 1, which shows schematically apparatus for carrying out the process of this invention, an oil containing aromatics and saturated hydrocarbons is fed continuously from a storage tank 10 through feed line 12 to a center feed, double solvent extraction column 14. This column may be of any convenient design capable of furnishing a sufficient number of theoretical stages to effect the desired separation. For example, a packed tower, a pierced plate column or a bubble plate column may be used, or a column containing alternate zones of turbulence and quiescence may be employed. An aqueous methanol solution containing from 75 to 96 per cent methanol by weight is fed continuously from a storage tank 15 through line 18 into the top portion of the extraction column 14. A low boiling naphtha fraction is fed continuously from storage tank 20 through line 22 into the bottom portion of the extraction tower 14.

For each volume of oil fed to the extraction column, the amount of naphtha is preferably less than about five volumes, depending, of course, on the composition of the feed oil as well as the desired product purity and recovery. However, in general, 2 to 5 volumes of naphtha are preferably employed for each volume of feed oil.

The quantity of aqueous methanol is preferably from about 1.5 to 20 volumes for each volume of oil, depending upon the composition of the oil and also upon the naphtha-to-oil ratio. In general, where little naphtha is employed, less aqueous methanol is required.

Naphtha, because of its lower density, rises through the column 14 countercurrently to the aqueous methanol and dissolves the feed oil. Aqueous methanol descends through the column, stripping out the aromatic constituents dissolved in the naphtha. While it is possible to operate with either the aqueous methanol phase or the naphtha phase as the continuous phase, it is preferred wherever practical to employ the naphtha phase as continuous in center feed operations. Accordingly, the aqueous methanol phase is dispersed in and falls as tiny globules downwardly through the continuous naphtha phase. The interface in the column is maintained at the bottom of the extraction tower, below the point at which the naphtha solvent is introduced.

Naphtha solvent, rising through the column, dissolves the feed oil and contacts successively aqueous methanol globules containing decreasing amounts of dissolved aromatics and ultimately, at the top of the column, contacts fresh aqueous methanol globules containing no aromatics. These contacts serve to transfer the aromatics from the naphtha phase to the aqueous methanol phase which has a greater affinity for the aromatics.

While the extraction process of the present invention is relatively independent of the temperature at which the column is operated, it is preferred to operate the extraction column within the range of 60 to 120° F. The increased viscosity of the oils at temperatures below this range introduces column operation difficulties while the altered solubility relationships at higher temperatures affect yields and purity of the products adversely. If necessary, the extraction column may be heated or cooled in any convenient manner.

Aqueous methanol solvent, containing dissolved aromatics leaves the bottom of extraction tower 14 through line 24 and is fed into a methanol

5

stripping column 26. Methanol passes overhead through line 28 and cooler 30 to the methanol storage tank 16. A portion of the cooled methanol may be returned to the column 26 through line 32 as reflux.

Water and aromatics are removed as bottoms from the column 26 through line 34 and collected in a phase separator 36. The aromatics, insoluble in water, are recovered by decantation. Water is removed from phase separator 36 and returned through line 38 to the methanol storage tank to make up recycle aqueous methanol. Since the very slight solubility of aromatics in water decreases with temperature, a cooler 40 may be inserted in line 34 to increase the recovery of aromatics from phase separator 36.

It should be noted that the recycle methanol contains some dissolved naphtha, the presence of which should be considered when preparing the aqueous methanol solution.

The paraffinic naphtha solvent, containing dissolved saturated hydrocarbons from the feed material, leaves the top of the extraction tower 14 through line 42 and enters a naphtha stripping column 44. The naphtha passes overhead through line 46 and cooler 48 and is returned to the naphtha storage tank 20 for recirculation. A portion of the cooled naphtha may be returned to column 44 as reflux through line 50. Saturated hydrocarbons are recovered as bottoms from column 44 through line 52, in which a cooler may be inserted if desired.

My new process also removes corrosive contaminants along with the aromatics which are present in the oil. Substantially all the nitrogen containing and oxygen containing compounds (especially any tar acids), as well as most of the sulfur containing compounds from the oil are recovered in the aqueous methanol phase. Thus the naphtha solvent product is extremely valuable as a diesel fuel for example, since the corrosive contaminants are removed by my process, and since the removal of the aromatics and certain unsaturated hydrocarbons has the effect of improving the cetane rating of the naphtha soluble product over that of the feed oil. The saturated hydrocarbons also have utility as special solvents, or as plasticizers, or as a cracking stock for thermal or catalytic cracking operations.

Heat and light stability of the naphtha soluble product is greatly improved over that of the feed material because of the elimination of the aromatic components.

The aromatics contained in the aqueous methanol product can be further separated and purified or can be used as a plasticizer, for example. Also, because of their high carbon-to-hydrogen ratio, the aromatics would be a satisfactory raw material for the production of carbon black.

A modification of the present invention is its operation in conjunction with the process disclosed in copending United States application Serial No. 184,474 entitled "Refining of Tar Acid Oil" and filed on even date herewith by E. Gorin and M. B. Neuworth, which describes a method for the separation of tar acid oils into a tar acid fraction and a neutral oil fraction by a double solvent extraction employing aqueous methanol (55 to 75 per cent methanol by weight) and a low boiling naphtha as solvents. The neutral oil fraction in the process of the copending application is extracted and recovered substantially free of tar acids, as solute in a low boiling naphtha solvent of the present invention. According to

6

the copending application, the neutral oil can be separated from the naphtha solvent by distillation. The neutral oil thus recovered, containing a mixture of aromatic and saturated hydrocarbons, can be treated by the process of the present invention and separated into an aromatic fraction and a saturated fraction by contacting it with aqueous methanol and naphtha solvents as already described in connection with Figure 1. However, since the neutral oil from the process of the copending application is produced in solution with the naphtha solvent common to both processes, it is possible to eliminate the intermediate distillation operation in whole or in part by performing the extraction operation of the present invention in an end feed, countercurrent extraction column. Accordingly, aqueous methanol (75 to 96 per cent methanol by weight) is fed to the top of the column and a solution of neutral oils in naphtha, produced in accordance with the above-mentioned copending application, is fed into the bottom of the column.

Figure 2 is a diagrammatic illustration of apparatus for carrying out the modification of the present invention in conjunction with the process described in the above-mentioned copending application.

Neutral oil dissolved in a low boiling naphtha solvent from a tar acid extraction process similar to that described in the copending application, is stored in a storage tank 60. It may be desirable to strip some naphtha from the solution subsequent to the extraction operation of the copending application and prior to the storage of the solution. In fact, it may be desirable to strip substantially all the naphtha from the solution. The exact procedure is determined after a consideration of the composition of the neutral oil and the desired end products in view of the conditions set forth herein.

The solution of neutral oil and naphtha, containing ordinarily (as the product of the process of the aforementioned copending application) from 1 to 4 parts by volume of naphtha for each part of neutral oil, is fed continuously from storage tank 60 through line 52 into the bottom of a countercurrent end feed extraction column 64 of any convenient design. An aqueous methanol solution containing 75 to 96 per cent by weight of methanol is fed continuously from a storage tank 66 through line 68 into the top of an extraction column 64. From 1 to 4 parts by volume of aqueous methanol are preferably employed for each volume of naphtha solution. Because of the difference in density, the aqueous methanol passes downwardly through the column countercurrently to the lighter naphtha solution which flows upwardly through the column. It is essential to maintain a difference in density between the aqueous methanol and the naphtha solution of neutral oils to assure rapid phase separations. The aqueous methanol phase must be employed as the continuous phase in the end feed extraction operation wherein the feed material is dissolved in the naphtha solvent. Accordingly, the interface in the end feed column will be located near the top of the column above the point at which the aqueous methanol is introduced. With aqueous methanol as the continuous phase, tiny globules of naphtha solution containing dissolved neutral oils are dispersed in and rise upwardly through the aqueous methanol continuous phase. The contacting of the naphtha solution globules and the aqueous methanol continuous phase serves to transfer the aromatic con-

stituents of the neutral oil to the aqueous methanol phase which possesses a greater affinity for the aromatics. In its passage upwardly through the column, the naphtha solution contacts successively, aqueous methanol containing decreased amounts of dissolved aromatics and ultimately contacts fresh aqueous methanol at the top of the column.

The aqueous methanol extract is removed from the bottom of the column 64 through line 70, and fed into a stripping column 72 whence methanol is recovered and recirculated through the system in the same manner as described in connection with the apparatus of Figure 1. Aromatics and water are recovered as the bottom product from column 72 and separated by decantation. Water is recycled in the same manner as described in connection with Figure 1. Naphtha and dissolved saturated hydrocarbons leave the top of the column 64 through line 74 and are fed to a naphtha stripper 76. Naphtha passes overhead through line 78 and is recycled to the extraction process described in the above-mentioned copending application. Saturated hydrocarbons are removed from the stripper 76 through line 80 and recovered.

Example 1

A prepared binary mixture containing 26 per cent by weight of decalin, a typical saturated naphthenic hydrocarbon, and 74 per cent by weight of beta-methyl naphthalene, a typical aromatic, was dissolved in a hexane cut of petroleum naphtha, boiling in the range 67 to 72° C. The solution, containing 20.7 weight per cent of the prepared mixture, was fed into the bottom of a 1 inch diameter, 8 feet long, extraction column containing 28 turbulent zones alternately disposed between 29 calming zones. For each part by volume of hexane solution fed to the column, two parts of aqueous methanol were fed into the top of the column at concentrations of 85, 88 and 90 weight per cent of methanol respectively. The aqueous methanol was the continuous phase.

Analysis of the results of these experiments is presented in Figure 3, which shows that at solvent concentrations between 86 and 96 per cent methanol by weight high yields of aromatics in high purity can be recovered; also within this critical range high yields of high purity saturated hydrocarbons can be recovered from the feed material. For methanol concentrations below 86 weight per cent (but above 75 weight per cent) the present process also is effective. However, where both high purity and high recovery are desired, the methanol concentration must fall within the range of 86 to 96 per cent by weight.

Example 2

A tar acid free neutral oil produced from a low temperature coal carbonization tar fraction boiling in the range of 160 to 325° C., in accordance with the method described in the aforementioned copending United States patent application, was fed in a solution of hexane which has a boiling range of 67 to 72° C. (4 parts hexane to 1 part by volume of neutral oil) into the bottom of the same extraction column described in connection with Example 1. The tar acid free neutral oil contained 79 per cent by weight of aromatics and 21 per cent by weight of naphthenes and paraffins. Aqueous methanol containing respectively 88, 92 and 95 per cent by weight of

methanol was fed into the top of the extraction column and constituted the continuous phase within the column. Two parts of aqueous methanol by volume were used for each volume of the hexane solution containing dissolved neutral oils.

Neutral oils from distillate fractions of tar produced by the low temperature carbonization of coals, for example, contain inter alia aromatic compounds comprising one or more conjugated rings. These aromatics are predominantly alkylated, i. e., containing one or more paraffinic side-chains attached to the ring. There are substantially no paraffinic or olefinic compounds in the neutral oils, whose remaining principal constituents comprise saturated naphthenic-type materials, containing one or more completely saturated ring structures, predominantly alkylated.

The neutral oil also may contain a small amount of naphthenic material with one or more olefinic side-chains. These materials are recovered in the aqueous methanol extract by my new process.

For the purpose of these experiments, the products were analyzed by the method reported in Anal. Chem. 20, 456-7, (1948), in which both olefinic and aromatic compounds are dissolved in a mixture of fuming sulfuric acid and glacial acetic acid, capable of dissolving any hydrocarbons except naphthenes, paraffins or combinations thereof. Therefore, any naphthenic component with an olefinic side-chain is reported in the results here as an aromatic compound.

An analysis of the results of these experiments is presented in Figure 4, where it is clearly seen that for products with both high purity and high recovery, the methanol concentration must be within the critical range of 86 to 96 weight per cent. However, where (as here) the feed stock is the neutral oil produced from the tar of a low temperature carbonization of bituminous coal, the critical range of methanol concentrations is even more narrow if both high purity and high recovery are desired. The critical range for neutral oils as described is from 91 to 96 per cent by weight of methanol.

A further analysis of the results of these experiments is presented in Figures 5 and 6 wherein respectively the hydrogen-to-carbon ratio and the density of the feed material and the recovered products are employed as indicative parameters to demonstrate the effectiveness of this new process. Feed density was 0.95 and the hydrogen-to-carbon ratio was 1.27. The aqueous methanol extract density was greater than that of the feed stock and the hydrogen-to-carbon ratio of the aqueous methanol extract was less than that of the feed stock, indicating increased aromaticity of the extract. Conversely, the naphtha soluble product showed an increased hydrogen-to-carbon ratio and a decreased specific gravity when compared with the feed material. It is significant to note that the feed material in each instance was predominantly aromatic.

According to the present invention, therefore, an oil containing aromatics and saturated hydrocarbons can be separated into two fractions, of which one possesses an increased aromatic concentration and the other an increased concentration of saturated hydrocarbons. Under certain conditions, the aromatics can be recovered in high yield and substantially free of saturated hydrocarbons, which themselves are recovered in high yields and substantially free of aromatics.

The process is continuous and can be performed with negligible solvent losses.

According to the provisions of the patent statutes, I have explained the principle, preferred construction, and mode of operation of my invention and have illustrated and described what I now consider to represent its best embodiment. However, I desire to have it understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically illustrated and described.

I claim:

1. An extraction process for separating aromatic and saturated hydrocarbons, contained in a substantially tar acid free oil boiling below 350° C., which comprises feeding said oil into a vertical countercurrent extraction zone, feeding a solvent consisting of paraffinic naphtha boiling within the range 60 to 130° C. and having a density of less than 0.8 into the bottom of said extraction zone, feeding a second solvent consisting of an aqueous methanol solution containing 75 to 96 per cent by weight of methanol and the balance water into the top of said extraction zone, passing said naphtha through said extraction zone countercurrently to and in intimate contact with said aqueous methanol solution, and recovering an aqueous methanol extract and a naphtha solution from said extraction zone.

2. The process of claim 1 in which the naphtha boils in the range of 60 to 100° C.

3. The process of claim 1 in which the aqueous methanol solution contains 86 to 96 per cent methanol by weight.

4. An extraction process for separating aromatic and saturated hydrocarbons, contained in a substantially tar acid free oil boiling below 350° C. of which aromatics are the predominant constituent, which comprises the steps of feeding said oil into a vertical, countercurrent extraction zone, feeding a solvent consisting of paraffinic naphtha boiling within the range 60 to 130° C. and having a density of less than 0.8 into the bottom of said extraction zone, feeding a second solvent consisting of an aqueous methanol solution containing 75 to 96 per cent by weight of methanol and the balance water into the top of said extraction zone, passing said naphtha through said extraction zone countercurrently to and in intimate contact with said aqueous methanol solution, and recovering an aqueous methanol extract and a naphtha solution from said extraction zone.

5. An extraction process for separating aromatic and saturated hydrocarbons from the neutral oil separated from a distillate fraction of the tar produced by the low temperature carbonization of bituminous coal, which comprises feeding that portion of said neutral oil boiling below 350° C. into a vertical, countercurrent extraction zone, feeding a solvent consisting of paraffinic naphtha boiling within the range 60 to 130° C. and having a density of less than 0.8 into the bottom of said extraction zone, feeding a second solvent con-

sisting of an aqueous methanol solution containing 91 to 96 per cent by weight of methanol and the balance water into the top of said extraction zone, passing said naphtha through said extraction zone countercurrently to and in intimate contact with said aqueous methanol solution, and recovering an aqueous methanol extract and a naphtha solution from said extraction zone.

6. The process of claim 5 in which the naphtha is the hexane cut of petroleum naphtha having a boiling range of about 67 to 72° C.

7. An extraction process for separating aromatic and saturated hydrocarbons contained in a substantially tar acid free oil boiling below 350° C. which comprises feeding said oil into a vertical countercurrent extraction zone at a point between the ends thereof, feeding a solvent consisting of paraffinic naphtha boiling within the range 60 to 130° C. and having a density of less than 0.8 into the bottom of said extraction zone, feeding a second solvent consisting of an aqueous methanol solution containing 75 to 96 per cent by weight of methanol and the balance water into the top of said extraction zone, passing said naphtha through said extraction zone countercurrently to and in intimate contact with said aqueous methanol solution, and recovering an aqueous methanol extract and a naphtha solution from said extraction zone.

8. The process of claim 7 in which the naphtha constitutes the continuous phase in the extraction zone.

9. An extraction process for separating aromatic and saturated hydrocarbons contained in a substantially tar acid free oil boiling below 350° C., which comprises preparing a solution of said oil in a solvent consisting of paraffinic naphtha boiling within the range 60 to 130° C. and having a density of less than 0.8, feeding said naphtha solution into the bottom of a vertical, countercurrent extraction zone, feeding a second solvent consisting of aqueous methanol containing 75 to 96 per cent by weight of methanol and the balance water into the top of said extraction zone as the continuous phase within said extraction zone, passing said naphtha solution through said extraction zone countercurrently to and in intimate contact with said aqueous methanol and recovering an aqueous methanol extract from the bottom of said extraction zone and a naphtha solution from the top of said extraction zone.

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