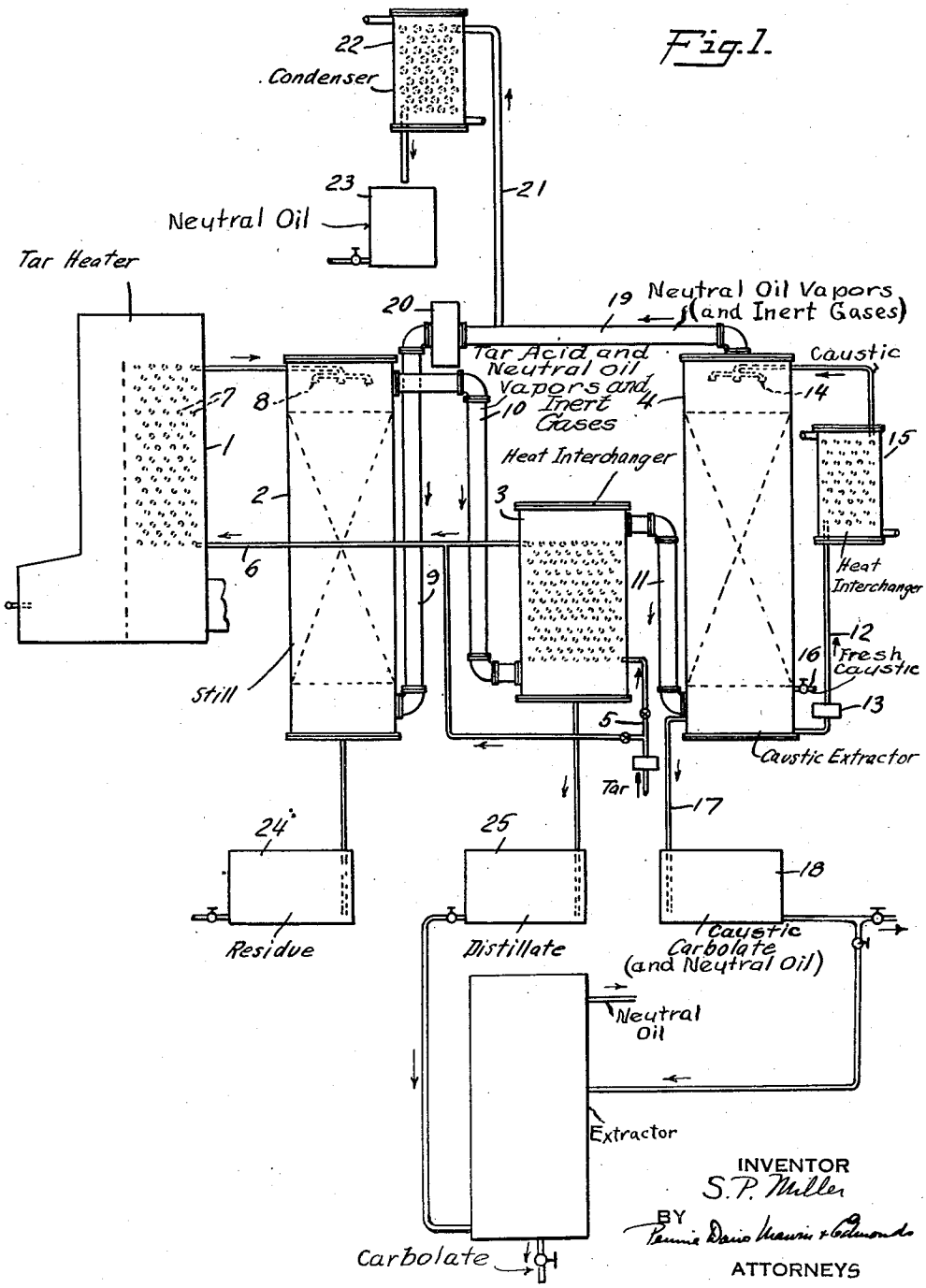
May 12, 1936. S. P. MILLER 2,040,100

TREATMENT OF TAR

Original Filed March 23, 1931 2 Sheets-Sheet 1

Fig. 1.



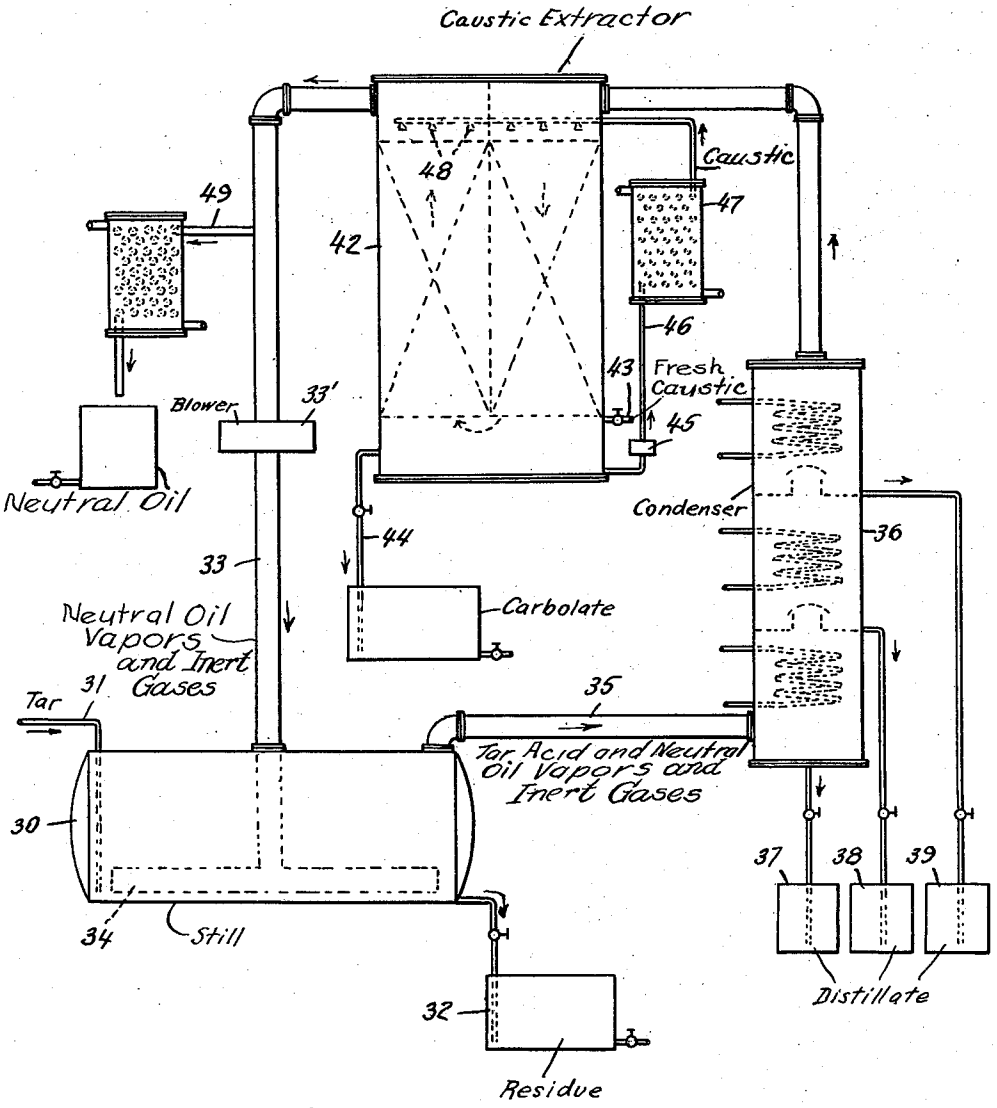
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TREATMENT OF TAR

Original Filed March 23, 1931 2 Sheets-Sheet 2

Fig. 2.



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UNITED STATES PATENT OFFICE

2,040,100

TREATMENT OF TAR

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Application March 23, 1931, Serial No. 524,616
Renewed November 4, 1932

9 Claims. (Cl. 196—76)

This invention relates to the treatment of heavy hydrocarbon material such as tar or oil and obtaining distillate and at least a portion of the tar acids as carbolate. The invention includes both the method of operation and apparatus therefor.

The process of the present invention is applicable to the treatment of tars such as coke-oven tars, gas-retort tars, vertical-retort tars, low-temperature tars, and other tar-acid and neutral-oil containing hydrocarbon materials such as coal-tar distillates. It may be applied to the treatment of raw, moisture-containing tar, or topped tar, etc., or to the separate treatment of tar fractions such as the heavy tar from the collector main of a coke-oven plant or the light tar or tarry oil from the condensers of such a plant. It may be applied to the treatment of mixtures containing coal tars, etc. The invention will be described more particularly as applied to the treatment of coke-oven tar.

According to this invention, tar acids and neutral oils are volatilized from the tar in a current of inert gases. Any suitable inert gases may be used in the distillation, although ordinarily steam will be employed. If raw tar is treated the moisture present in the raw tar during the treatment is converted into steam and this may be used for the inert gas distillation. By carrying out the operation in a current of steam, the tar acids and neutral oils in the tar vaporize at reduced temperatures.

The gases and vapors resulting from the steam or other inert gas distillation are cooled to condense part of the oil vapors of the boiling range desired, and this condensate is separately recovered as distillate oil. If this distillate contains desirable tar acids, it may be extracted with an alkaline reagent to remove them.

After the cooling step, the gases, which contain vapors of both tar acids and neutral oils, are treated with an alkaline reagent to extract and recover the tar acids from the gases as carbolate. This tar-acid extraction may be accompanied by cooling, or after the tar-acid extraction the gases may be cooled to condense neutral oils from the resulting gases and vapors. However, any such condensation of oils is only partial and the resulting cooled gases and vapors are at least in part recirculated for further inert gas distillation of the tar. According to this process, therefore, there are produced as products from the treatment of tar, a distillate, tar acids as carbolate, and a residue. Neutral oils may also be separately condensed from the gases from which tar acids have been removed and collected as product.

The heat for the vaporization will ordinarily be supplied by heating the tar before it enters the still in which the tar acids and neutral oils are vaporized from the tar. The recycled gases are reheated, in part by bringing them into heat interchange relation with the hot residue. The residue is thereby cooled and heat is economized. Additional heat may be supplied to the gases from some outside source, but ordinarily no more than a portion of the heat for the distillation will be supplied in this manner and according to the preferred method of operation all the heat is supplied to the tar before it is treated and no outside heat is added to the gases.

By proper temperature control throughout the system, products of different characteristics are obtainable. By heating the tar to a high temperature so that the gases and vapors leave the still at a high temperature, and then cooling them to a temperature of about 200° C., or lower, before bringing them into contact with the caustic used for extraction, the oil recovered as distillate will be of the boiling range desired for creosote oil.

On the other hand, the gases leaving the still may contain no substantial amount of oils of higher boiling range than the distillate fraction ordinarily separately recovered as carbolic oil and extracted for tar acids. On cooling these gases and vapors, a fraction containing part of the tar acids of low boiling range will be condensed. The balance of the tar acids will be extracted by direct contact with caustic. Different methods may be utilized for extracting the tar acids from both the distillate and the gases. For example, the alkaline solution used for extracting the gases may contain an appreciable excess of caustic to insure complete removal of the tar acids from the gases and the caustic-carbolate solution thus formed may then be used for extracting tar acids from the tar-acid containing distillate. By regulating the temperature at which the gases and vapors leave the still and the temperature to which they are cooled before they enter the caustic extractor, the boiling range of the distillate oil which is separately collected can be controlled. Any suitable form of condensing apparatus may be employed, such as fractionating condensers, either with or without fractionating columns, etc., and two or more oil fractions of regulated boiling range may thus be recovered from the gases before they enter the caustic extractor.

The caustic employed for scrubbing tar-acid vapors from the gases may be ordinary caustic solution of suitable strength. Caustic potash or

sodium sulfide solutions may also be employed in some cases. Where the gases and vapors pass thru the caustic scrubber at a much higher temperature than the caustic soda solution, the gases will be cooled by contact with the solution and the solution itself will be heated and may be concentrated by evaporation of part of the water. In this way relatively dilute caustic solutions may be employed. The strength of the carbolate solution in the caustic absorber may be regulated either by regulating the strength of the caustic soda solution employed, or the temperature of the scrubber, or the temperature of the gases and vapors passing thru the scrubber. If any steam is allowed to condense on its way thru the scrubber, the carbolate will be diluted, while by maintaining the gases and the scrubber at a regulated temperature any particular strength of carbolate solution that is desired may be produced. It will be understood that the tar acids contained in vapor form and carried by the gases will combine with the sodium or other metal of the caustic solution at least to some extent to form sodium or other metal phenolates which will remain in solution until the absorbing capacity of the solution is exhausted or reduced to the desired extent.

Ordinarily the gases will be below 200° C. before being treated with the caustic. If they are at a higher temperature it will be desirable to cool them to about 200° C., or lower, before they are brought into contact with the caustic to prevent excessive vaporization of water from the caustic and carbolate and the formation of solutions which are difficult to handle.

The caustic absorber may be constructed in stages so that a completely saturated carbolate can be made in the stage first in contact with the gas while there is still an excess of caustic in the second and other stages. In this way the caustic solution can be exhausted of caustic soda and at the same time the tar acid vapors extracted with substantial completeness from the gases.

Selective extraction of tar acids from gases and vapors containing a mixture of tar-acid vapors may be effected by installing and employing several caustic scrubbers and carrying out the scrubbing operation in stages. If, for example, a carbolate rich in phenol and a second carbolate rich in higher boiling tar acids including cresols is desired, a three-stage absorber or scrubber can be employed with the supply of caustic to the lower or first section or scrubber equivalent only to the phenol entering the system with the inert gas being circulated. The carbolate will be allowed to become completely saturated and to build up to the maximum percentage of the phenols before any is withdrawn. Phenol being more strongly acidic than the higher phenols, e. g. cresols and xylenols, will to a considerable extent replace these higher phenols from their combination with caustic soda so that when the caustic soda is consumed and a carbolate solution is produced, the passing of additional gases carrying phenols and higher phenols will result in the driving off of higher phenols and the absorption of phenol until the carbolate solution in the lower section will be greatly enriched in sodium phenolate while the higher phenols will be absorbed in the higher sections. With a multi-stage operation of this kind it is possible to produce carbolates predominating in phenol, the cresols, the xylenols, etc. If still more selective extraction is desired the carbolate absorber may be constructed in a larger number of stages, for example, five or six, and a plurality of different tar-acid fractions obtained.

It is not necessary to effect complete neutralization of the tar acids in the extractor and in a commercial plant complete removal of the tar acids will not be effected because of the practical difficulty of obtaining contact of the alkaline reagent with all particles of the gas. For example, the oil content of the gases returned to the still may contain under good commercial operating conditions, no more than a few tenths of a percent of tar-acid vapors.

The blower for recycling the gases will normally be located between the caustic extractor and the still. The entire system is preferably operated under substantially atmospheric pressure, but pressures above or below atmospheric may be used. In order to maintain a constant pressure in the cycle, gases and vapors may be bled from the cycle to compensate for the steam and gases generated. A bleed condenser is advantageously provided to recover oil from the vapors in the bleed.

It is possible to control the cooling and condensation of neutral-oil vapors subsequent to the caustic treatment so that a small amount of steam is condensed which is just the amount necessary to offset the steam generated and thus keep the system in balance. Air or fresh steam or other inert gases may be introduced into the system to keep it balanced or to aid in the distillation of the tar. The bleed will be sufficient to compensate for any excess inert gas in the system.

The tar which is subjected to the distillation may be preheated by the use of a steam preheater or by the use of waste heat from the flue gases in the flues at coke or gas plants or by the use of waste flue gases from tar stills, steam-boiler plants, etc., or by the use of heat interchangers in which hot tar or pitch is employed as the heating medium, or by separate heating stills, etc.

Where the recovery of distillate and tar acids from the tar is carried out at coal-distillation plants at which coal tar is produced, such as coke-oven plants or gas plants, the tar separated from the gases at an elevated temperature may be kept hot and employed in a heated condition. In many cases the temperature will be sufficient for carrying out the inert-gas distillation without added heat or at least without any great amount of additional heat. Where the collector mains of a coke-oven plant are operated at high temperature with the use of hot tar for flushing the main, the hot tar drawn off from the main may be at a sufficiently high temperature so that it does not require further heating and so that it can be led directly from the main to the apparatus for the distillation.

In the case of an ordinary tar still containing a large batch of tar, the still can be heated until the tar is at the proper temperature and inert gases can then be recirculated thru the tar, the resulting gases and vapors passed to the condenser, thru the caustic solution and the gases recirculated until the distillation has been completed. The residue may then be subjected to further distillation if desired. Where the tar is distilled in a pipe coil still with heating of the tar in a pipe coil and discharge of the heated tar into a vapor box or vapor-separating chamber, the initial heating of the tar can be so regulated that the tar will be at the proper temperature in the vapor-separating chamber and the current of inert gases can be passed thru the tar in such

chamber to remove the desired constituents after which the residue can be heated to a higher temperature by circulation thru another pipe coil which discharges into another vapor-separating chamber, etc.

Oil vapors will in many cases serve satisfactorily as the gaseous medium employed for the removal of the tar acids from the tar. This will particularly be the case with tars containing a considerable percentage of lower boiling oils, such as benzol, toluol and xylols or when dehydrated tars are treated. By heating the tar to the point at which these lower boiling oils are vaporized, the vapors may be recirculated thru the tar and the caustic solution. These oil vapors serve at least, in part, as the inert gases employed in the process.

In some cases it will be desirable to operate with relatively high temperatures in the still and, after cooling of the gases to condense part of the contained oil vapors as distillate, to pass the steam or other inert gases at high temperature to the caustic absorber. To avoid evaporation of the caustic or carbolate solution beyond any particular desired point, it may be cooled by means of cooling coils, or water may be continually or intermittently added to the caustic-carbolate solution to maintain the desired concentration. Under such conditions part of the oil vapors in the gas stream will be condensed in the extractor and may be separated from the carbolate produced by decantation, centrifuging, etc. The separation may be carried out continuously or intermittently and the neutral oil thus recovered may be returned to the tar residue or kept separate as a secondary product of the process. However, it will generally be advantageous to cool the gases before they enter the extractor to such a temperature that it will be unnecessary to cool them to any great extent in the extractor.

The distillation of this invention is preferably carried out as a continuous process, with continuous feed of the tar to the still, and with continuous discharge of the residue and with batch-wise or continuous operation of the caustic absorber using successive batches of caustic soda solution for forming phenolates or supplying additional fresh caustic continuously and withdrawing the carbolate solution continuously. With batch-wise operations caustic absorbers may be installed in parallel or in duplicate with piping arranged for allowing saturated carbolates to be produced first in one and then in the other assorber.

The invention will be further described in connection with the accompanying drawings but it is intended and is to be understood that the invention is not limited thereto:

In the drawings:

Fig. 1 shows vaporizing apparatus or still in connection with a tar heater; and

Fig. 2 shows apparatus in connection with a still which is itself heated externally.

Fig. 1 of the drawings shows a tar heater 1, a still 2, a condenser shown as the heat interchanger 3, and a caustic extractor 4. The still and caustic tower are both shown as packed towers. The tar enters thru the pipe 5 and is preheated by heat interchange with the hot gases and vapors from the still in the heat interchanger 3. It then passes by the pipe 6 into the tar heater 1 where it is heated in the coil 7 preferably above its initial boiling point. It is then introduced into the still 2 thru the spray nozzles 8. In the still the lower boiling constituents are flashed and separated from the residue. Inert gas is introduced into the still thru the main 9. Those vapors of neutral oils in the inert gases which are of the same boiling range as the tar acids removed from the gas stream in the caustic extractor 4 tend to prevent or repress the vaporization of neutral oils of this boiling range from the tar in the still, but tar acids of this boiling range are vaporized because after removal of some of these tar acids in the extractor the gases are no longer saturated with vapors of these tar acids, and if complete extraction of the tar acids is effected by the caustic there will be a complete absence of such vapors in the gases entering the still. The degree to which the vaporization of neutral oils is repressed depends upon the boiling range of the neutral oil constituents in the tar and in the vapor and on the operating conditions. By such selective distillation in the still, tar acids may be more clearly completely removed from tar or other hydrocarbon material than the neutral oils of the same boiling range.

From the still the gases and vapors, which include vapors of both neutral oils and tar acids, pass to the main 10 into the heater interchanger 3. Here they are cooled by heat interchange with the tar whereupon distillate separates from the gases. This cooling is so regulated that only a portion of the oil vapors are condensed and separate from the gas stream as distillate. This distillate will be of higher or lower boiling range depending upon the temperature at which the gases and vapors leave the still and the temperature to which they are cooled. The uncondensed vapors, which include both vapors of tar acids and neutral oils, pass from the heat interchanger thru the main 11 into the caustic extractor 4. Other suitable condensing means such as a direct contact condenser, etc. may be substituted for the heat interchanger shown, and a plurality of different means may be employed to separately recover a plurality of oil fractions.

In the caustic extractor the tar-acid vapors combine with the caustic and are separated from the gas stream as carbolate. It is advantageous to recirculate a portion of the carbolate produced, together with the fresh caustic in order to expose a large surface of liquid to the action of the gases. For this purpose a pipe 12 and pump 13 are shown for recycling at least a portion of the caustic and respraying it into the gases in the extractor 4 thru the spray nozzles 14. A heat interchanger 15 is provided to control the temperature of the caustic solution. Fresh caustic is introduced thru the line 16. Excess of the carbolate solution is drawn off thru the line 17 and collected in the tank 18. It may be treated in the usual way for the recovery of tar acids as by acidification with carbon dioxide, etc.

Neutral oils may be recovered from the gases before they are returned to the still, either by subjecting the gases to regulated cooling in the extractor 4 or in a separate condenser located between the extractor and the still.

In Fig. 1 a main 19 is provided to convey the gases from the extractor 4 and they are returned to the still by means of the blower 20 and the main 9.

In order to maintain the apparatus under constant pressure, preferably at approximately atmospheric pressure, a vent is provided at 21 to bleed off any excess gases and vapors which may result from decomposition of tar constituents,

vaporization of water present in the tar, etc. A bleed condenser 22 is shown for condensing oils from the excess gases. These oils, which are essentially neutral oils, although they may contain some tar acids, are collected in the tank 23. The apparatus may be maintained at atmospheric pressure or somewhat above or below atmospheric pressure. The apparatus shown contains no valves restricting communication with the atmosphere and is operated at substantially atmospheric pressure.

The products of the operation are the residue, which is drawn off from the still 2 and collected in the tank 24, distillate, which is drawn off from the condenser 3 and collected in the tank 25, carbolate, which is drawn off from the caustic extractor 4 and collected in the tank 18, and also neutral oil, which may be condensed from the excess gases and collected in the tank 23, and additional neutral oil, which may be recovered from the extractor or from a separate condenser thru which the gases pass after they leave the extractor and before they reenter the still.

The apparatus of Fig. 1 can be operated under different conditions of temperature, etc. to produce different products. For example, by heating the tar to a high temperature of, for example, 350° C., in the tar heater and operating so that on flashing the vapors leave the vapor-separating chamber and still 2 thru the line 10 at a temperature of 300° C., for example, these gases and vapors may be cooled to around 175° C. before entering the caustic tower, thus causing an oil suitable for creosote oil to separate in the condenser 3. This oil contains tar acids of higher boiling range but ordinarily only a small amount of the lower boiling tar acids. If ordinary coke-oven tar is being treated, the gases and vapors leaving the condenser at 175° C. are rich in vapors of the lower boiling tar acids. These are extracted in the extractor, preferably without condensation of any substantial amount of neutral oil of this boiling range. The neutral-oil vapors are then recirculated thru the still. The gases may be cooled after leaving the caustic extractor and before re-entering the still to condense neutral oil, if desirable. The products of such an operation are creosote oil, tar acids, a separate neutral-oil fraction, if desirable, and a pitch residue, which may be of high melting point, particularly where neutral oils are condensed from the gases after they leave the caustic extractor.

It is not necessary to heat the tar to such a high temperature. For example, coke-oven tar containing in the neighborhood of 3%–6% of low boiling tar acids may be heated in the tar heater to about 200° C. so that, on flashing, the vapors leave the chamber 2 at 150° C. On cooling to 105° C. in the condenser 3 these gases will yield an oil containing 10% to 20% or more of tar acids. The resulting gases and vapors are then extracted in the caustic extractor and returned to the distilling chamber 2 without substantial condensation of neutral oil. The residue may contain neutral oils of a particular boiling range, although tar acids of this boiling range may have been substantially completely vaporized therefrom. The tar-acid oil from the condenser may be used as such, or it may be extracted or otherwise treated.

In carrying out the process the rate at which the gases are recirculated will depend upon the temperature at which the tar enters the still or separating chamber, whether this still is heated, the temperature at which the gases leave the still, the extent to which the gases are cooled before they re-enter the still, and the quantity of gases and vapors bled from the cycle, etc. For instance, in the last example given the temperatures of operation are 200° C. or above for the tar entering the chamber 2 from the heater 1, 150° C. for the gases in the main 10, and about 105° C. for the gases from the time they leave the condenser 3 until they re-enter the chamber 2. The distillate recovered from the condenser 3 represents about 15% based on the volume of the tar treated, when the gases are recirculated at the rate of about 300 cubic feet per gallon of tar treated. Similar results are obtained by changing the rate of recirculation to 200 cubic feet per gallon of tar treated and introducing the tar into the chamber 2 at about 250° C. and maintaining a gas temperature leaving the chamber 2 at about 175° C.

In Fig. 2 a still 30 heated by direct fire is shown for treating the tar. Tar is fed to the still continuously thru the line 31 and the residue is drawn off continuously or intermittently to storage 32. Inert gases are introduced into the still thru the main 33 by the blower 33' and continuously bubble up thru the tar in the still thru the perforated distributing pipe 34. The gases and vapors leave the still thru the main 35 and enter the condenser 36.

The condenser is shown as a fractionating condenser. The distillate, collected in several fractions, is drawn off into the receivers 37, 38, and 39. Any suitable condensing medium may be used. The cooling is regulated so as to effect only partial condensation.

The gases which leave the condenser include vapors of both tar acids and neutral oils. The are sprayed with caustic in the extractor 42. Fresh caustic is added at 43. Carbolate is drawn off at 44. A portion of the carbolate containing excess caustic is recirculated by the pump 45 thru the line 46 and heat interchanger 47 and sprayed into the gases thru the sprays 48. A bleed is shown at 49 to maintain the system under atmospheric pressure.

The extent to which the tar is distilled according to this invention may be so regulated as to produce a residue of higher or lower melting point as desired, or to produce distillate of higher or lower boiling range which may or may not be extracted for its tar acid content, and a separate neutral oil distillate may or may not be separated from the gases after they leave the extractor, as desirable. The process may be varied within rather wide limits to produce various products. For example, the residue from the still may be road pitch with a melting point of 105° F. or pitch with a melting point of 145° F. suitable for roofing pitch; or pitch of much higher melting point, e. g. 300° F. or higher, may be produced, which may or may not be blended with tar, etc. to produce blended pitches suitable for roofing pitch or road tars, etc.

The process is applicable generally to tar stills of the type in which the distillation is effected in a current of inert gases. The distillation may be carried on as a continuous process with the continuous addition of fresh hydrocarbon material to the still, or the distillation may be carried out batchwise.

Caustic extractors of various types may be employed including towers and apparatus with vari-

ous types of mechanical spray means. The drawings illustrate the use of packed towers for this purpose. It is advantageous to use apparatus of the type in which the resistance offered to the flow of the gases is low in order that a low pressure blower may be used for circulating the gases thru the system.

It will be understood that variations and modifications can be made in the process as hereinbefore described, without departing from the spirit and scope of the invention.

This application is in part a continuation of my co-pending application Serial Number 383,859, filed August 6, 1929.

I claim:

1. The method of treating hydrocarbon material, which comprises selectively distilling liquid tar-acid-and neutral-oil-containing hydrocarbon material in a current of inert gases including vapors of neutral oils, thereby vaporizing both tar acids and neutral oils therefrom, subjecting the resulting gases and vapors to regulated cooling to condense distillate therefrom, simultaneously extracting tar acids and condensing neutral oils from the resulting cooled gases by direct contact with a cooled alkaline reagent which reacts with tar acids to form a carbolate and then bringing at least a portion of the neutral oil vapors remaining in the gas stream together with inert gases into direct contact with hydrocarbon material to effect selective distillation thereof.

2. The method of treating tar, which comprises heating tar, distilling tar acids and neutral oils from the heated tar in a current of inert gases, subjecting the resulting gases and vapors to regulated cooling to separate distillate therefrom while leaving tar acids and neutral oils in vapor form, then extracting tar acids from and removing at least a part of the neutral oil vapors from the resulting gases and vapors by directly contacting and cooling them with an alkaline reagent which reacts with the tar acids to form a carbolate, dividing the resulting inert gases into two portions and recycling one portion, bringing it into direct contact with the heated tar in the inert gas distillation.

3. The method of treating tar, which comprises distilling oils including creosote oil from coal tar in a current of inert gases including neutral oil vapors, at elevated temperatures such that the gas and vapors leaving contact with the tar are at a temperature of about 150° C. or higher, cooling the resulting gases and vapors to separate creosote oil therefrom as distillate while leaving gases and vapors in vapor form, extracting tar acids from the cooled gases and vapors by directly contacting them with an alkaline reagent which reacts with the tar acids to form a carbolate while leaving neutral oil vapors in vapor form therein and then recycling at least a portion of the remaining neutral oil vapors in admixture with inert gases for use in the distillation of the tar.

4. The method of treating tar, which comprises distilling tar in a current of inert gases including neutral oil vapors, fractionally cooling the resulting gases and vapors to separate at least two distillate fractions therefrom, while leaving tar acids in vapor form in the cooled gases, extracting tar acids from the gases while leaving neutral oils in vapor form therein and then recycling at least a portion of the remaining neutral oil vapors in admixture with inert gases for use in the distillation of the tar.

5. The method of treating tar to distill it and simultaneously to recover a creosote oil distillate, a solution containing carbolates of low boiling tar acids and a pitch having a melting point of about 105° F. or higher, which comprises passing an inert gas containing neutral oil vapors in contact with tar to selectively vaporize tar acids therefrom, withdrawing the resulting mixture of gas and vapors from contact with the tar, maintaining an elevated temperature of the tar and mixture of gas and vapors in the foregoing steps such that the gas and vapors leaving contact with the tar are at a temperature above about 200° C., cooling the resulting gas and vapor mixture to a temperature in the range of about 200° C. and lower to condense a creosote oil distillate therefrom while leaving low boiling tar acids and neutral oils in vapor form, passing the thus cooled mixture of inert gas and vapors in contact with an alkaline solution which reacts with the tar acids to form a carbolate solution to extract tar acids therefrom and to form a solution containing the aforesaid carbolates of low boiling tar acids, and continuing the aforesaid distillation treatment of the tar until the pitch residue has a melting point of about 105° F. or higher.

6. The method of treating tar containing creosote oil fractions which comprises passing an inert gas containing neutral oil vapors in contact with said tar to selectively vaporize tar acids therefrom, withdrawing the resulting mixture of gas and vapors from contact with the tar, maintaining an elevated temperature of the tar and mixture of gas and vapors in the foregoing steps such that the gas and vapors leaving contact with the tar are at a temperature of about 300° C., cooling the resulting gas and vapor mixture to about 175° C. to separate a condensate of creosote oil therefrom while leaving tar acids and neutral oils in vapor form, and passing the thus cooled mixture of inert gas and vapors in contact with an alkaline solution to extract tar acids therefrom.

7. The method of treating tar which comprises passing an inert gas containing neutral oil vapors in contact with tar to distill it and simultaneously to recover a distillate containing tar acids, a solution containing carbolates of low boiling tar acids and a road pitch, to selectively vaporize tar acids therefrom, withdrawing the resulting mixture of gas and vapors from contact with the tar, maintaining an elevated temperature of the tar and mixture of gas and vapors in the foregoing steps such that the gas and vapors leaving contact with the tar are at a temperature of about 150° C., cooling the resulting gas and vapor mixture to 105° C. to condense a tar-acid distillate therefrom while leaving low boiling tar acids and neutral oils in vapor form, passing the thus cooled mixture of inert gas and vapors in contact with an alkaline solution which reacts with the tar acids to form a carbolate solution to extract tar acids therefrom and to form a solution containing the aforesaid carbolates of low boiling tar acids, and continuing the aforesaid distillation treatment of the tar until the residue has the character of a road pitch.

8. The method of treating tar which comprises passing an inert gas in contact with tar at an elevated temperature to vaporize tar acid and neutral oils therefrom, cooling the resulting mixture of gas and vapors to separate a condensate therefrom while leaving tar acids in vapor form, passing the uncondensed gas and vapors in contact with an alkaline solution supplied in amount

in excess of that required for reaction with the tar acids in the vapors contacted therewith and employing the resulting alkaline carbolate solution for the extraction of tar acids from the aforesaid condensate.

9. The method of treating tar to distill it and recover therefrom a carbolate solution, an oil distillate and a pitch residue which comprises passing an inert gas containing neutral oil vapors in contact with tar to selectively vaporize tar acids therefrom, withdrawing the resulting mixture of gas and vapors from contact with the tar, maintaining an elevated temperature of the tar and mixture of gas and vapors in the foregoing steps such that the gas and vapors leaving contact with the tar are at a temperature of about 150° C. or higher, cooling and contacting the resulting gas and vapor mixture with an alkaline solution which reacts with the tar acids to form a carbolate solution to condense a portion only of the oil vapors from the gases to produce said oil distillate and to extract tar acids to form said carbolate solution, recirculating the thus treated gas into renewed contact with the tar, and continuing the aforesaid distillation treatment of the tar until the residue has a melting point corresponding to that of road pitch or higher.

S. P. MILLER.

CERTIFICATE OF CORRECTION.

atent No. 2,040,100. May 12, 1936.

STUART PARMELEE MILLER.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, first column, line 53, for the syllable "as-" read ab-; and second column, line 20, for "clearly" read nearly; page 5, second column, lines 45-48, claim 7, strike out the words and comma "to distill it and simultaneously to recover a distillate containing tar acids, a solution containing carbolates of low boiling tar acids and a road pitch," and insert the same after "tar" in line 43, same claim; and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 4th day of August, A. D. 1936.

Henry Van Arsdale

Seal) Acting Commissioner of Patents.