

July 25, 1933.

S. P. MILLER

1,920,097

DISTILLATION OF TAR, ETC

Filed Dec. 18, 1928

6 Sheets-Sheet 1

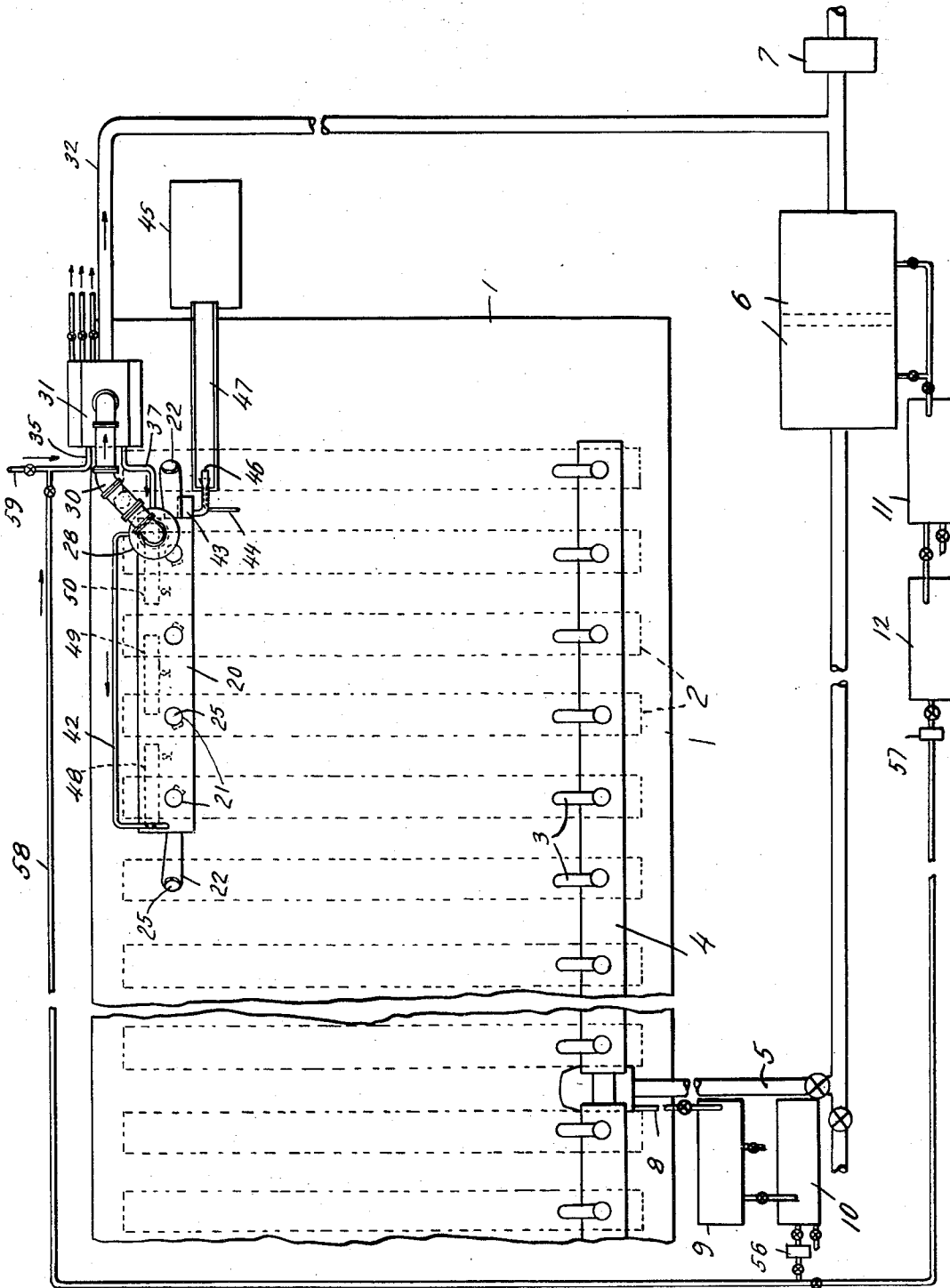


Fig. 1.

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6 Sheets-Sheet 2

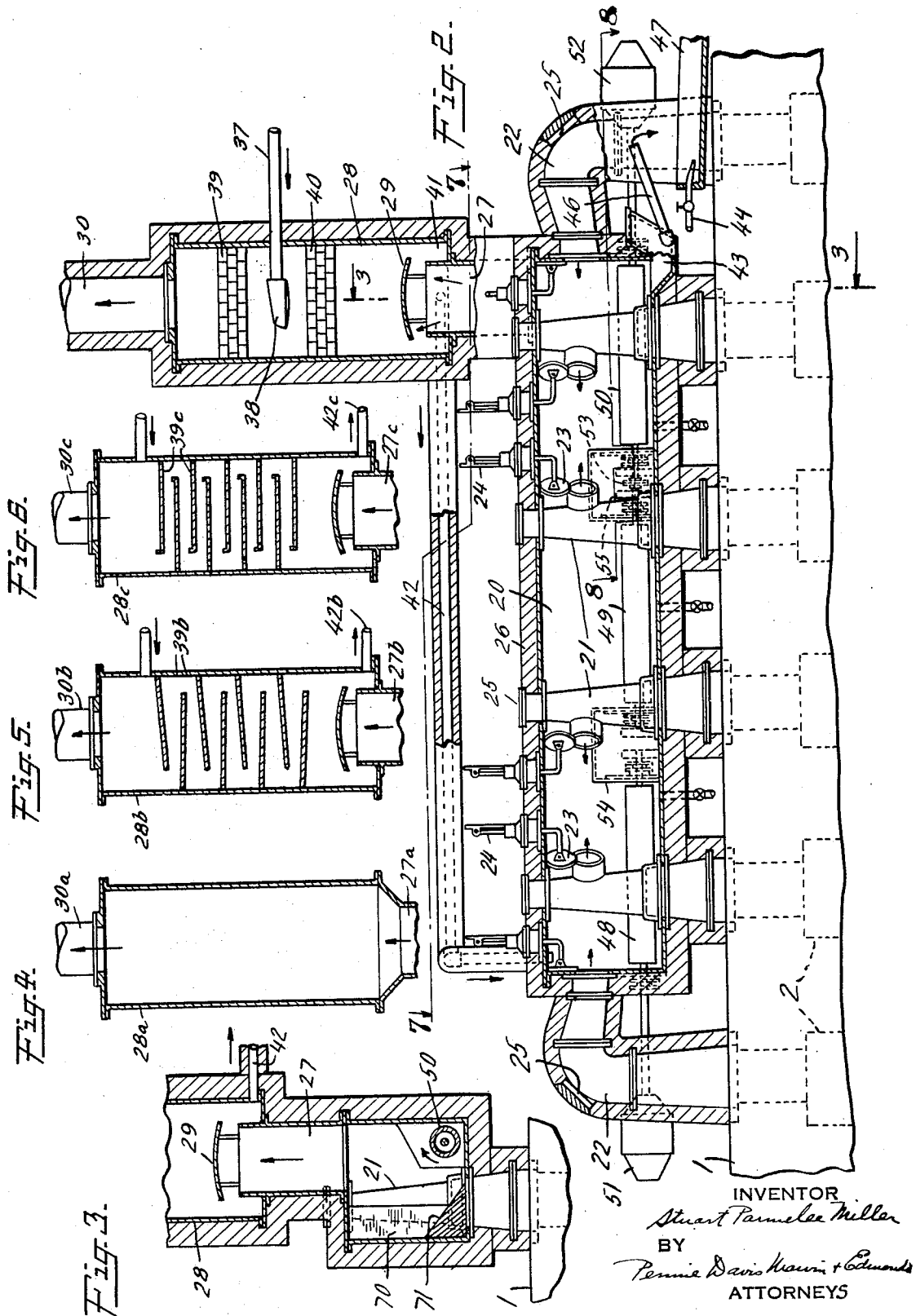


Fig. 2.

Fig. 9.

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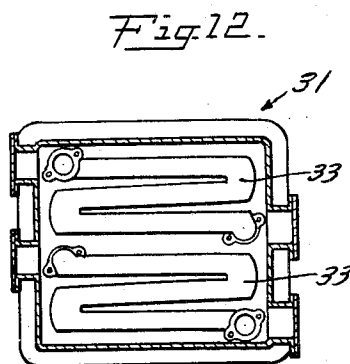
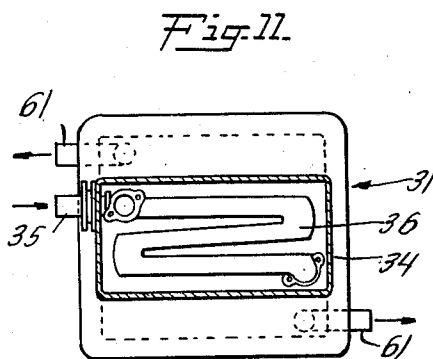
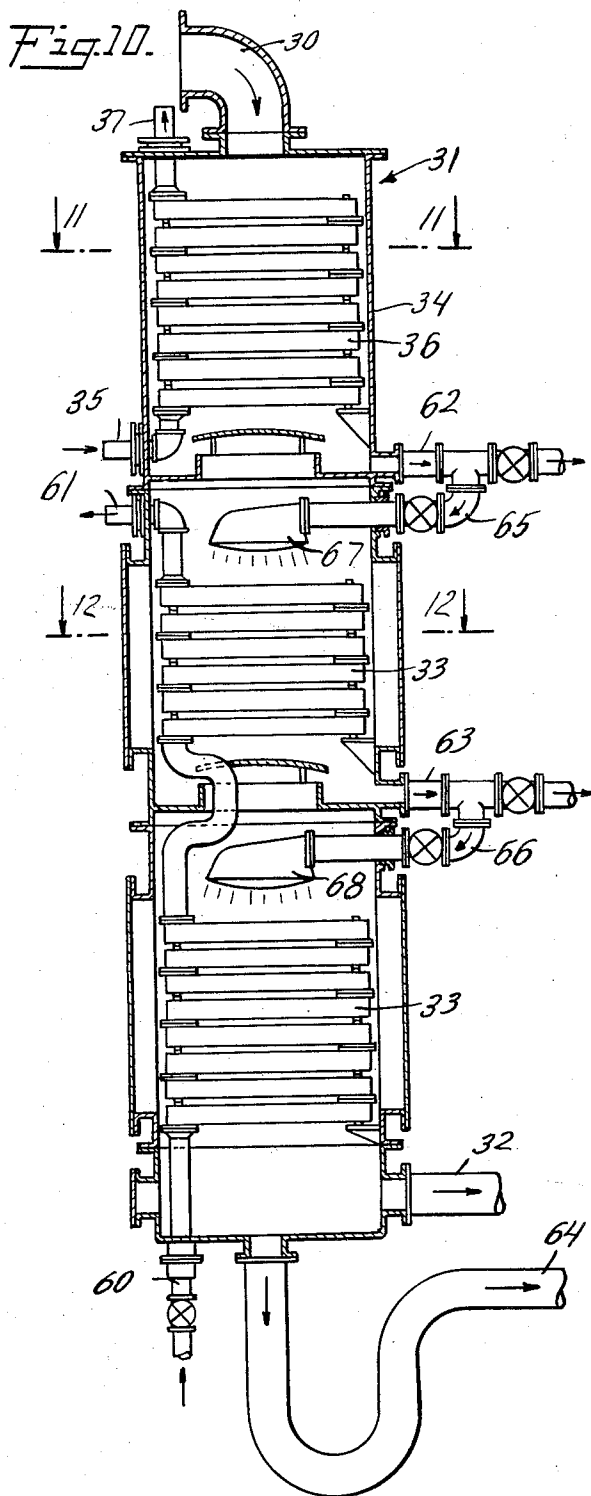
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Filed Dec. 18, 1928

6 Sheets-Sheet 4



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1,920,097

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Fig. 13.

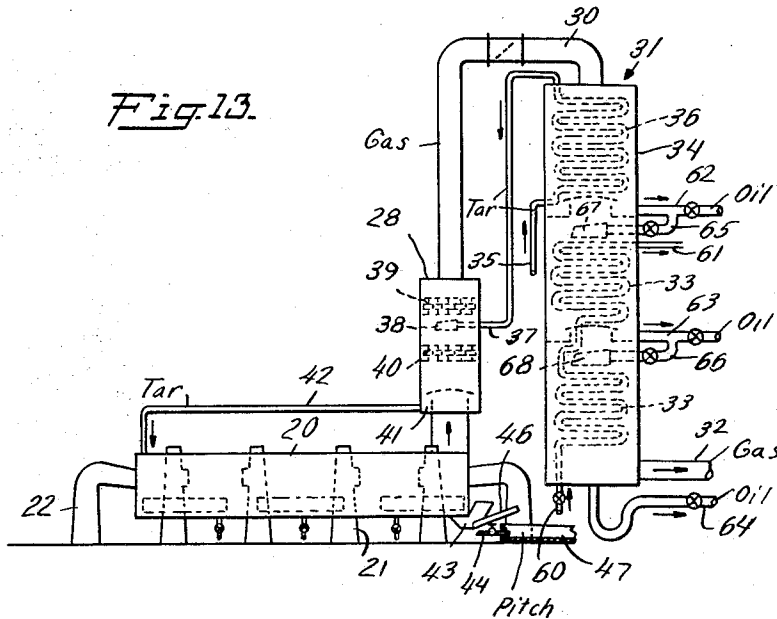


Fig. 14.

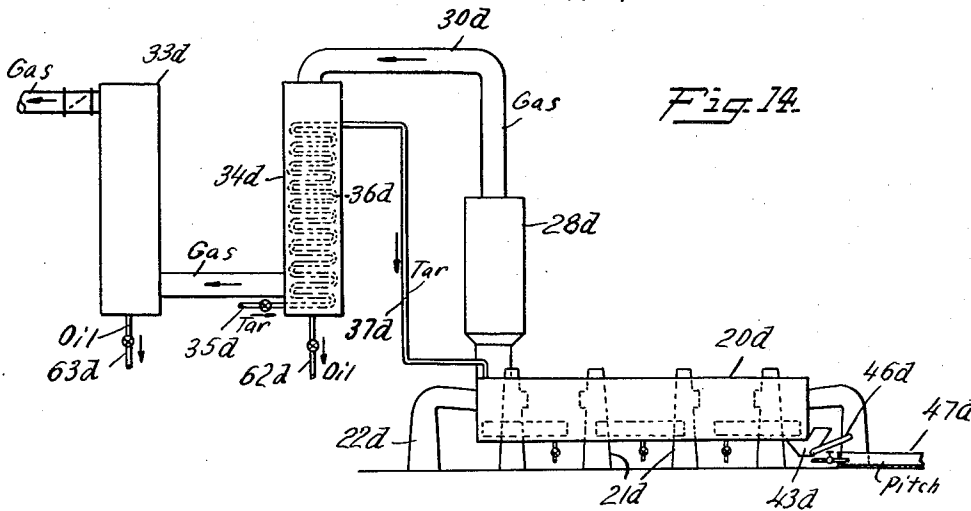
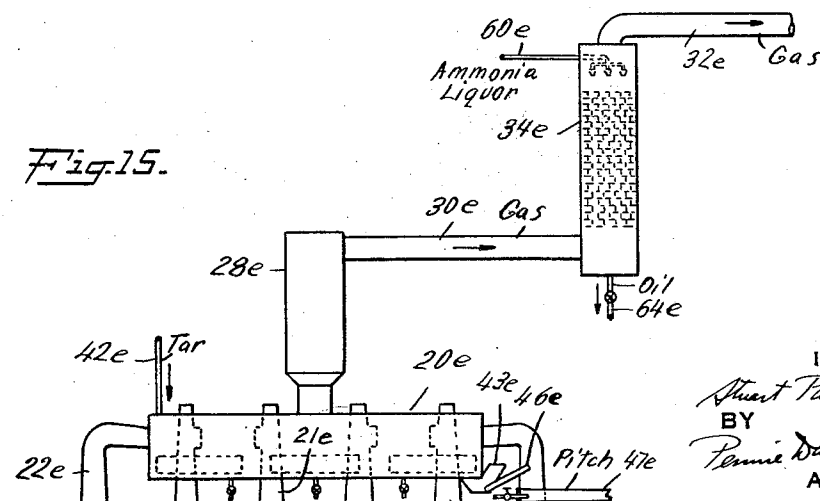


Fig.15.



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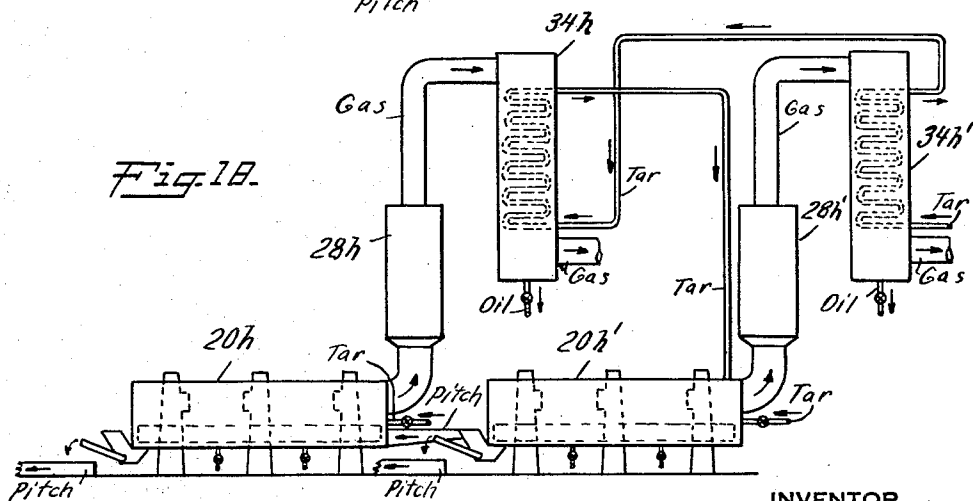
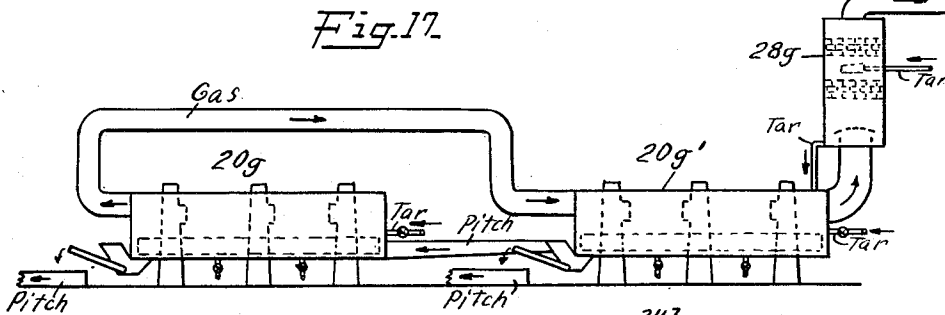
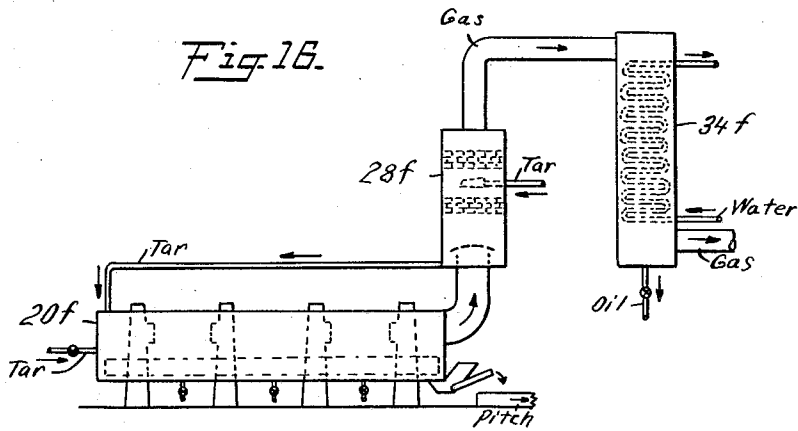
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DISTILLATION OF TAR, ETC

Filed Dec. 18, 1928

6 Sheets-Sheet 6



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UNITED STATES PATENT OFFICE

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DISTILLATION OF TAR, ETC.

Application filed December 18, 1928. Serial No. 326,769.

This invention relates to improvements in the distillation of hydrocarbons such as tar, tarry oils, pitch, etc., and includes an improved process and apparatus for such distillation.

The present invention provides an improved process and apparatus whereby tar can be distilled for the production of high melting point pitch and distillate oils therefrom, with considerably higher yields of distillate oils from the tar distilled than has heretofore been obtained, so far as I am aware, by any distillation method previously employed.

The process of the present invention is a continuous process in which the tar to be distilled is heated by direct contact with highly heated gases, in which the tar is atomized or sprayed into such gases in such volume and with such intensity and uniformity and thoroughness of distribution that the tar is rapidly heated and distilled, the superheated gases quickly cooled to a temperature considerably below their initial temperature, the gases effectively freed from entrained pitch and carbon content, and in which the rate of supply of tar to be distilled and of superheated gases for the distillation is so regulated that high melting point pitch can be directly and continuously produced and withdrawn, and so that an unusually high yield of clean oil can be recovered from the distillation by withdrawing the admixed gases and vapors while still at a high temperature and cooling the same to condense the distillate oils therefrom.

The present invention provides a particularly advantageous process for the distillation of coke oven tar at a coke oven plant, and enables such tar to be distilled with the production of an unusually high yield of distillate oils, for example, around 75% or more of the tar distilled, and a high melting point pitch, for example, having a melting point around 400° F. or higher. The present process also presents advantages for the distillation of tar where the highest melting point pitch and the maximum oil yield are not to be obtained, and enables tar to be distilled to produce lower melting point

pitches and a lower percentage oil yield with many advantages, including thorough scrubbing and cleaning of the gases, almost instantaneous cooling of the gases to a temperature approaching that of the tar or pitch being distilled, rapid and effective heating of the tar or pitch to a temperature approaching that of the gases, and utilization of practically the maximum distillation capacity of the gases down to the temperature to which they are cooled in the still.

The improved process, as carried out at such coke oven plants, makes use of the hot coke oven gases by introducing them continuously, and at or near their maximum temperature as they leave the individual coke ovens, into a still. The material to be distilled, e. g., tar or partially distilled tar, is continuously introduced into the still and caused to flow therethrough, and atomized or sprayed into the hot coke oven gases with such volume, intensity and uniformity of spraying that the coke oven gases are immediately cooled from their maximum temperature to a materially lower temperature, with simultaneous heating of the tar or pitch and rapid distillation thereof and with such thorough scrubbing of the hot gases as will free them from all or a greater part of their entrained carbon and pitch particles. The resulting admixed coke oven gases and vaporized oils are continuously withdrawn from the still while at a high temperature and subsequently cooled to condense the oils therefrom; and the pitch produced is also continuously withdrawn from the still.

Highly heated gases, such as those coming from coke ovens, leave the incandescent upper portions of the ovens at a high temperature, e. g. around 550° to 800° C. or higher, depending upon the construction of the coke ovens, the coking cycle, the degree of coking, etc. Such highly heated gases are at a temperature above that at which coking of pitch will occur; but such coking is practically eliminated in the present process by subjecting the hot gases immediately, as soon as they enter the still, to contact with an excessively large volume of spray of tar or pitch which is at a considerably

lower temperature and which has adequate capacity for absorbing the heat of the gases as sensible heat and as latent heat. The effect of an excessive and intensive showering or spraying of tar or pitch into the gases is to suddenly and rapidly cool the gases to a temperature considerably lower than their initial temperature while at the same time rapidly but uniformly and in controlled manner heating the tar or pitch and distilling vaporizable constituents therefrom. In this way, the gases are rapidly cooled to a temperature such that objectionable coking can readily be avoided, while the high temperature and heat content of the highly heated gases is nevertheless employed in a particularly advantageous manner for the rapid distillation of materials such as tar, pitch, tarry oils, etc.

The use of such highly heated gases has the further advantage that the gases, even after being cooled by distillation of tar and pitch, may still be at a high temperature, for example, when making 400° F. melting point pitch, around 350 to 400° C. and can be withdrawn from the still at such high temperatures, with advantages which will hereinafter be pointed out. The scrubbing of the highly heated gases is so effectively carried out that the gases themselves are thoroughly cleaned of entrained dust, pitchy material, etc. by the scrubbing operation, and the distillate oils subsequently condensed from the gases are free or substantially so from such pitchy and other extraneous constituents, these constituents being scrubbed from the gases and added to the pitch residue.

The present invention provides an improved process for distilling oils, tars, tarry oils, pitches, etc., from various sources. In the case of tars, the tars may be coke oven tars, gas retort tars, water gas tar, or tars from other sources. Tarry oils or other oils can be similarly and advantageously distilled, particularly those which on distillation leave a fluid residue and those which when distilled by the usual methods are subject to a high degree of decomposition with attendant loss in oil yield. In the case of coke oven tar, for example, the tar may be the total tar produced at the coke oven plant, or it may be the heavy tar separated in the collector main, or the light tar or tarry oil separated in the condensers. With coke oven tar, distilled at a coke oven plant, and with the use of hot coke oven gases, the gases employed for the distillation, after the cooling thereof to separate distillate oils therefrom, can be combined with the remainder of the gas produced at the coke oven plant.

While the highly heated gases employed for the distillation may be obtained from various sources, including gas retorts, gas

producers, water gas machines, and other coal carbonization, distillation and gasification processes, the process is particularly advantageous when carried out with hot coke oven gases which are available in large volume at coke oven plants, and which contain a large amount of heat which is commonly wasted in the ordinary operation of such plants.

The gases from horizontal gas retorts leave the retorts at temperatures of 500 to 700° C. or even much higher and may be used to advantage for distillation of the tar normally collected from the gases on cooling. Likewise producer gas leaves the gas producer at 500 to 800° C. more or less, and water gas which leaves the machines at 600° to 800° C. are of value for use by my process. These and other highly heated gases are employed in my process for distilling tar, tarry oils, pitches, etc. in a particularly advantageous manner.

The apparatus employed, according to the present invention, includes a still provided with means for introducing the hot coke oven or other highly heated gases, with means for introducing the tar or pitch or other product to be distilled, with means for withdrawing the distillation residue produced, and with means for withdrawing the admixed gases and vapors from the still and for cooling the same to separate the distilled oils, etc. therefrom. If advantage is to be taken of the maximum distillation capacity of the gases the still should be so located that the hot coke oven gases can enter it at a temperature which is not greatly below that at which they leave the individual coke ovens. The still is therefore advantageously placed on top of the coke oven battery and with direct communication from the required number of individual ovens into the still, so that the gases from the ovens enter the still without material drop in temperature. The hot gases can however be led to the still at another or different location. Any moderate drop in temperature of the gases can be compensated for by use of added quantity of the highly heated gases. The volume and composition of the coke oven gases given off from adjacent ovens varies, the volume of gases being greater at the beginning of the coking period, after the oven is charged, and being considerably less near the end of the coking period before the oven is pushed. Adjacent ovens are commonly charged at different times so that, for example, at the time one oven is charged, the adjacent oven on one side will be well advanced in coking period, and the adjacent oven on the other side will also be well advanced, but to a different extent. Accordingly, by connecting gas off-takes from the individual ovens so that as the gases from them enter the still, the varia-

tions in the amount, composition and temperature of the gases from any particular oven with the progress of the coking cycle of that oven, will be equalized with the gases coming from the other ovens and an approximately uniform volume of gases of approximately uniform average composition can be continuously obtained for the distillation.

The individual coke ovens are commonly connected, at one end, with a collector main by individual uptake pipes leading from the individual ovens to the main. The still employed in the present process can advantageously be located at the other side of the coke oven block and connected with the requisite number of the ovens through individual connecting or uptake pipes so that part or all of the gases from such ovens can be drawn into the still and employed for distillation. The location of the still can vary, depending on the construction of the coke oven plant, the location of the collector main or mains, etc. Instead of locating it immediately above the coke ovens and connecting the individual ovens separately to the still, the still can be located away from the ovens, at one end or side of the block, and connected by a heavily insulated header with the requisite number of ovens, as more fully disclosed and claimed in my companion application, Ser. No. 326,770, filed December 18, 1928. In general, when the gases from the ovens are employed for distillation, these ovens are valved off from the ordinary collector main so that all of the gases from those ovens pass through the still and the gas handling system connected therewith. It will be evident that a greater or lesser number of individual ovens can be connected to the still, depending upon the amount of tar to be distilled, and other considerations.

In order to take full advantage of the high temperature and heat content of the superheated gases, e. g. to attain maximum distillation capacity and high melting point pitch, it is important to provide the still with heavy insulation and to as well as possible avoid cooling of the gases after they leave the coke ovens and before they enter the still. Heat losses can be reduced to a minimum by providing the still and connecting pipes with heavy insulation.

The tar to be distilled may be coal tar containing the usual small percentage of water. Due, however, to the high sensible and latent heat of water as compared with coal tar oils and due to the low boiling point of water, the distilling capacity of a definite amount of the hot gases can be greatly increased by preheating the tar to remove water from it before it enters the still, for example, to a temperature around but preferably somewhat higher than 100° C.; and the distillation capacity can be still further materially

increased by preheating the tar to a higher temperature in order to supply from low temperature sources as much as possible of the latent and sensible heat required for distilling the oils of low and medium boiling points. For example, an increase in preheating of the tar from 100° C. to 200° C. before admitting it into the still resulted in an increased capacity of distillation for unit quantity of hot gas of about 40% to produce pitch of the same melting point (around 400° F.); and the distillation capacity of the gases per thousand cubic feet was increased from about 7.8 gallons of tar to about 10.8 gallons of tar. The following examples indicate the distillation capacity of hot gases with tar added at various temperatures. The tar when preheated to higher temperatures was preheated by the hot enriched gases leaving the still. The hot gases in each case entered at about 650° C. and the tar used contained about 2% water by volume. The distillation capacity is expressed as gallons of tar per thousand cubic feet of gas (the volume being calculated to standard conditions). Pitch of 400° F. melting point is made in each case.

Tar temperature	Distillation capacity
° C.	Gallons
70	7
100	7.5
150	9.4
200	11.7
250	16.3

It will be understood by those skilled in the art that by utilizing low temperature heat, e. g. preheat for supply of low temperature sensible and latent heats, the high temperature heat of the hottest gases may be conserved for supply of the high temperature latent and sensible heats required for production of high melting point pitch and high oil yields; the distillation capacity of the gases may as shown be greatly increased by preheating the tar or other product to be distilled.

The preheating of the tar, before introducing it into the still, can advantageously be accomplished by heat interchange with the hot gases and vapors after they leave the still. The unusually high temperature of these gases and vapors is such that a very important preheating of the tar can be accomplished at the same time that the gases and vapors are themselves cooled and part of the heavier oil constituents condensed therefrom. Other sources of heat may of course be utilized if desired either alone or in addition to the heat from the distillation gases for preheating the tar.

A particularly advantageous method of supplying the tar is to preheat it and then

bring it into direct contact with the hot gases and vapors leaving the still. Accordingly, by bringing preheated tar into intimate contact with the escaping gases and vapors, the tar can be partially distilled, and the percent concentration of oil vapors in the gases increased without condensing and removing from the admixed gases and vapors any substantial amount of the heavier vapor constituents carried thereby. The thus preheated and partially distilled tar can then be supplied to the still. By operating in this way, the distillation capacity of the still can be greatly increased, and the percentage of oil vapors in the escaping gases and vapors can also be materially increased by such preheating of the tar and introduction of the preheated tar into contact with the hot gases and vapors leaving the still. For any particular pitch melting point the temperatures of the gases leaving the still will vary somewhat depending on the degree of preheat imparted to the tar brought in contact with said exit gases but even with relatively low degree of preheat the exit gas temperature is still sufficiently low as to permit no difficulties in operation.

It is important, in carrying out the present process to provide for a rapid and intensive showering or spraying or atomizing of the tar or pitch into the hot gases so that the gases will be brought into immediate, continual and thorough contact with the spray and so that all parts of the still will be thoroughly flushed with liquid pitch and the walls kept clean from coke deposit. It is important that there shall be no local overheating of any portion of the still and that to avoid such overheating at all times there shall be in atomized form within the still a sufficient quantity of tar or partially distilled tar to absorb the heat of the hot gases by being itself progressively and in regulated manner distilled to pitch of the desired melting point. It is important to avoid a deficiency of pitch or of partially distilled tar on any and all exposed surfaces of the distillation apparatus since such a deficiency would result in local over distillation and the formation of coke, because of the high temperature of the highly heated gases. Consequently, all interior surfaces of the distillation apparatus should be continuously and intensively supplied and washed with the pitch or partially distilled tar. The same thorough and abundant scrubbing of the hot gases with the tar or partially distilled tar, which results in the cooling of the gases and the rapid distillation of the tar, also serves to keep all parts of the still surfaces flushed with partially distilled tar or pitch and prevent local overheating and coking thereon. It is of importance, for most satisfactory operations, to have the still of such size and shape that

large quantities of spray reach all interior surfaces. It is of equal importance that interior fittings such as uptake pipes, valve mechanisms, etc., be so arranged that all exposed surfaces are at all times heavily flushed with the pitch spray.

This thorough and abundant spraying of the gases with the tar or pitch is accomplished by suitable mechanical devices of such a character and so located and operated that the tar or partially distilled tar will be projected into the hot gases and efficient subdivision of the tar accomplished. This is important, not only to provide a large surface of evaporation and distillation, but also to provide sufficient tar in the gas space of the still to absorb the heat of the hot gases therein, to keep the interior surfaces of the still flushed with the tar or partially distilled tar, to prevent objectionable coke formation and to adequately wash or scrub the gases to remove pitchy or carbon particles from them. Various mechanical devices can be employed to accomplish these objects. In general, they should dip into the tar or pitch in the bottom of the still and, by rapid rotation or other motion, cause the tar or pitch to be intensively sprayed in large quantities up into the gases, and into all parts of the still and against all exposed surfaces. One satisfactory atomizing device comprises a horizontal roll, or a plurality of rolls, or elongated cylinders, rotating rapidly, and with the cylindrical surface dipping into the tar or pitch at the bottom of the still, and rotating at such a rate that it will spray the tar and pitch into the gas space of the still. Such a roll may be a cylindrical roll, with smooth surfaces, or it may have circumferential grooves or ribs or disc-like projections.

The invention is not limited to any particular mechanical atomizing device, in its broader aspects, but includes any suitable atomizing means that will insure sufficiently thorough agitation of the tar or pitch and atomizing or spraying of the same into the gases to keep the still walls flushed with the tar or pitch and to accomplish the sudden cooling of the gases and the rapid distillation hereinbefore referred to as well as to thoroughly scrub the gases. An atomizing device of generally cylindrical contour, such as a cylindrical roll, either smooth or with circumferential grooves or disc-like projections, is particularly advantageous, since, with such spray devices, the distillation can be readily carried out with a minimum of difficulty from the formation of coke, and with the production of a high melting point pitch and an unusually high percentage of distillate from the tar distilled. It will be understood that the pitch is repeatedly thrown up into the gases and over exposed still surfaces so that in its progress through

the still any particular unit of pitch may be thrown into the gases a great many times. In this way a high degree of intimacy between gases and pitch and a regular and controlled increase in temperature of the pitch is accomplished. These various factors contribute to the highly satisfactory operation of the system as a whole.

With a still connected individually to a plurality of different coke ovens, the hot coke oven gases will enter the still at a corresponding number of points. The tar will ordinarily be admitted at one end of the still and the high melting point pitch withdrawn from the other. If the outlet for the gases and vapors is located near the middle of the still, the flow of gases and of tar at one end of the still will be in a general concurrent direction, and in the other end of the still in a general countercurrent direction. If the outlet for the gases and vapors is near one end of the still, the pitch may be withdrawn from the same or opposite end, and the tar added at the end opposite the pitch outlet. The general flow of the gases and tar will then either be concurrent or countercurrent. Each of these methods of operation has certain advantages and limitations.

When the flow of gases and of tar and pitch is in a general concurrent direction, the hot gases from the successive individual ovens will enter the still in contact with tar, partially distilled tar and pitch of increasing melting point and the gases and vapors will leave the still in contact with the final pitch. In this case, the temperature of the exit gases and vapors will be relatively high and the distillation capacity of the hot gases lower than with a general concurrent flow.

With a general countercurrent flow of the gases and pitch, the hot gases from the individual ovens first come into contact with hot more or less completely distilled material and flow in a direction opposite to that of the tar and pitch being distilled and leave the apparatus in contact with the incoming tar of relatively low temperature. In this case, the temperature of the exit gases and vapors will be lower than with concurrent flow and the distillation capacity of the hot gases will be relatively higher. With concurrent flow the final pitch will leave the still at a lower temperature than in case of countercurrent flow.

When producing pitch of as high melting point as 400° F. or higher, care must be taken since there is considerable danger of the production of coke unless the process is properly regulated. It requires only a relatively small degree of improperly regulated distillation to convert high melting point pitch of about 400° F. melting point into what is commonly termed "coke" and, to avoid such conversion, local overheating

should be avoided and a proper and well regulated balance maintained between the quantity of gas employed for the distillation, the quantity of tar being distilled, and the intensity of atomization of said tar or pitch into the gases.

It will be evident therefore, that various factors of the process require consideration and should be so regulated with respect to each other as to accomplish the desired distillation. The highly heated gases employed may vary in their temperatures and distillation capacity as well as in the volume of gases available. In order to obtain maximum distillation of the tar, with the production of a high yield of distillate oils, and a pitch of high melting point, while avoiding objectionable coking of the pitch produced, it is important to regulate carefully the amount of tar and pitch contacted with a given quantity of the hot gas, the temperature of the tar and other conditions of the process, as hereinbefore pointed out.

The present process and apparatus have proved well adapted for use on a large commercial scale. As an illustration of the application of the invention may be mentioned the distillation, at one plant during a period of about a year's time, of two million gallons of coke oven tar with the production of nearly one and one-half million gallons of distillate oil and with pitch having an average melting point of around 380° F. Oil yields from the coke oven tar distilled have been obtained of 75% or more over long periods of time, with the production of pitch of melting point around 400° F. or higher. Pitch of 435 and 450° F. melting point has been made over considerable periods of time. The coke oven gases entering the still have been at a temperature around 550° C. or higher. The temperature of the gases and vapors leaving the still has varied, for example, between 350° and 400° C. As illustrative of the operation of the process when pitch of 300° F. melting point was produced and with hot gases at about 550° C., the gases and vapors had an outlet temperature from the still of about 306° C. and the pitch left the still at a temperature of about 328° C.; while with pitch of 400° F. melting point the gases and vapors left the still at a temperature of about 324° C. and the pitch left the still at a temperature of about 355° C. The still employed was about 20 feet long, of rectangular cross section about 3½ feet wide and high and with six ovens connected by individual uptake pipes with the still.

The total distillate produced by the present process, which may represent as much as 75% or more of the tar distilled, can be condensed as a single composite oil and employed, for example, as creosote oil; or the admixed gases and vapors can be fraction-

ally cooled and fractionally condensed and a plurality of oil fractions recovered. A sample of the oil produced showed a specific gravity at 38° C. of 1.110, only a trace of free carbon, and, when subjected to distillation in accordance with the A. S. T. M. method showed up to 200° 1.1%, up to 210° 2.3%, up to 235° 8.3%, up to 270° 22.4%, up to 315° 34.3%, up to 355° 48.8%, and the percent of coke residue on the oil was 1.9%. It will be evident that the distillation can be carried out without the production of the maximum oil yield and the highest melting point pitch, and that under such conditions the process is well adapted for distilling an increased amount of tar for the production of a pitch of lower melting point and a correspondingly lower oil yield; but the distillation will nevertheless be carried out with the production of an unusually high oil yield corresponding to the melting point of the pitch produced. When distilling coke oven tar, for example, in simple externally heated stills of 10,000 gallons capacity, the oil yield is only around 44% when the distillation is carried to the point of producing pitch of around 300° F. and coking may begin before pitch of 400° F. melting point is reached. With other methods of distillation somewhat higher yields of oils can be obtained, to 300° melting point pitch but pitch as high as 400° F. has been made only with extreme difficulty but by the present process 400° melting point pitch is produced in a thoroughly successful manner and yields of around 75% or more of distillate oil can be obtained from coke oven tar when distilled to the point of producing pitch of melting point of around 400° F. or higher; while with the production of pitch of lower melting point, around 300° F. melting point, it is possible to produce oil yields representing around two thirds or more of the tar distilled. It will be understood, of course, that different tars vary in their oil content, depending upon the coal subjected to coking, and the nature of the coking operation, so that the percentage of oil obtainable from coke oven tar from one plant may not represent that obtainable from another plant operating with different types of ovens upon different coals and with a coking period extending over a different period of time, or with ovens of varying dimensions.

The invention will be further described in connection with the accompanying drawings, which illustrate the improved apparatus of the invention, and apparatus adapted for the carrying out of the improved process of the invention. The illustration is of a somewhat conventional and diagrammatic character.

In the drawings,

Fig. 1 is a plan, with parts broken away, of part of a coke oven plant, showing part

of the ordinary by-product recovery system, and also showing the improved apparatus of the present invention;

Fig. 2 is an enlarged vertical sectional view of one type of still embodying the invention;

Fig. 3 is a partial sectional view taken on the line 3—3 of Fig. 2;

Figs. 4, 5 and 6 are vertical sectional views showing modified forms of settling chambers or towers adapted to replace the tower shown in Fig. 2;

Fig. 7 is a horizontal view taken on the line 7—7 of Fig. 2;

Fig. 8 is a partial sectional view taken on the line 8—8 of Fig. 2;

Fig. 9 is a transverse sectional view taken on the line 9—9 of Figs. 7 and 8;

Fig. 10 is an enlarged view partially in section showing one type of fractional condenser;

Figs. 11 and 12 are horizontal sectional views taken on the lines 11—11 and 12—12 respectively of Fig. 10;

Fig. 13 is a diagrammatic view showing the still and condensing system of Figs. 1 to 3 and 7 to 12.

Fig. 14 is a diagrammatic view of a still adapted for countercurrent operation;

Fig. 15 is a similar view of a still adapted for partial concurrent and partial countercurrent operation;

Fig. 16 is a diagrammatic view of a still adapted for concurrent operation, with provision for supply of tar directly to the still or to the tower and with an indirect down draft condenser;

Fig. 17 is a diagrammatic view of two stills, one adapted for concurrent operation and one for countercurrent operation, with provision for supplying tar to both, and for drawing off pitch from both or for distilling the pitch successively in the two stills. This view also provides for direct fractional condensation of oils; and

Fig. 18 is a diagrammatic view of two stills adapted for countercurrent operation, with flow of pitch from one to the other, and with separate gas and vapor outlets, and provision for preheating the tar by indirect contact with the hot vapors and gases from both stills.

In the accompanying drawings, part of a coke oven block or battery is indicated conventionally at 1, and part of the conventional by-product recovery system is illustrated in Fig. 1, the individual coke ovens 2 having uptake pipes 3 leading to a collector main 4 common to a number of ovens of the battery. The cross over main 5 connects the collector main with the condensers 6 and the gases then pass through the exhausters 7 to the ammonia absorber, the benzol scrubber, etc. Ordinarily, ammonia liquor, or ammonia liquor and tar, are in-

roduced into the collector main to cool the gases and keep the main flushed. A drain for the tar and ammonia liquor is indicated at 8 leading to the decanter 9 from which the tar is drawn off into the tar receptacle 10. The condensers 6 which may be direct condensers, are provided with means for drawing off the cooling liquids and light tar into the decanter 11 from which the ammonia liquor collects in a suitable receptacle (not shown) and the light tar in receptacle 12.

The apparatus thus far described, which is illustrated only schematically, varies somewhat in different coke oven plants and is described merely for purposes of illustration, and, except as modified by the present invention, it is operated in the ordinary way.

Located for convenience at the other side of the coke oven block or battery to that where the collector main is located, I provide one or more stills, one being illustrated in the drawings. This still 20, as illustrated, is of rectangular cross section and is of such a length that four uptake pipes from four individual ovens pass upwardly into it, these four uptakes being shown as internal uptakes 21. Two additional uptake pipes 22 connect the still with ovens located beyond the ends of the still and these uptake pipes enter the still at its ends. The internal uptake pipes 21 have lateral openings into the still and closures 23 operated by operating handles 24 for closing individual uptake pipes during the charging of the coke ovens or when it is desired to valve off any oven from the still. Clean-out openings 25 are provided for each of the uptake pipes. It will be evident that the location and arrangement of the uptake pipes leading from the individual ovens to the still can be modified and varied, for example, all of the uptakes may be external uptakes.

The still and external uptake pipes are shown as provided with heavy insulation to prevent or reduce heat losses and reduction of temperature of the gases entering the still. In some of the figures, the insulation is omitted and some parts of the apparatus are shown without insulation, but it will be understood that where the lines or parts of the apparatus contain hot tar or pitch, or hot gases, insulation will ordinarily be provided to prevent heat loss. If heavy insulation is provided, the gases will enter the still at a temperature not materially below that at which they leave the individual ovens and they will thus be available for distillation at their maximum temperature and in a highly heated condition.

The outlet for gases and vapors from the still is shown at 27 leading into an enlarged chamber 28 which serves as a settling chamber and which may also serve as a dis-

tillation chamber for preheating and distilling tar, as hereafter described. A baffle 29 is located above the inlet to the chamber. The gas and vapor outlet 30 from the chamber 28 leads to a condenser 31, shown as a three stage fractional condenser, from the bottom of which the cooled gases pass through the pipe 32 to the gas handling system for the coke oven plant, entering that system between the condensers 6 and the exhaustor 7.

The condenser illustrated is a three stage condenser having two lower cooling sections 33 cooled by a suitable cooling liquid such as water and with an upper cooling section 34 which serves as a tar preheater and as a preliminary condenser for the gases and vapors. The upper tar preheating section has a tar supply pipe 35 leading to the tar coils 36 from which the preheated tar passes through the pipe 37 to the nozzle 38 located in the tower 28 between two packed sections 39 and 40 filled with broken up material or cylindrical rings, e. g. the well-known Raschig rings. The upper section 39 serves to reduce entrainment of suspended particles, while the lower section 40 serves the same purpose and in addition serves to bring the preheated tar into intimate contact with the admixed gases and vapors leaving the still. The preheated tar is thus partially distilled and the residue collects at the bottom of the tower 28 in the collecting section 41 and flows through the pipe 42 to one end of the still 20.

At the other end of the still is located a pitch outlet shown as a trap 43 having an adjustable overflow outlet 46 leading to the trough 47 which is supplied with a rapidly moving stream of water through a supply pipe 44. The pitch is granulated by rapid chilling in the water in the trough which discharges the water and granulated pitch into the receptacle 45. This method of cooling and handling the pitch is only one of various methods which can be employed, and, for certain purposes, the pitch can be cooled without admixture of water to give a solidified pitch product free from water.

Within the still 20 are located suitable atomizing or spraying devices for intensively atomizing or spraying the tar and pitch into the gases in such a way that all parts of the gases in the still are thoroughly scrubbed by the tar and pitch, and so that all of the internal surfaces of the still are intensively flushed by an excess of the tar or pitch. Where internal uptakes are employed, the space back of these uptakes where the spray will not reach is filled with suitable filling material 70 as shown in Figure 8. The mechanical spray devices shown are cylindrical rolls, approximately 10" in diameter, three in number indicated at 48, 49 and 50 and driven at relatively high speed

e. g. at 900 to 1000 R. P. M. by motors 51 and 52 located at the ends of the still. The middle roll 49 is coupled with one of the end rolls and is driven therewith by one of the
5 motors, the coupling being shown at 53. In order to provide bearings 55 for the individual rolls, part of the still is cut away as indicated at 54. The rolls are so located as to dip into the tar or pitch in the bottom
10 of the still, and the adjustable outlet for the pitch enables the depth to be regulated. By such regulation of the depth of the tar or pitch the extent to which the roll dips into the material is governed. With small immersion
15 the particles of spray produced are relatively fine and the volume of tar or pitch sprayed is relatively low. By increasing the depth of immersion the quantity of tar or pitch sprayed may be greatly increased and
20 the proportion of larger droplets of tar or pitch is also increased. Hence the character of the spray and the intensity may be governed at will by regulation of depth of tar or pitch. The volume of pitch in the bottom
25 of the still can be very materially reduced by filling in the lower corner opposite the roll to provide a ramp 71 as shown in Fig. 9. This reduces the size of the body of pitch, insures that the pitch is repeatedly
30 returned to the roll for respraying, and reduces the time during which the pitch is kept in the still.

In its broader aspects, the invention is not limited to the preheating of the tar, nor to
35 the introduction of the tar into the escaping gases and vapors to effect partial distillation before it enters the still; but such preheating and partial distillation is particularly advantageous and enables important
40 advantages to be obtained.

The condensing system shown in some of the figures of the drawings is a fractional or multi-stage condensing system, the first
45 stage of which is a preheater for the tar, in which the tar is preheated by indirect contact with the hot gases and vapors. The rate of distillation can be greatly increased by preheating the tar in this way, while the gases are at the same time cooled to
50 condense part of the distillate therefrom. The admixed gases and vapors are then further cooled in the lower stages of the condenser, for example, by indirect contact with cooling coils containing water. The condensing
55 system shown in Figs. 10 to 13 has a water inlet 60 and a water outlet 61 supplying water to the lower cooling sections 33. Three outlets 62, 63 and 64 are provided for the condensate from the respective sections.
60 Two return pipes 65 and 66, each leading to distributing nozzles 67 and 68, provide for returning the condensate from the upper section into the next lower section where it will blend with the condensate separated in
65 such section. By returning the condensate

from each of the two upper sections to the lower sections, the condenser becomes a total condenser where all of the constituents are condensed and collected together to be
70 drawn off through the bottom outlet 64. The construction of the condenser may vary, and that shown in Figs. 10 to 12 is only one of various types of condensers which can be employed, but it is a valuable type which
75 provides for the preheating of the tar to a high temperature before it enters the still.

The gases and vapors may be cooled in condensers of the direct type in which the cooling is by means of ammonia liquor directly in contact with the gases.
80

The cooling of the gases may be done as well by the indirect as by the direct type of condenser. However, from the standpoint of subsequent treatments to which the
85 gases may be subjected each type of condenser has definite advantages in special cases. The direct condenser is particularly well adapted for use in plants employing the indirect system for recovery of ammonia from the gases, e. g., the Semet-Solvay System, since in that system large quantities, of
90 especially cooled ammonia liquor normally used for cooling the gases are available. The required quantity of this cooled liquor is employed for cooling the gases and condensing the oils from the distillation unit. The ammonia liquor after separation from the oil is returned to the regular liquor circulation system.
95

In plants using the semi-direct system for ammonia recovery, e. g., the Koppers system, the greater proportion of the ammonia is recovered direct as ammonium sulfate by passing the coke oven gases thru dilute sulfuric acid. A relatively small proportion
100 of the ammonia is recovered from the ammonia liquor condensed from the gases in the collector and crossover mains and in the primary coolers. In this system it is desired to keep the production of such liquor at a minimum whereas in the indirect system very large quantities of water are added continually to the system for absorption of ammonia. Furthermore on the semi-direct system of the Koppers type, since no
105 cooling equipment for cooling ammonia liquor is provided, no cooled liquors are available for direct cooling of the gases from the distillation unit. In such a system it is preferable to employ the indirect type condenser since from it, a relatively small
110 amount of ammonia liquor is recovered and this liquor may be blended with that recovered from the gases not used for distillation.
115

The tar supplied to the still may be the tar collected in the by-product system of the same plant and pumped from the tanks
120 10 and 12 by pumps 56 and 57 through pipe 58 to the still, or tar from another source
125 130

of supply can be introduced through pipe 59, or mixtures of different tars, etc., can be introduced and distilled. Thus not only may pitches of special character be obtained
5 from the distillation operation but also the character of the oils recovered may be governed. The light and heavy tars from tanks 12 and 10 may be blended in a tank not shown and then pumped to the still through
10 line 58 or they may be left separate and separately distilled. The light tars will give relatively high yields of lighter oils, relatively rich in tar acids and naphthalene whereas the heavier tars will give relatively
15 lower yields of heavier oils and higher yields of pitch and the oils will be relatively poor in the tar acids and naphthalene. Thus by separately distilling such light and heavy tars or by blending them in proper proportion not only may the oil yields to various
20 pitches be regulated within limits but also the character of the oils and pitches recovered may be controlled.

When tar is not introduced into the still
25 through the chamber 28, that chamber may be an open chamber as indicated at Fig. 4. In such case, the tar can be directly supplied to the still at the end opposite the gas outlet. When the chamber 28 is such an open chamber, it will serve as a settling chamber where
30 entrained particles of spray from the zone of intensive spraying within the still are permitted to settle out and return to the still. In a still where the atomizing or
35 spraying is so thorough and effective as to accomplish the purposes of the present invention, some of the spray particles tend to leave the still with the gases and vapors and it is therefore important to provide
40 for the settling of these suspended particles out of the gases if they are not to be contaminated with suspended pitch particles when the oils are condensed therefrom. A settling chamber, well insulated to avoid
45 cooling of the gases with condensation of oil and in which the velocity of the gases is sufficiently low to allow settling out of the entrained pitch particles is well suited for the purpose.

50 In Figs. 5 and 6, modified forms of settling chambers or towers are shown. In Fig. 5, the chamber 28b has inclined baffles 39b over which the preheated tar passes so that it presents a large surface contact with
55 the gases and vapors rising through the chamber. The gases and vapors are thus brought into intimate contact with the preheated tar to effect distillation of it, and at the same time, the arrangement of the baffles assists in the removal of entrained
60 particles of pitch from the gases.

In Fig. 6 the chamber 28c has a series of trays 39c so arranged that the preheated tar flows from one tray to the next. The up-
65 rising gases and vapors from the still pass

over the surfaces of the preheated tar and aid in distilling it.

The progressive distillation of the tar to produce pitch results in vaporizing a large part of the oils in the tar to form vapors which admix with the gases and remain in
70 vapor form. The admixed gases and vapors pass from the still to the outlet 27 to the settling chamber 28. With a settling chamber such as 28a shown in Fig. 4, entrained
75 pitch particles are permitted to settle out and the admixed gases and vapors then pass to the condensing system. With chambers such as shown in Figs. 2, 5 and 6 in Fig. 13 when employed with a condenser in
80 which the tar is preheated, the preheated tar is brought into intimate and direct contact with the outgoing gases and vapors with the result that the tar is further heated and partially distilled, and the resulting oil vapors
85 added to the gases and vapors passing through the tower. The preliminary heating and distillation of the tar in this way accomplishes a greatly increased distillation
90 by means of heat which would otherwise be lost, and enables the oil vapor content of the gases to be increased, and enables the rate of distillation to be greatly increased, since the preheated and partially distilled tar entering the still requires less heat in the still for
95 its distillation to produce high melting point pitch, than is the case with tar not preheated or partially distilled.

The following data were collected when pitch of about 400° F. melting point was being produced and serve as an example and illustration of the process. Tar was supplied to the preheater at a temperature of around 63° C. and preheated by indirect contact
100 with the hot gases and vapors to around 221° C. while the gases and vapors were cooled from around 293° C. to around 175° C. The tar preheated to about 221° C. then entered the settling chamber where it was further heated and distilled by direct contact
105 with the hot gases and vapors therein, which were thereby cooled to a temperature of about 293° C. before they went to the preheater.

The intensive scrubbing to which the gases
115 are subjected in the still, during the distillation, results in effectively cleaning the gases from suspended carbon and pitch particles. As a result, when the gases and vapors from the still are subjected to condensation, the oils condensed are clean oils, substantially free from tarry or pitchy constituents, and show only traces of so-called free carbon when subjected to the usual benzol
120 extraction test.

When tar is distilled according to the present process to produce pitch of high melting point, around 400° F. or higher, the distillate includes heavy constituents which in an isolated state, are solid or semi-solid
130

at ordinary temperatures. When the gases are cooled either directly or indirectly to condense all or most of the vapors therefrom in a single composite product, the heavy constituents remain dissolved in the lighter oils and form with the lighter oils a valuable creosote oil. The condensing system illustrated in the drawings, provides for producing a total condensate by combining the condensate from each of the three sections of the condensing apparatus.

In the operation of the apparatus, and in the carrying out of the process of the present invention therein, the hot coke oven gases pass from the individual coke ovens into the still at practically their maximum temperature. Tar, which may be preheated and partially distilled, is introduced continuously into the still and a layer of tar or pitch maintained in the bottom of the still. This tar or pitch is intensively atomized into the gases in the still with such thoroughness of distribution and such abundance of spray that the gases are rapidly cooled from their highly heated condition while rapidly distilling the tar and pitch. All exposed surfaces of the still are heavily flushed with the tar and pitch spray, and the atomizing or spraying is so excessive that local overheating by the highly heated gases is avoided, and so that rapid and effective distillation of the tar and pitch takes place with simultaneous and immediate reduction of gas temperature. The pitch is drawn off at one end of the still while tar which may be preheated or even partly distilled, is continuously supplied at the other. The tar supplied at one end is progressively distilled and pitch of progressively higher melting point produced up to the maximum melting point of the pitch discharged from the still.

In the apparatus illustrated, all six ovens connected to the still may be continuously connected, and all of the gases from the six ovens passed through the still; or one or more ovens can be disconnected by closing one of the outlet openings into the still, and by connecting that oven with the ordinary collector main. When all six ovens are connected with the still, they are disconnected from the usual collector main, the arrangement being such that all of the ovens may be connected either with the still or with the collector main, or part with each.

The cooling of the hot gases and vapors for the condensation of oils can be so carried out as to produce a single total condensate suitable for example for use as creosote oil; or it can be carried out so as to produce heavier and lighter oils. In the apparatus of Figs. 1 to 13 separate fractions can be drawn off from the individual sections of the condenser illustrated, that is, through the pipes 62, 63 and 64 of Fig. 10, the condensate from the top section being a heavy

condensate and those from the lower sections being lighter condensates. Such separate fractions may be used separately or blended in any desired proportion for production of particular oil products for specific purposes.

The condenser shown in Figs. 1 to 13 is an indirect condenser in which the gases and vapors do not come into direct contact with the cooling liquid. In some cases it is more advantageous to employ direct condensers in which ammonia liquor or water is brought into direct contact with the hot gases and vapors to effect their cooling and condensation; or to effect fractional condensation by direct cooling with oils of lower boiling point.

The apparatus illustrated in Figs. 1 to 13 is an apparatus in which the flow of gases and tar or pitch through the still is of a generally concurrent character, in which the pitch leaves the same end of the still that the hot gases and vapors leave. With this arrangement of apparatus, the hot gases come into contact with fresh tar and with partially distilled tar, and the relatively cooler and more or less saturated gases leave in contact with the more or less completely distilled final pitch. In this type of operation, the hot exit gases are not cooled in the still as much as would be the case if they left the distilling main in contact with the relatively cool and fresh tar, and hence they may leave the still at a higher temperature, in which case they are available for increased distillation of the tar in the chamber 28. With this generally concurrent flow of the gases and tar, there is less danger of the formation of coke by local overheating, since the more or less completely distilled pitch is in contact with relatively cooler gases which are already more or less saturated with oil vapors than is the case with countercurrent flow wherein the final pitch leaves the system immediately after contact with fresh hot gases at maximum temperature. In the concurrent system the final pitch leaves the system at a temperature lower than is the case with countercurrent flow, assuming the pitches are of the same melting point.

The apparatus of Fig. 14 is a countercurrent apparatus in which the gases have a flow which is generally countercurrent to the flow of tar and pitch through the still. In Fig. 14 the chamber 28*d* is shown as an open settling chamber from the top of which the gases and vapors pass to the condenser 34*d* containing the preheating coil 36*d* for the tar. The tar supplied through pipe 35*d* is preheated by indirect contact with the hot gases and vapors and serves to cool them, the condensate being drawn off through 62*d*. The gases and vapors then pass to a condenser 33*d* where further cooling and con-

densation takes place, the condensed oil being drawn off at 63*d*. The flow of gases and tar in the still is countercurrent. Fresh hot gases at practically maximum temperature come into contact with final pitch and the final partially cooled gases leave in contact with relatively cooled tar.

This enables the gases to be cooled to a lower temperature and more effectively employed in the still, but the need of care in preventing overheating and coking of the pitch leaving the still is increased, since the pitch more readily cokes in contact with the hotter gases and the hotter still surfaces.

Instead of using a generally concurrent flow of gases and pitch, or a generally countercurrent flow, a combination of concurrent and countercurrent flow can be employed and many of the advantages of both types of flow obtained. Such a combined concurrent and countercurrent flow will take place in the apparatus of Fig. 15. In this arrangement the tar (which may be preheated and partially distilled) enters through the pipe 42*e* at one end of the still while the pitch escapes from the other end. During the first part of the travel of the tar and pitch through the still the flow is generally concurrent with the flow of gases, while during the travel of the pitch through the other part of the still its travel is generally countercurrent to that of the hot gases. The intensity of the spray is such that the gases are rapidly brought into or approaching equilibrium with the pitch with which they are being sprayed. The arrangement of Fig. 15 is such that the initial distillation of the tar and the production of softer pitch is with concurrent flow while the production of hard pitch is obtained with countercurrent flow.

In Fig. 15 a tower of the type shown at 28 in Fig. 1 may be used or a simple settling chamber 28*e*. The condenser shown in Fig. 15 is a condenser cooled by direct contact with ammonia liquor entering at 60*e* through suitable distributing devices and passing downwardly over grids or other filling material in the tower 34*e*. The admixed condensate and liquor escapes at 64*e* and the ammonia liquor after separation can be recirculated or other liquor employed.

In the apparatus of Fig. 16 provision is made for admitting tar directly to one end of the still and for withdrawing pitch from the other end. Provision is also made for introducing part or all of the tar directly to the tower 28*f* into direct contact with the hot enriched gases passing through the tower to preheat and partially distill the tar after which the resulting pitch enters the still. The general arrangement of flow of gases and pitch in the distilling main 20*f* is concurrent. The tar may be in part introduced through the tower and in part di-

rectly into one end of the still without preheating.

The condenser shown in Fig. 16 is an indirect down-draft condenser in which the cooling liquid flows upwardly in indirect contact with the gases and vapors. The oil condensed in the upper part of the condenser flows downwardly and washes the lower coils of the condenser, thus freeing the lower coils from any danger of naphthalene deposit.

It is sometimes desirable to produce simultaneously pitches of high and low melting point. This can readily be accomplished by withdrawing pitch from an intermediate point in the still as well as from the pitch outlet at one end. So also, two or more stills can be provided for distilling pitch to progressively higher melting points and the stills can be operated to produce pitches of different melting points. Two arrangements of multiple stills are shown in Figs. 17 and 18.

In Fig. 17 the stills 20*g* and 20*g'* are so arranged that the gases and vapors from the still 20*g* pass through the still 20*g'*. Provision is made for introducing tar directly into each still or directly into the tower through which the hot enriched gases leave the still 20*g'*. Provision is made for drawing off pitch from both stills and also for introducing low melting point pitch from the still 20*g'* into the still 20*g* for further distillation. The arrangement shown provides for countercurrent flow of pitch and gases in the still 20*g'* and concurrent flow in the still 20*g*. Part of the low or intermediate melting point pitch produced in the still 20*g'* can be withdrawn as a separate product and the remainder introduced to the still 20*g* for further distillation and the production of a higher melting point pitch. This arrangement provides for a combined countercurrent and concurrent flow in a somewhat different manner from that provided for in the arrangement of Fig. 15.

The tower 28*g* of Fig. 17 may be similar to the tower 28 of Fig. 13 with introduction of tar (with or without preheating and partial distillation) through the tower and thence into the still. The condenser shown in Fig. 17 provides for fractional condensation of the oils by direct contact with oils introduced into the tower and which oils may be themselves vaporized. The fractional condenser 34*g* is shown as a two-stage condenser with provision for introducing oil into each stage and for withdrawing the condensate from each stage.

The two stills shown in Fig. 18 are arranged to permit production of pitches of different melting points in the different stills, or, by operating the stills in series, producing a high melting point pitch from

the second still. The settling chambers 28h and 28h' are shown without introduction of tar or pitch through them, but the condensers 34h and 34h' are indirect condensers through which the tar is passed in indirect contact with the hot gases and vapors to preheat the tar, which is then fed into the still 20h'. The preheating of the tar is in a countercurrent manner. Provision is made for discharging the low melting point pitch from the still 20h' into the still 20h and also for withdrawing low melting point pitch from this still. Tar (which may or may not be preheated), may be introduced directly into either or both stills.

In Figs. 13 to 18, which are of a diagrammatic nature, various arrangements are shown of concurrent, countercurrent, or combined concurrent and countercurrent flow of gases and pitch. So also, in these figures there are shown various arrangements of condensers and various arrangements of preheaters for preheating the tar either indirectly or directly before it is introduced into the still. It will be evident that the different methods of preheating and condensation shown in the different figures can be combined with the different types of concurrent, countercurrent or combined operations, and, for that reason, the different types of preheaters and condensers are shown with different types of gas and pitch flow in the still, instead of showing each type of still with the various types of preheaters and condensers which can be employed.

Where a preheating and distilling tower is provided for distilling tar by direct contact with the hot gases and vapors leaving the still, the action in the tower is a generally countercurrent action. This countercurrent distillation of the tar before it reaches the still may be combined with either a concurrent or a countercurrent distillation in the still, or with a partial concurrent and partial countercurrent distillation, as generally illustrated in Figs. 13 to 18. With the countercurrent system operation, where the highest melting point pitch is in contact with the hottest gases in the still, the pitch leaves the still at a higher temperature and, as above stated, there is greater danger of overheating and coke formation, especially with pitches of high melting point, e. g. 400° F. Less heat is lost with the pitch, and the pitch leaves the still at a lower temperature with concurrent operation.

The temperature of the pitch leaving the still, and also the temperature of the gases and vapors leaving the still, will vary somewhat with the type of operation, whether with concurrent flow of gases and pitch, or countercurrent flow, or partly concurrent and partly concurrent and partly countercurrent. With parallel or concurrent flow

of the gases and pitch in the still, without distillation of tar in the settling tower, and with gases initially at about 550° C., pitch of around 400° F. melting point may leave the still at a temperature around 360° C. and the gases and vapors may leave the settling tower at a temperature around 325° C. With similar parallel or concurrent flow of gases and pitch in the still and with preheating and partial distillation of tar in the settling or scrubbing tower, pitch of around 400° F. melting point may leave the still at a temperature around 375° C. and the gases and vapors may leave the top of the scrubbing or settling tower at a temperature around 255° C. With combined concurrent and countercurrent flow, as illustrated in Fig. 15, together with preheating and distillation of the tar in the scrubbing and settling tower, the pitch of around 400° F. melting point may leave the still at a temperature around 410° C. and the gases and vapors may leave the top of the scrubbing tower at a temperature around 270° C.

It will be understood that the temperatures given will vary, depending upon the initial temperature of the hot coke oven gases, upon the character, the temperature and water content of the tar, upon the extent and efficiency of the insulation used on the equipment, etc. The temperatures given were those which prevailed under existing conditions in apparatus of the types described.

When the tar is preheated by indirect contact with the hot gases and vapors in a separate heat interchanger and then introduced directly into the still, as in Figs. 14 and 18, the tar can be preheated to a higher temperature than in the apparatus shown in Fig. 13 where the gases and vapors leave the tower and enter the tar preheater at a lower temperature; but with the apparatus of Fig. 13 the preheated tar will be further heated and partially distilled in the tower, as hereinbefore described.

The temperatures above mentioned will vary with the melting point of the pitch produced and with other conditions of operation, and the particular temperatures above mentioned are intended to be illustrative of the process and not in a limiting sense.

In an apparatus such as shown in the drawings in which the gases from six coke ovens pass through the still, and in which the tar is distilled to pitch of around 400° F. melting point, the average time required for the gases to pass through the still is only around 1 to 5 seconds, and, during this period of time, the gases are cooled from an average temperature around 600° C. to a temperature e. g. around 250-350° C. It will be understood that this period of time will vary, depending upon the initial temperature of the gases, the size of the coke oven,

character and degree of coking of the coal, etc. The figure shown indicates the order of magnitude of the period. With distillation of tar in the still to produce 400° F. melting point pitch, the average time required for the passage of the tar through the still and its distillation therein is only a few minutes, for example, 2 to 10 minutes. This period of time will depend upon several factors but that given indicates the general order of magnitude of the period. It will be seen accordingly that the heat of the hot coke oven gases is almost instantaneously employed during the brief period of travel of the gases through the still, and that the superheated gases are almost instantaneously cooled to a much lower temperature by their intimate and thorough contact with the sprayed tar and pitch. The tar and pitch, in turn, is repeatedly sprayed into the gases and rapidly distilled so that only a short time is required for the distillation of the tar and the production of high melting point pitch. This time will vary with the size and operation of the still and with the volume and character of tar or pitch in the still during its operation, but the distillation will be a rapid distillation carried out continuously with highly heated gases.

The present invention enables a wide range of pitches and of oils to be produced, and pitches of various melting points as well as oils of various characteristics can be simultaneously produced. Pitches of high melting point and pitches of intermediate or low melting point can be simultaneously produced and withdrawn from the same still or from different stills. Creosote oils, tar acid or carbolic oils, or other oil fractions can be directly produced. The oils, moreover, will be clean oils substantially free from carbon or heavy pitch constituents, because of the intensive and excessive scrubbing to which the gases are subjected during the distillation, which effectively removes "tar fog" particles from the gases.

It will also be seen that the present invention provides an improved process and apparatus for the distillation of tar and the production of an exceptionally high yield of distillate oils and with production of high melting point pitch therefrom, in which the distillation is carried out by direct contact of the tar with highly heated gases, in which the gases are suddenly and rapidly cooled by extremely intimate contact with an excessive and intensive amount of spray of the tar or pitch to be distilled, in which the tar or pitch in a finely divided form is rapidly distilled, in which the gases are effectively scrubbed and cleaned by the spray of tar and pitch, in which the distillation is carried out in a continuous manner with the production of a liquid pitch of melting point very much higher than obtainable by

any other process with which I am familiar and an exceptionally high oil yield, and with other singular advantages, such as those hereinbefore pointed out.

It will further be seen that the distillation capacity of the process and apparatus can be radically increased by preheating the tar and introducing it into direct contact with the hot gases and vapors leaving the still, with resulting material increase in the vapor content of the gases, with partial distillation of the tar so that hot partially distilled tar is supplied to the still, with resulting heat economy in distillation and advantages in subsequent cooling because of the partial cooling of the gases and vapors by the distillation of the tar, and with other advantages. This preheating and preliminary distillation of the tar by the hot gases and vapors enables the size of the distillation plant to be greatly reduced, or, for a plant of a particular size, enables its distillation capacity to be greatly increased.

The present invention, as previously pointed out, is particularly advantageous for the distillation of coke oven and other tars at coke oven plants and with the use of hot coke oven gases for the distillation. In its broader aspects it includes the employment of other highly heated gases at a similar high temperature. The process and apparatus are advantageously employed at coal distillation or coal carbonization plants where highly heated fuel gases are produced and are available for the distillation and furnish a source of heat which is commonly lost as waste heat.

In the specification and claims references to pitch melting points refer to melting points determined by the method described in "Methods of Analysis Used in the Coal Tar Industry", by J. M. Weiss in the Journal of Industrial Engineering and Chemistry, vol. 10, No. 10, October 1918, page 817.

I claim:

1. In combination with a coal distillation plant a tar still adapted to contain a small body of tar, means for supplying hot fresh coal distillation gases from the ovens or retorts of the plant directly into the still, spraying means in the still adapted to spray and respray the tar from the bottom of the still up into the gases passing therethrough in the form of a fine intense spray and with sufficient force to impinge against and wash the interior surfaces of the still, a settling chamber, condensers and means for passing gases and vapors from the still through the settling chamber to the condenser.

2. The method of distilling a hydrocarbon liquid by means of a hot gas that is at a temperature at which rapid decomposition of the hydrocarbon occurs, which comprises introducing the hydrocarbon into a still having a rapidly rotating spray roll of sub-

stantially circular cross-section, maintaining in the still a body of the hydrocarbon liquid of such depth that said spray roll dips therein, introducing the hot gases into the still, repeatedly spraying the hydrocarbon into the gases by rapidly rotating the spray roll, the axis of rotation being substantially parallel to the surface of the body of liquid, controlling the rotation of the spray roll and the introduction of the hydrocarbon liquid so that the spray produced is of sufficient volume and intensity that the gases are rapidly cooled to a temperature below that at which rapid decomposition of the hydrocarbon occurs and the inner surfaces of the still are continuously flushed with hydrocarbon liquid so that the accumulation of material on these surfaces is prevented.

3. The method of distilling liquid hydrocarbons of the class consisting of oils, tars, tarry oils, and pitches by contact with hot fresh coal distillation gases in a still which comprises introducing into the still hot fresh coal distillation gases that are yet at a temperature not greatly below that at which the gases left the coal distillation oven or retort and which contain suspended impurities, introducing into the still continuously the hydrocarbon to be distilled, intensively spraying and respraying the hydrocarbon into the gases and so regulating the volume and temperature of the spray with respect to the volume and temperature of the gases that the hydrocarbon is rapidly heated and distilled and suspended impurities are thoroughly scrubbed and cleaned from the gases and the gases are immediately cooled to a temperature approximating that of the hydrocarbon spray with which they are in contact, continuously withdrawing distillation residue from the still, and continuously withdrawing and cooling the gases and vapors to condense oils therefrom.

4. The method of distilling a liquid hydrocarbon of the class consisting of coke oven tar, coke oven tarry oil, and coke oven tar pitch by contact with hot coke oven gases which method comprises intensively spraying the hydrocarbon into hot coke oven gases while the gases are at a temperature not greatly below that at which they leave the coke ovens, so regulating the amount of hydrocarbon supplied and the thoroughness and intensity of spraying with respect to the volume and temperature of the coke oven gases employed for distillation that almost instantaneous cooling of the gases to a substantially lower temperature and rapid heating and distillation of the hydrocarbon are effected, and there are thereby obtained a high melting point pitch and a high yield of distillate oil, withdrawing the pitch from the still, and withdrawing the mixed gases

and vapors and cooling the same to condense the distillate therefrom.

5. The method of distilling coke oven tar by contacting the tar with hot coke oven gases in a still which method comprises supplying hot coke oven gases from the coke ovens to a still without any considerable reduction of temperature, repeatedly spraying the tar or pitch resulting from distillation of tar into the gases, the volume, thoroughness, and intensity of the spray being so regulated with regard to the volume and temperature of the gases that the gases are immediately cooled to a much lower temperature, regulating the amount of tar supplied so as to obtain distillation therefrom of at least 75 percent of the tar as distillate oil and a pitch residue of a melting point of about 400° F. or higher, maintaining the pitch produced in a fluid state and withdrawing the same continuously from the still, and withdrawing the admixed gases and vapors and cooling the same to separate distillate oils therefrom.

6. The method of distilling liquid hydrocarbons of the class consisting of oils, tars, tarry oils, and pitches by contact with highly heated gases in a still, which method comprises supplying the gases at a high temperature continuously to a still, continuously introducing into the still the hydrocarbons to be distilled, maintaining in the still a small body of liquid comprising distillation residue distilled from the hydrocarbons, repeatedly spraying the hydrocarbons and distillation residue into the gases, the volume, thoroughness and intensity of the spray being so regulated with respect to the volume and temperature of the gases that the gases are immediately cooled to a temperature approaching that of the hydrocarbons with which they are being sprayed, and the hydrocarbons are rapidly distilled to produce a residue, continuously withdrawing the resulting residue from the still, passing the gases and vapors leaving the still through a settling chamber to remove entrained spray particles therefrom, and then cooling the gases and vapors to condense oils therefrom.

7. The method of distilling tar and producing pitch by contacting tar with highly heated gases in a still, which method comprises conveying the gases to a still while at a high temperature, spraying tar and pitch into the gases, the volume, intensity, and thoroughness of the spray being so regulated that the hot gases are scrubbed and immediately cooled to a temperature approaching that of the tar and pitch with which the gases are in contact, regulating the supply of tar so that it is rapidly distilled to produce pitch of high melting point and a high oil yield, continuously withdrawing the pitch from the still, continuously

removing the hot admixed distillation gases and vapors and passing them in heat exchange relation with the tar to preheat the tar before it enters the still thereby partly cooling the admixed gases and vapors, and further cooling the gases and vapors to condense the oils therefrom.

8. The method of distilling tar in a still, which method comprises conveying highly heated gases to a still, spraying tar or pitch resulting from distillation of the tar into the gases in the still, the volume, intensity, and thoroughness of the spray being so regulated with respect to the volume and temperature of the gases that the temperature of the gases is immediately reduced to approach that of the tar and pitch with which the gases are in contact, and the tar is rapidly distilled to produce pitch of high melting point and a high oil yield, continuously withdrawing the pitch from the still, withdrawing the hot gases and vapors from the still and passing them into direct contact with tar to effect partial distillation thereof, introducing the resulting partly distilled tar to the still, and withdrawing the resulting gases and vapors and cooling them to condense oils therefrom.

9. The further improvement in the process of the next preceding claim in which the hot gases and vapors are cooled by indirect contact with tar to preheat the tar before it is introduced into direct contact with the hot gases and vapors leaving the still.

10. The method of operating a still provided with a rapidly rotating spray roll of a generally cylindrical contour for distillation of tar, which comprises introducing into the still highly heated gases, introducing into the still continuously the tar to be distilled, maintaining in the still a liquid body of tar and pitch resulting from the tar distillation of such depth that said spray roll dips therein, repeatedly spraying the tar into the gases by rapidly rotating the spray roll dipping into the liquid body thereby producing a spray of tar and pitch in the still, controlling the operation of the spray roll and the volume of tar so that the spray produced is of such volume and intensity that the gases are rapidly cooled to a temperature below that at which rapid decomposition of the hydrocarbon occurs and the inner surfaces of the still are continuously flushed with tar and pitch so that the accumulation of coke on these surfaces is prevented, continuously withdrawing the resulting pitch from the still, continuously withdrawing the resulting gases and vapors from the still, and cooling the same to condense oils therefrom.

11. The method of distilling tar by contact with highly heated gases in a still which comprises preheating the tar by in-

direct contact with hot gases and vapors leaving the still, introducing the preheated tar into the still at a point remote from that at which the gases and vapors leave, introducing highly heated gases into the still and withdrawing hot gases and vapors from the still, passing the hot gases and vapors into indirect contact with the tar to preheat the same, repeatedly spraying the tar and pitch resulting from distillation of tar into the gases and regulating the volume and intensity of the spray so that the gases are rapidly cooled and the tar is rapidly heated and distilled, withdrawing pitch from a point remote from that at which the gas enters the still, and causing the gases and pitch to leave the still in a generally concurrent direction.

12. The further improvement in the process of the next preceding claim in which preheated tar is further heated and distilled by bringing it into direct contact with the hot gases and vapors from the still before introducing the tar into the still.

13. The process according to claim 4 in which the hot gases and vapors after leaving the still are cooled to give a total oil condensate suitable for creosote oil.

14. The process according to claim 11 in which the tar and gases during part of the distillation flow in a generally counter-current direction.

15. In the operation of a by-product coal distillation plant wherein coal is distilled to form coke and tar-containing gases, the process for distilling a hydrocarbon liquid by such gases while they are hot and fresh without adversely affecting the coking operation, which process comprises passing the hot fresh coal distillation gases in unrestricted flow through an intensive spray of the liquid to be distilled, said spray being of the type obtainable by rotating at about 900 to 1000 R. P. M. a smooth circular member having a diameter of about 10 inches and dipping slightly below the surface of a body of liquid, the axis of rotation being substantially parallel to the liquid surface, whereby the gases are enriched in oil vapors and simultaneously cleaned and whereby the gases are maintained at substantially the same pressure throughout the distillation.

16. The method of distilling tar, which comprises subjecting tar to partial distillation by heating it under pressure and passing the heated tar into a vapor separating chamber at a lower pressure, the temperature of the tar being raised in said heating step to a point where only part of the volatile constituents will be vaporized at the pressure of the vapor separating chamber, simultaneously passing a stream of coal carbonization gases through said chamber in direct contact with the tar, and then distilling the hot liquid residue from said par-

tial distillation by subjecting it to intimate contact with said stream of coal carbonization gases prior to their introduction to said vapor separating chamber and while they
5 are at a high temperature after their production.

17. The method of distilling a hydrocarbon of the group consisting of tar, tar oil, and pitch by means of a hot gas that
10 is at a temperature at which rapid decomposition of the hydrocarbon occurs, which comprises introducing the hydrocarbon into a still having a rapidly rotating spray roll of substantially circular cross-section, maintaining in the still a liquid body of the
15 hydrocarbon of such depth that said spray roll dips therein, introducing the hot gases into the still, repeatedly spraying the hydrocarbon into the gases by rapidly rotating the spray roll, the axis of rotation being
20 substantially parallel to the surface of the body of liquid, controlling the rotation of the spray roll and the introduction of the hydrocarbon so that the spray produced is of such volume and intensity that the gases
25 are rapidly cooled to a temperature below that at which rapid decomposition of the hydrocarbon occurs and the inner surfaces of the still are continuously flushed with hydrocarbon liquid so that the accumulation
30 of material on these surfaces is prevented.

18. The method of distilling a hydrocarbon of the group consisting of tar, tar oil, and pitch by means of fresh hot coal distillation gases that are yet at a temperature at
35 which rapid decomposition of the hydrocarbon occurs, which comprises introducing the hydrocarbon into a still having a rapidly rotating spray roll of substantially circular cross-section, maintaining in the still a liquid
40 body of the hydrocarbon of such depth that said spray roll dips therein, introducing the hot gases into the still, repeatedly spraying the hydrocarbon into the gases by rapidly rotating the spray roll, the axis of rotation
45 being substantially parallel to the surface of the body of liquid, controlling the rotation of the spray roll and the introduction of the hydrocarbon so that the spray produced is of such volume and intensity that the gases are
50 rapidly cooled to a temperature below that at which rapid decomposition of the hydrocarbon occurs and the inner surfaces of the

still are continuously flushed with hydrocarbon liquid so that the accumulation of material on these surfaces is prevented.

19. The method of distilling a hydrocarbon of the group consisting of tar, tar oil, and pitch by means of fresh hot coke oven
20 gases that are at a temperature at which rapid decomposition of the hydrocarbon occurs, which comprises introducing the hydrocarbon into a still having a rapidly rotating spray roll of substantially circular cross-section, maintaining in the still a liquid body
75 of the hydrocarbon of such depth that said spray roll dips therein, introducing the hot gases into the still, repeatedly spraying the hydrocarbon into the gases by rapidly rotating the spray roll, the axis of rotation being substantially parallel to the surface of
80 the body of liquid, controlling the rotation of the spray roll and the introduction of the hydrocarbon so that the spray produced is of such volume and intensity that the gases are rapidly cooled to a temperature below that at which rapid decomposition of the
85 hydrocarbon occurs and the inner surfaces of the still are continuously flushed with hydrocarbon liquid so that the accumulation of material on these surfaces is prevented.

20. The method of distilling tar by means of hot fresh coal distillation gases that are
95 at a temperature at which rapid decomposition of tar constituents occurs, which comprises introducing the tar into a still having a rapidly rotating spray roll of substantially circular cross-section, maintaining in the
100 still a body of the tar and pitch resulting from the tar distillation of such depth that such spray roll dips therein, introducing the hot gases into the still, repeatedly spraying the tar and pitch into the gases by rapidly rotating the spray roll, the axis of rotation
105 being substantially parallel to the surface of the body of liquid, controlling the rotation of the spray roll and the introduction of tar so that the spray produced is of such volume and intensity that the gases are rapidly cooled to a temperature below that at which
110 rapid decomposition of the tar constituents occurs and the inner surfaces of the still are continuously flushed with tar or pitch so that the accumulation of material on these
115 surfaces is prevented.

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