May 12, 1936. S. P. MILLER 2,040,097

DISTILLATION OF TAR

Original Filed Jan. 15, 1931 5 Sheets-Sheet 1

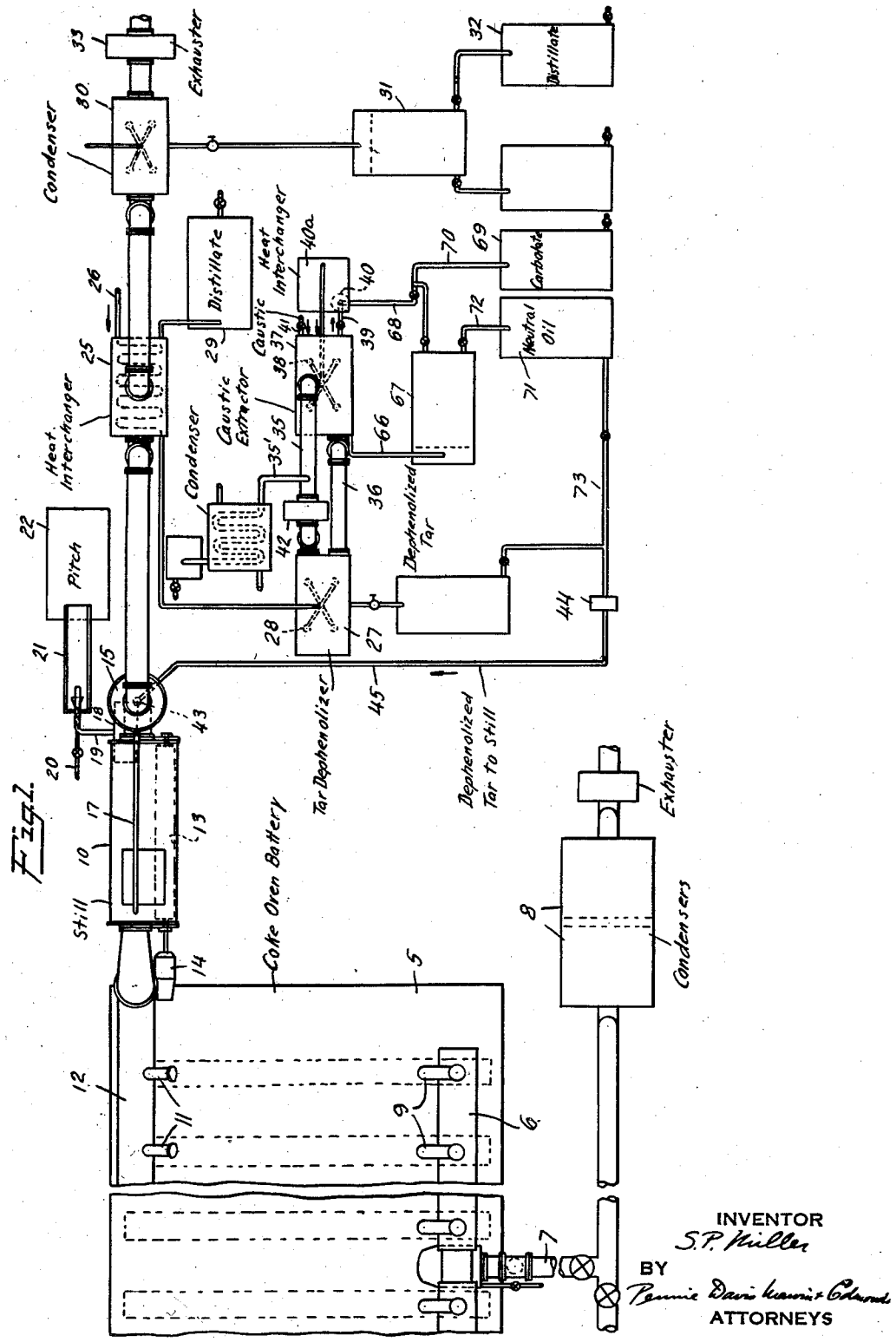


Fig. 2.

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DISTILLATION OF TAR

Original Filed Jan. 15, 1931 5 Sheets-Sheet 2

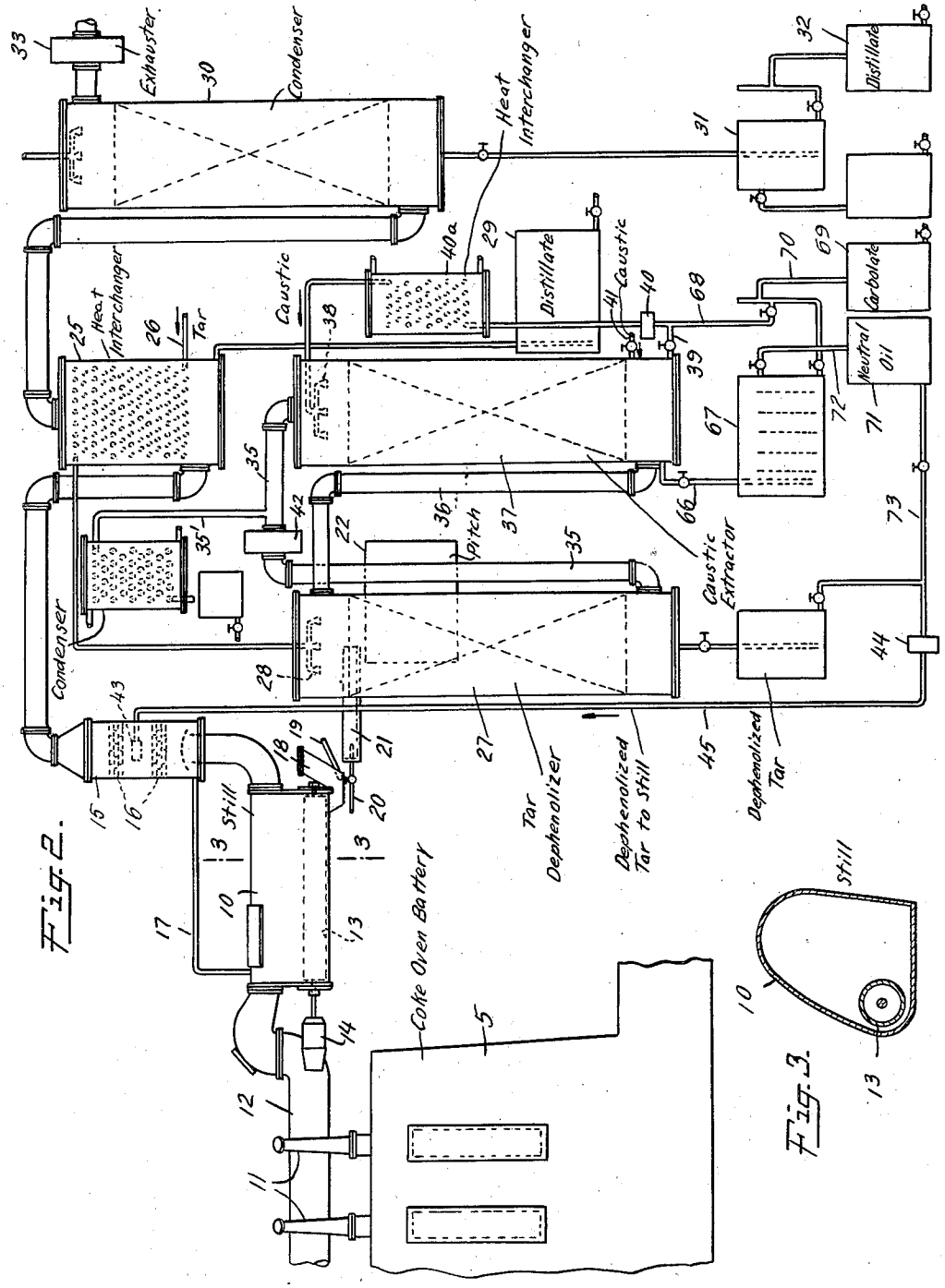


Fig. 2.

Fig. 3.

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DISTILLATION OF TAR

Original Filed Jan. 15, 1931 5 Sheets-Sheet 3

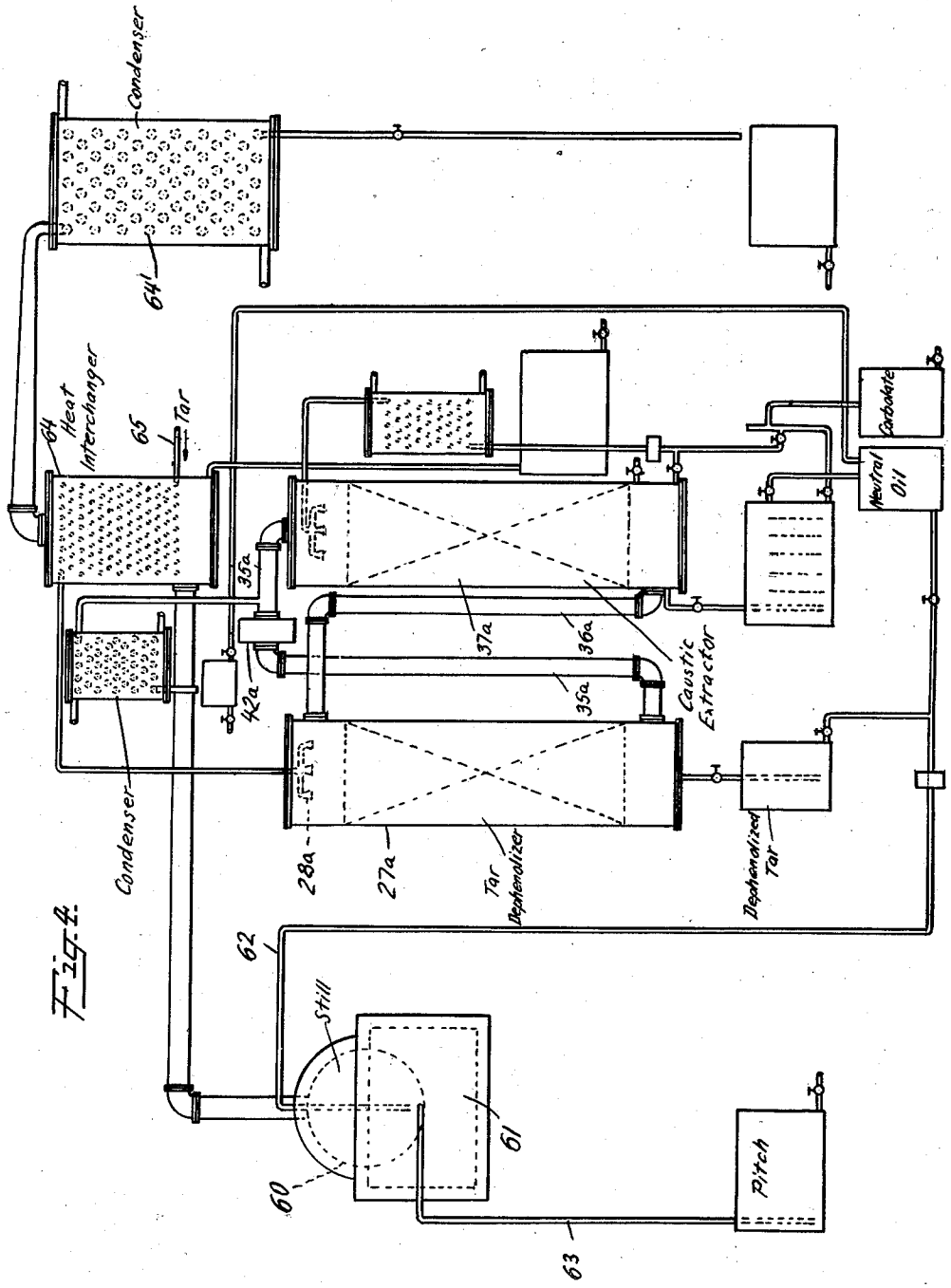


Fig. 4.

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May 12, 1936. S. P. MILLER 2,040,097

DISTILLATION OF TAR

Original Filed Jan. 15, 1931 5 Sheets-Sheet 4

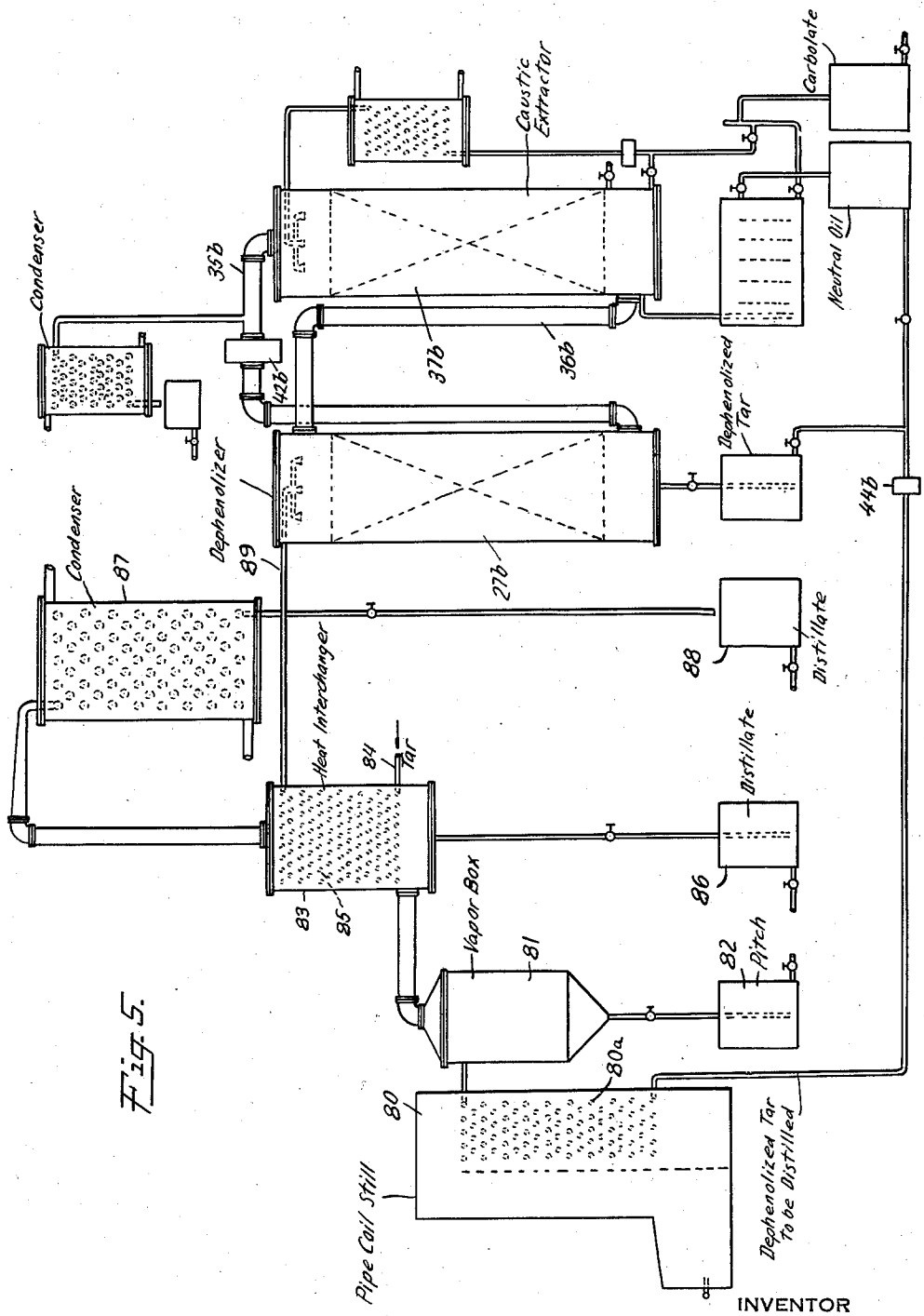


Fig. 5.

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May 12, 1936. S. P. MILLER 2,040,097

DISTILLATION OF TAR

Original Filed Jan. 15, 1931 5 Sheets-Sheet 5

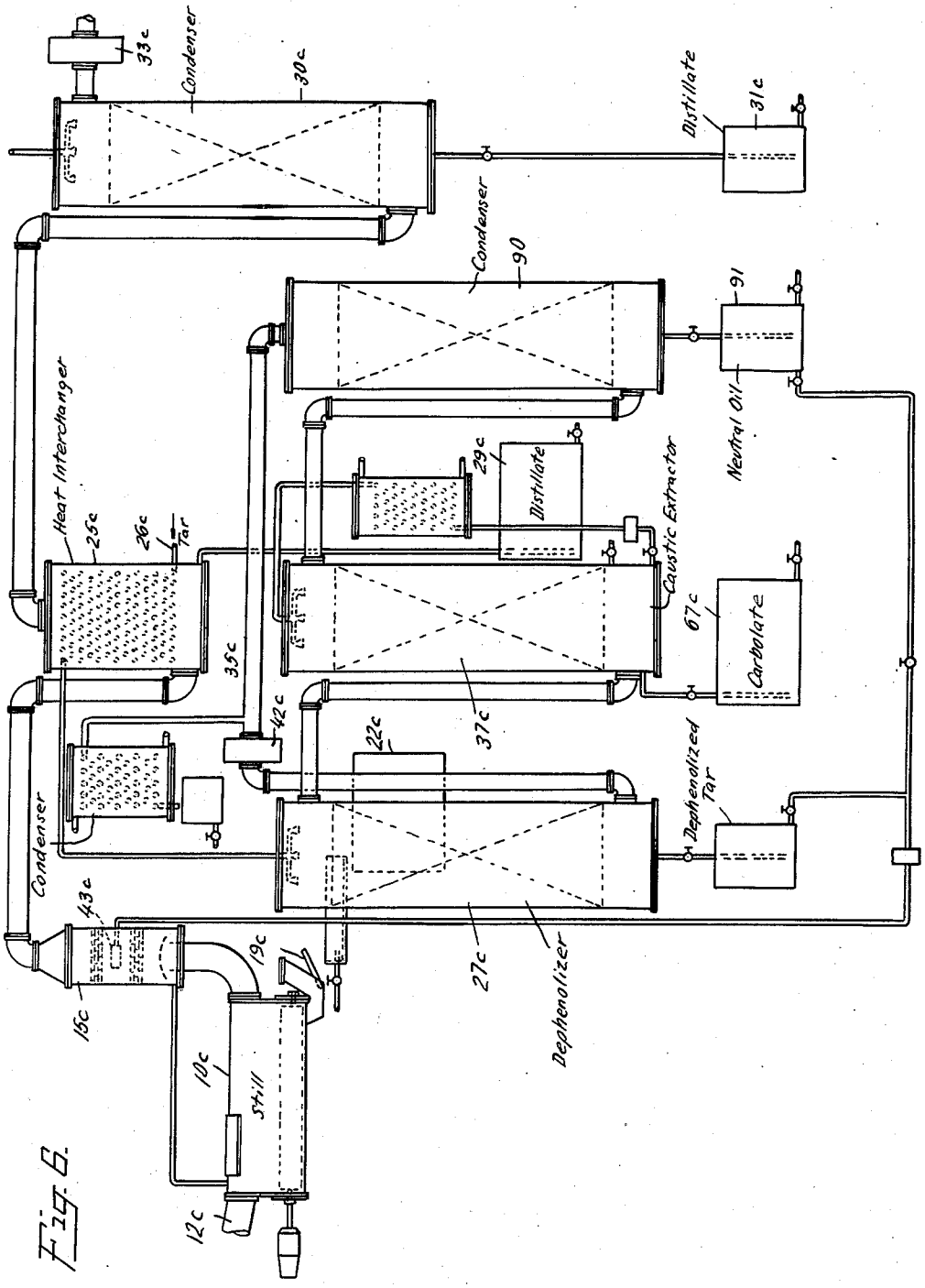


Fig. 6.

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UNITED STATES PATENT OFFICE

2,040,097

DISTILLATION OF TAR

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Application January 15, 1931, Serial No. 508,848
Renewed July 25, 1935

18 Claims. (Cl. 202—30)

This invention relates to the distillation of tar-acid- and neutral-oil-containing hydrocarbon material. More particularly the invention relates to a method of producing and then distilling dephenolized hydrocarbon material involving the separate vaporization of tar acids therefrom. By "dephenolized hydrocarbon material" I mean hydrocarbon material from which at least a portion of the tar acids have been removed without the removal of the same considerable percentage of neutral oils as is required by the present customary methods of tar acid recovery. The distillate and distillation residue obtained from the distillation are both of relatively low tar acid content. The invention includes both the method and apparatus for carrying it out.

According to this invention, the hydrocarbon material containing tar-acids and neutral oils is heated and tar acids are vaporized from the heated material, and separately recovered. The hot residue from this treatment is then subjected to distillation, and a distillate low in tar acids is produced. If the dephenolized material is heated by direct contact with hot gases such as steam or combustion gases or hot coal distillation gases as described in my copending application Serial No. 326,769, filed December 18, 1928, which has issued as U. S. Patent No. 1,920,097 for example, the hot gases and vapors resulting from the distillation may be used to heat the material for the separate tar acid vaporization. If the material is distilled without the aid of hot gases the heat of the vapors may be utilized for the separate tar acid vaporization. The hot residue resulting from the separate and selective tar acid vaporization is then distilled.

While the process of the present invention is applicable in general to hydrocarbon material containing tar acids and neutral oils, it finds its primary application in the distillation of tar such as coal tar which contains appreciable amounts of phenols and especially the lower homologues of phenol and designated collectively herein as "tar-acids".

According to the present invention tar acids are removed from the tar prior to the main distillation by volatilizing them in an atmosphere of inert gases and vapors, the vapors comprising constituents other than tar acids, of the tar being distilled. The passage of these gases and vapors in contact with the tar effects vaporization of tar acids at a temperature below their normal boiling points, and the presence of the vapors of tar constituents other than tar acids in admixture with the inert gases inhibits or represses further vaporization of such constituents from the tar. Selective vaporization of the tar acids is thus effected. The invention includes partial removal of the tar acids by such selective vaporization and is advantageously carried out in a manner to remove 50% or more of the tar acids by such selective vaporization.

The tar acids are separated from the gases and vapors resulting from the tar acid vaporization by contact with an alkaline reagent, such as an aqueous solution of caustic soda. The tar acid extraction is substantially complete or as nearly complete as is feasible in a commercial operation and all or a portion of the resulting mixture of inert gases and neutral oil vapors is recycled for further tar acid vaporization. If desired, neutral oils can be condensed from the recycled vapors either simultaneously with the tar acid extraction by cooling with the caustic solution or by separate cooling means, but condensation of neutral oil vapors is advantageously kept at a minimum at this stage of the process, and the neutral oils then recovered as distillate in the subsequent distillation of the hot residue from this tar acid extraction operation.

In the tar acid vaporization step of this process, the heated tar may be sprayed into the mixture of inert gases and neutral oil vapors or the vapors may be bubbled through a body of the heated tar or the tar and vapors may be brought into contact in a packed tower or similar device. The tar is advantageously heated to a temperature sufficient to effect volatilization of the desired tar acids without heating the recycled gases. If the recycled gases are sufficiently heated before being brought into contact with the tar, the tar need not be heated to such a high temperature.

The recycled gases are substantially free from tar acid vapors, but they contain vapors of neutral oils volatilized from the preheated tar. In commencing a tar acid vaporization of the type here referred to, steam or other inert gas is passed through a body of the heated tar. This distills not only tar acids, but also neutral oils. The resulting gases and vapors are then extracted with an alkaline reagent, e. g. caustic soda, the tar acid vapors being removed from the cycle. The vapors of neutral oils and steam or other gas employed for the volatilization are then recycled. If the tar contains moisture this is vaporized and forms steam which blends with the circulating oil vapors. A bleed-off is provided through which the make of steam and attendant oil vapors is removed. If the bleed-off is in communication with the atmosphere the pressure in

the system will be approximately atmospheric. This bleed-off is advantageously placed at a point in the cycle after the acids have been extracted by the alkaline reagent. Neutral oil vapors which are bled off with the inert gas may be condensed and added to the dephenolized tar or to the distillate obtained by distillation of the dephenolized tar or may be separately collected. As the process proceeds the inert gas originally employed will be gradually eliminated and replaced by the steam and oil vapors formed in the process. If dry tar is treated the recycled gas may consist largely of vapors of non-acid constituents of the tar. Throughout the specification I use "inert gases" to designate the gases (such as steam and neutral oil vapors of tar, used in the dephenolizing stage of the process to effect the vaporization of tar acids) which are chemically inert towards the tar and caustic soda.

The tar acid vapors are advantageously extracted by effecting contact of caustic soda solution with the mixture of gases and vapors as in a packed or grid tower or the like. The temperature and concentration of the caustic solution are preferably so regulated that a minimum of neutral oils is condensed in contact with the carbolate solution produced. If no neutral oils are condensed in the cycle the recycled gases will be brought into substantial equilibrium with the non-acid constituents of the preheated tar and after the recycled gases have once been brought into such equilibrium, there will be no further volatilization of neutral oils, except for the small amount required to compensate for the oils dissolved in the carbolate and those bled off from the system with the excess of steam to compensate for the oils flashed with the steam at the temperature of the tar fed to the dephenolizer. The process of the invention is not limited to a process in which no neutral oils are condensed from the cycle, but the extent of this condensation of neutral oils is advantageously kept at a minimum and the dephenolized residue from the tar acid extraction which is subsequently distilled then contains substantially all of the constituents of the tar except the tar acids removed and the oils removed with steam at the bleed-off. The tar acids removed will ordinarily amount to 50 to 70% of those present, but this invention is not limited to the removal of any specific percentage of tar acids. The neutral oil removed at the bleed-off ordinarily amounts to 3 to 5% of the tar but this will vary with the temperature and the kind of tar treated and the water content.

The residue from the tar acid extraction is subjected to distillation. The selective tar acid vaporization which precedes the distillation may be so co-ordinated with the subsequent distillation that the distillate is substantially free from tar acids, distilled from the tar, particularly the low boiling tar acids. If coke-oven gases are used to distill the dephenolized tar, the distillate will contain acids from the tar from the oven gases. Ordinarily the tar acid extraction will be controlled to recover from the tar a high percentage of tar acids of low boiling range, which have greater commercial value. The boiling range of the tar acids vaporized from the tar can be controlled, within certain limits, by the temperature to which the tar is heated and the rate at which the gases are circulated. By providing two or more controlled extraction steps in the cycle, different tar acid fractions can be obtained.

In the accompanying drawings, means for carrying out the invention are illustrated more or less diagrammatically. Figs. 1–3 illustrate means for distilling tar by direct contact with hot coal distillation gases. The tar may be distilled in a current of steam or hot combustion gases, for example. Figs. 4 and 5 illustrate means for distilling tar by processes in which there is no current of gases passing through the still. The heat of the undiluted vapors (and gases) resulting from the distillation is employed for preheating the tar. Fig. 6 is a modification of the apparatus shown in Figs. 1 and 2 with separate means for condensing neutral oils in the dephenolization of the tar. A similar modification may be applied to the processes illustrated in Figs. 4 and 5. In all cases the preheating of the tar by the heat of the substances coming from the still may be supplemented by heating the tar from another source either before or after it enters the heat interchanger, or by heating the recirculated gases and vapors in the dephenolizing stage. Further, the heat of the substances coming from the still may be used for heating the recycled gases before they enter the tar dephenolizer. It is intended and is to be understood that the invention is not limited to the specific application cited.

In the drawings:

Fig. 1 is a plan view of a coke oven battery equipped with a tar still and selective tar acid vaporization apparatus;

Fig. 2 is an elevation in part of Fig. 1, showing the still and tar acid vaporization apparatus;

Fig. 3 is a section through the still on the line 3—3 of Fig. 2;

Fig. 4 is an elevation of another type of still equipped with selective tar acid vaporization apparatus;

Fig. 5 is an elevation of a pipe coil still equipped with selective tar acid vaporization apparatus; and

Fig. 6 is a modified form of the apparatus of Figs. 1–3 provided with separate means for the condensation of neutral oil.

The coke-oven battery 5 of the drawings is equipped in the usual way with a collector main 6, and a cross-over main 7, which conveys the gases from the collector main to the condensers 8. The gases pass from the individual ovens of the battery through the uptake pipes 9 into the collector main 6. At the opposite side of the battery is a still 10. Hot gases from selected ovens of the battery pass through the especially provided uptake pipes 11 into the hot gas header 12. By proper manipulation of valves in the uptake pipes 11 and valves in the uptake pipes 9 which connect the opposite ends of these same ovens with the collector main 6, the flow of the gases from these selected ovens either to the collector main 6 or the hot gas header 12 is controlled.

In the still is a spray roll 13, which is driven at a high rate of speed, for example, 900 to 1200 R. P. M., by the motor 14. The spray roll dips to a slight extent into the tar and pitch in the still and sprays them in the form of a fine, intense spray up into the current of hot gases. The gases enter the still at substantially the temperature at which they leave the ovens. They are rapidly cooled by the fine, intense spray of tar and the tar is rapidly distilled.

The spray of tar and pitch in the still 10 is

so intense that the gases are cleaned; that is, fine entrained particles of pitch mist and coke, etc., are scrubbed from the gases.

The gases and vapors pass from the still into a saturator **15** where they are sprayed with the residue of dephenolized tar. Lower boiling oils are volatilized in this saturator and the liquid sprayed into the gas removes from the gas fine entrained particles of spray which are carried over from the still by the gases and vapors. The gases and vapors leaving the saturator are substantially clean and on cooling yield clean oils.

Baffles **16** are provided in the saturator to aid in the removal of spray particles and to provide a large surface of contact between the material to be distilled and the gases and vapors. The residue from the distillation in the saturator is drawn off through the line **17** and enters the still near the hot gas header. This residue is distilled in the still and pitch of desired melting point is produced. Pitch with a high melting point, for example 350° F. to 400° F., or higher, may be produced. The pitch product is drawn off from the still through the trap **18**, and the leveling arm **19**. The position of this leveling arm controls the depth of liquid maintained in the still. If a high melting point pitch is produced it may be solidified and granulated by spraying with water from the line **20**, in the trough **21**, and the granulated solid pitch which results is collected in the bin **22**. Other means for solidifying the pitch or means for collecting a softer pitch may be provided as conditions require.

The clean oil vapors and gases from the saturator **15** are cooled in the heat interchanger **25** and the tar to be dephenolized and then distilled is preheated therein. The tar enters at **26** and after being preheated in the heat interchanger it is sprayed into the dephenolizer **27**, through the spray means **28**. Clean oil which is condensed from the gases and vapors in the heat interchanger **25** is drawn off into the tank **29**. The resulting gases and vapors are sprayed with water or otherwise further cooled in the condenser **30**. A decanter **31** and an oil tank **32** are provided for decanting and collecting the condensate from the condenser **30**. An exhauster **33** is shown for drawing the gases and vapors through the system. This exhauster may be the same as the exhauster drawing the gases through the condensers **8**, or a separate exhauster may be used. Means for the recovery of light oil, ammonia, etc., may be provided. The header **12**, the still **10**, the saturator **15** and the heat interchanger **25** and connecting mains are advantageously heavily insulated.

The tar acids are extracted from the tar in the dephenolizer **27** by vaporization in a current of inert gases and vapors of neutral oils. These inert gases and vapors enter the bottom of the dephenolizer through the line **35**. The hot tar is sprayed into the gases as they pass up through the dephenolizer and tar acids are vaporized and carried from the dephenolizer in the vapor state in admixture with the inert gases and neutral oil vapors through the line **36** into the caustic extractor **37**. In this caustic extractor the tar-acid-containing gases and vapors are sprayed with caustic from the spray **38**. The caustic combines with the tar acids and phenolates (carbolates) are formed.

It is generally advantageous to spray a large volume of liquid into the gases so as to provide a large surface of contact between the liquid and the gases and vapors in the extractor and thus insure complete removal of the tar acid vapors. By recycling a portion of the carbolate a large volume of an alkaline solution is obtained which presents a large surface of contact to the gases and vapors and substantially complete removal of the tar acid vapors from the cycle results. If operating conditions are so controlled that there is but little condensation of neutral oils the carbolate is recycled by means of the pump **40** without separation of any contained neutral oils by drawing the recycled carbolate off from the bottom of the caustic extractor through the line **39**. The temperature of the recycled carbolate is controlled in the heat interchanger **40a**. Fresh caustic is introduced through the line **41**.

The make of carbolate is drawn off from the caustic extractor through the line **66** into the tank **67** and disposed of as desired. Tar acids may be recovered from the carbolates by any of the usual processes, as by treatment with carbon dioxide, etc.

If the caustic treatment is regulated to effect considerable condensation of neutral oils simultaneously with the extraction of the tar acids and to form a separate oil layer, the neutral oils and carbolate are drawn off the tower through the line **66** into the tank **67**, which is constructed to serve as a decanter, and, after separation of the neutral oils, the carbolate is recirculated to the caustic extractor through the line **68** by means of the pump **40**. Condensation such as is obtained by partial cooling of the gases within the caustic tower does not effect complete removal of the neutral oil vapors from the cycle, whereas the extraction with caustic removes substantially all of the tar acid vapors.

The excess carbolate is drawn off to the storage tank **69** through the pipe **70**. Neutral oils when present in sufficient amount that they can be separated from the carbolate are drawn off from the decanter into the receiver **71** through the line **72**. These neutral oils may be separately treated, or they may be blended with distillate from the still or they may be blended through line **73** with dephenolized tar from the dephenolizer **27** for distillation in the still **10**.

The inert gases containing vapors of neutral oils coming from the caustic tower are carried from the caustic extraction tower to the dephenolizer **27** by the blower **42** in the line **35**, for reuse in selective vaporization of tar acids from fresh tar. Any excess of inert gas is vented from the system through a bleed **35'** which is equipped with a condenser to recover neutral oils present in the gases. These neutral oils may be separately collected or they may be blended with the neutral oil distillate obtained from the still. On recycling the gases from which tar acids have been removed but which include neutral oil vapors, there results a selective vaporization of the tar acids even though some neutral oils are condensed in the cycle.

Instead of the tower type of dephenolizer here shown, any other suitable means for spraying the tar into the inert gases may be employed. A chamber equipped with mechanical spray means, for example, of the spray roll type, shown in the still **10**, may be used to advantage.

The hot dephenolized tar from the dephenolizer or the blend of neutral oil and dephenolized tar is distilled. It is sprayed into the saturator **15** through the nozzle **43**, by means of the pump **44** in the line **45**. This dephenolized tar has a low

tar acid content and on distillation yields distillate of low tar acid content. This distillate will, however, include any tar acids condensed from the coke oven gases.

In the modified form of apparatus shown in Fig. 4, an ordinary tank still 60 is shown, which is heated by direct fire in firebox 61. Dephenolized tar is fed into the still through the line 62, and pitch is drawn off through the line 63. Vapors pass from the still into the heat interchanger 64 and then into the condenser 64'. The tar enters the heat interchanger through the line 65; it may be preheated before entering the heat interchanger. It is heated in the coil in the heat interchanger and then is sprayed into the tar dephenolizer 27a through the nozzles 28a. The caustic extractor is shown at 37a. The inert gases enter the bottom of the tar dephenolizer through the line 35a and the tar-acid-containing gases pass from the top of the tar dephenolizer through the line 36a into the bottom of the caustic extractor. In the caustic extractor the tar acids are neutralized and the carbolate formed is in part recirculated and in part withdrawn from the system in the manner above described. The inert gases are recycled from the caustic extractor through the line 35a into the tar dephenolizer 27a. A blower 42a is provided to force the gases through the cycle. The gases may be heated before they reenter the dephenolizer and a suitable heater may be provided where this is desirable.

Fig. 5 shows a pipe coil still, 80, for distilling the dephenolized tar. The dephenolized tar is heated in the pipe coil, 80a, to a temperature above its initial boiling point and then flashed into the vapor box 81. The residue from the flashing operation is drawn off as pitch into the receiver 82.

The vapors from the flashing operation which are substantially free from tar acids, at least lower boiling tar acids, are cooled in the heat interchanger 83 in which they are brought into indirect contact with tar which is introduced at 84 and passes up through the heating coils 85 in the heat interchanger. The oils condensed from the vapors in the heat interchanger are collected in the receiver 86. The vapors which pass off from the heat interchanger are further cooled in the condenser 87 and the distillate which is condensed is collected in the receiver 88.

The tar which is heated in the coil 85 passes through the line 89 to the dephenolizer 27b. A caustic tower 37b is provided as in the preceding examples for the recovery of carbolate, and also neutral oil if desired. Mains 35b and 36b and blower 42b are provided for the recirculation of inert gas through the dephenolizer and caustic tower. The operation of the dephenolizer and caustic tower may be regulated as described in connection with the preceding figures to produce only tar acids or both tar acids and neutral oil. The dephenolized tar drawn off from the dephenolizer is pumped by means of the pump 44b to the pipe coil still for distillation and the production of pitch and distillate oil.

The apparatus of Fig. 6 is a modified form of the apparatus shown in Figs. 1, 2 and 3. A still 10c is shown into which hot gases such as hot coal distillation gases or steam or hot combustion gases are introduced through the main 12c. Pitch is drawn off through the levelling arm 19c into the bin 22c. Hot gases and vapors from the still pass through the saturator 15c to the heat interchanger 25c where they are partially cooled and then through the condenser 30c where they are further cooled. An exhauster 33c is provided for drawing the gases and vapors through the system. Higher boiling distillate oils are condensed in the heat interchanger and drawn off into the storage tank 29c. Lower boiling oils are drawn off from the condenser 30c into the collector tank 31c. These lower boiling oils are substantially free from tar acids except where hot coal distillation gases or other tar-acid-containing gases are introduced into the still through the main 12c to effect the distillation. The higher boiling oils from the heat interchanger may or may not be free from tar acids depending upon the boiling range of the tar acids removed from the tar in the dephenolizer 27c, the extent of the distillation of the dephenolized tar, and the boiling range of the cuts made.

Tar is introduced into the heat interchanger 25c through the line 26c and is heated by indirect heat interchange with the hot gases and vapors passing through the heat interchanger. The heated tar is then introduced into the dephenolizer where tar acids and neutral oil vapors are vaporized in a current of inert gas which is recirculated through the dephenolizer 27c, the caustic tower 37c and the condenser 90 by means of the blower 35c. Tar acids and neutral oil vapors are vaporized from the tar in the dephenolizer 27c. These gases and vapors are extracted by an alkaline reagent in the extractor 37c where the tar acid vapors are neutralized and separated from the gas stream. The temperature and concentration of the caustic or other alkaline reagent used are preferably so regulated that there is no substantial cooling of the gas stream by the caustic and consequently a minimum condensation of neutral oils. The carbolate produced is in part recirculated and the excess carbolate is drawn off to storage in the tank 67c.

From the caustic extractor the gas stream passes through the condenser 90. The gases are partially cooled in this condenser and a portion of the neutral oil vapors are separated from the gases. The balance of the neutral oil vapors are returned with the inert gas and recirculated through the dephenolizer to effect further distillation of tar acids and neutral oils but due to the presence of the neutral oil vapors in the recirculated gases the vaporization is a selective vaporization in which the ratio of tar acids to neutral oils vaporized is greater than the ratio in the distillate from straight distillation. The neutral oil condensed in the condenser 90 is drawn off to storage in the receiver 91.

The dephenolized tar from the dephenolizer 27c is sprayed into the saturator 15c through the spray nozzles 43c and partially distilled and the residue is further distilled in the still 10c. If pitch of low melting point is produced the pitch will be a dephenolized pitch and differ from pitch produced by the straight distillation of tar by the absence of the tar acids removed from the tar in the dephenolizer. If the distillation is carried to pitch of higher melting point, that is to a point where all of the oils of the boiling range usually accompanying the tar acids removed in the dephenolizer are vaporized, the pitch produced will be of substantially the same composition as pitch produced by straight distillation without previous dephenolization. The distillate, however, differs from ordinary distillate

in that tar acids and a portion of the lower boiling neutral oils are separately collected in caustic extractor **37c** and the condenser **90** before the tar enters the still.

Where a distillate of suitable boiling range is produced by any of the previously described applications of the invention it may be used as creosote oil. Road tars, roofing pitch, etc., may be produced directly in the still by this process.

The process is applicable generally to tar stills of various types. The distillate may be condensed in a single fraction or a fractional condensing system may be employed to recover several fractions. The heat of condensation from one or more of the fractions may be employed to preheat the tar and this preheating may be supplemented by heating from some other source. The process may be employed for the treatment of coal tars such as coke oven tar or gas house tar or blends of tars or tar fractions; it may be employed for treatment of low temperature tars; it may also be employed for the treatment of tar-acid-containing oils. Various modifications and adaptations of the process and apparatus may be made all within the scope of this invention.

This application is in part a continuation of my application Serial No. 383,859, filed August 6, 1929.

I claim:

1. The method of distilling tar-acid- and neutral-oil-containing hydrocarbon material by first dephenolizing the material and then distilling the dephenolized residue, which comprises preheating the hydrocarbon material by heat interchange with hot vapors or a mixture of hot gases and vapors resulting from the distillation of the dephenolized material, selectively vaporizing tar acids from the preheated material in a current of inert gases comprising neutral oil vapors, extracting tar acids from the resulting gases and vapors and then subjecting the dephenolized residue to distillation.

2. The method of distilling tar which comprises preheating coal tar, selectively vaporizing and carrying off tar acids therefrom in a current of inert gases and neutral oil vapors, and then distilling the residue remaining after the selective vaporization of tar acids to produce a distillate of low tar acid content.

3. The method of distilling tar by dephenolizing the tar and then distilling the dephenolized residue, which comprises preheating coal tar by heat interchange with hot vapors or a mixture of hot gases and vapors, selectively vaporizing tar acids from the preheated tar in a current of inert gases and vapors of neutral oils, extracting tar acids from said gases and vapors, subjecting the dephenolized residue to distillation thereby obtaining hot vapors or a mixture of hot gases and vapors and employing them as the vapors or mixture of gases and vapors in the aforesaid preheating step.

4. The method of distilling tar by first dephenolizing it and then distilling the dephenolized residue which comprises heating the dephenolized residue above its initial boiling point, flashing the heated residue, separately collecting the vapors from the flashing operation, preheating coal tar by bringing it into heat interchange relation with the vapors whereby the vapors are cooled and distillate is condensed and then vaporizing tar acids from the heated tar in a current of inert gases which includes neutral oil vapors to form the dephenolized residue.

5. The method of distilling tar by dephenolizing the tar and then distilling the dephenolized residue which comprises passing a current of hot gases through a tar still, spraying the dephenolized residue into the hot gases, whereby distillation is effected, bringing the tar to be distilled into heat-interchanging relation with the gases and vapors resulting from the distillation of the residue whereby the tar is preheated, selectively vaporizing tar acids from the heated tar in a current of neutral oil-containing inert gases and extracting tar acids from said gases and distilling the residue from this treatment in the current of hot gases in the tar still.

6. The method of distilling tar by dephenolizing the tar and then distilling the dephenolized residue, which comprises passing hot coal distillation gases through a tar still, spraying dephenolized tar into the tar still in the form of a fine intense spray, whereby the coal distillation gases are substantially detarred and the dephenolized tar is distilled, bringing the tar to be distilled into indirect heat-interchanging relation with the gases and vapors coming from the tar still, whereby the tar is preheated, selectively vaporizing tar acids from the preheated tar in an atmosphere of inert gases and neutral oil vapors, and distilling the residue from this treatment in the tar still.

7. The method of distilling tar which comprises passing a current of hot gases through a tar still, preheating tar by bringing it into indirect heat-interchange relation with the gases and vapors coming from the tar still, bringing a current of hot inert gases containing neutral oil vapors into direct and intimate contact with the heated tar, whereby tar acid oils are vaporized therefrom, spraying the resulting inert gases and admixed vapors with a solution containing caustic soda in an amount sufficient to neutralize substantially all of the tar acid vapors, recycling the resulting inert gases and neutral oil vapors and bringing them into direct and intimate contact with further preheated tar, and then distilling the tar residue from which tar acids have been vaporized by introducing it into the tar still and spraying it into the gases passing therethrough.

8. The method of distilling tar which comprises heating tar in a still, bringing the tar to be distilled into indirect heat interchange relation with the resulting hot vapors, bringing the preheated tar into direct and intimate contact with hot inert gases containing neutral oil vapors, whereby tar acids are vaporized from the tar, removing the tar acids from the resulting mixture of gases and vapors by bringing them into direct contact with a neutralizing agent, further vaporizing tar acids from additional heated tar by recycling the resulting gases and vapors of neutral oils in contact therewith, and distilling the tar residue from which tar acid vapors have been vaporized by heating in the still.

9. In combination with a tar still, a preheater through which vapors from the tar still pass, means for bringing tar into heat-interchanging relation with the vapors in the preheater, a dephenolizer, means for passing gases through the dephenolizer, means for bringing the preheated tar into direct and intimate contact with gases in the dephenolizer, a caustic absorber, means for passing the gases from the dephenolizer to the caustic absorber, means for returning the gases coming from the caustic absorber to the

dephenolizer and means for withdrawing the treated tar therefrom and introducing it into the tar still.

10. In combination with a tar still, means for introducing hot gases thereto, a heat interchanger, means for bringing gases and vapors from the tar still into indirect heat interchange relation with tar therein, a dephenolizer, means for passing gases through the dephenolizer, means for bringing the heated tar into direct and intimate contact with gases in the dephenolizer, a caustic absorber, means for passing the gases from the dephenolizer into the caustic absorber, means for recycling gases from the caustic absorber thereto, and means for withdrawing tar therefrom and introducing it into the still.

11. In combination with a coke oven, a still, means for passing hot coal distillation gases from the oven through the still, a heat interchanger for bringing tar into indirect heat interchanging relation with the gases and vapors coming from the still, a tar tower with means for spraying heated tar from the heat interchanger therein, a caustic absorber, means for spraying caustic therein and withdrawing carbolate therefrom and for recycling a portion of the carbolate through the spraying means, means for recycling gases through the tar tower and the caustic absorber, and means for withdrawing tar from the tar tower and introducing it into the still.

12. The method of treating tar to separately recover tar acids and neutral oils, which comprises distilling the tar in a current of inert gases including neutral oil vapors, extracting tar acids from admixture with neutral oil vapors and cooling to condense oils, using in the inert gas distillation of the tar at least a portion of the neutral oil vapors remaining after the condensation and tar acid extraction, drawing off the residue from the inert gas distillation and distilling it.

13. The method of treating tar, which comprises distilling the tar in a current of inert gases including neutral oil vapors, extracting tar acids from the resulting gases and vapors, cooling the resulting gases and vapors to condense vapors of neutral oils, using at least a portion of the neutral oils remaining in vapor form for the inert gas distillation of the tar, withdrawing the residue from this distillation and subjecting it to further distillation whereby neutral oils are distilled.

14. In combination with a tar still, a tar-acid still, a caustic tower, a condenser, means for cycling gases from the tar-acid still to the caustic tower, from the caustic tower to the condenser, and from the condenser to the tar-acid still, means for bringing a changing surface of tar into contact with the gases in the tar-acid still, means for producing a spray of caustic solution in the caustic tower, and means for withdrawing distillation residue from the tar-acid still to the tar still.

15. The method of distilling tar and of recovering tar acids therefrom, which comprises heating the tar to a temperature below the distillation temperature of the tar acid oils, passing inert gases into intimate contact with the tar to vaporize tar acid oils at a temperature below their normal boiling point, passing the resulting admixed gases through a hot alkaline solution to recover tar acids therefrom, and, after the treatment of the hot tar to remove tar acids, further heating the tar to distill the same and produce distillate oils and a pitch residue therefrom.

16. The method of continuous distillation of tar and of extracting tar acids therefrom, which comprises preheating the tar to a temperature below that of distillation of tar acid oils, passing in intimate contact with the tar a current of hot inert gases to effect vaporization of tar acid oils, passing the resulting admixed gases and vapors into intimate contact with hot alkaline solutions to extract tar acid vapors therefrom, returning the inert gases recirculating the same through the tar for further extraction of tar acid oils, etc., and, after the treatment of tar for the extraction of tar acids therefrom, further heating the tar continuously to effect distillation thereof and the production of distillate oils and a pitch residue.

17. The method of distilling tar and of recovering tar acids therefrom, which comprises selectively vaporizing tar acids from the tar by contact with neutral oil vapor, separating the resultant vapor mixture from the residue remaining after the selective vaporization of tar acids, and then distilling the residue to produce a distillate of low tar acid content.

18. The method of distilling tar-acid and neutral-oil-containing hydrocarbon material and of recovering tar acids therefrom, which comprises selectively vaporizing tar acids from the hydrocarbon material by contact with a gaseous mixture substantially free from tar-acids but containing sufficient vapors of neutral oil constituents of the material treated boiling within the boiling range of the tar-acids vaporized to inhibit to a substantial extent distillation of said constituents from the hydrocarbon material, separating the resultant vapor mixture from the residue remaining after the selective vaporization of tar acids, and then distilling the residue to produce a distillate of low tar acid content.

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