Feb. 9, 1932. S. P. MILLER 1,844,896

PRODUCTION OF HIGH CARBON PITCHES, ETC

Filed Dec. 29, 1927 2 Sheets-Sheet 1

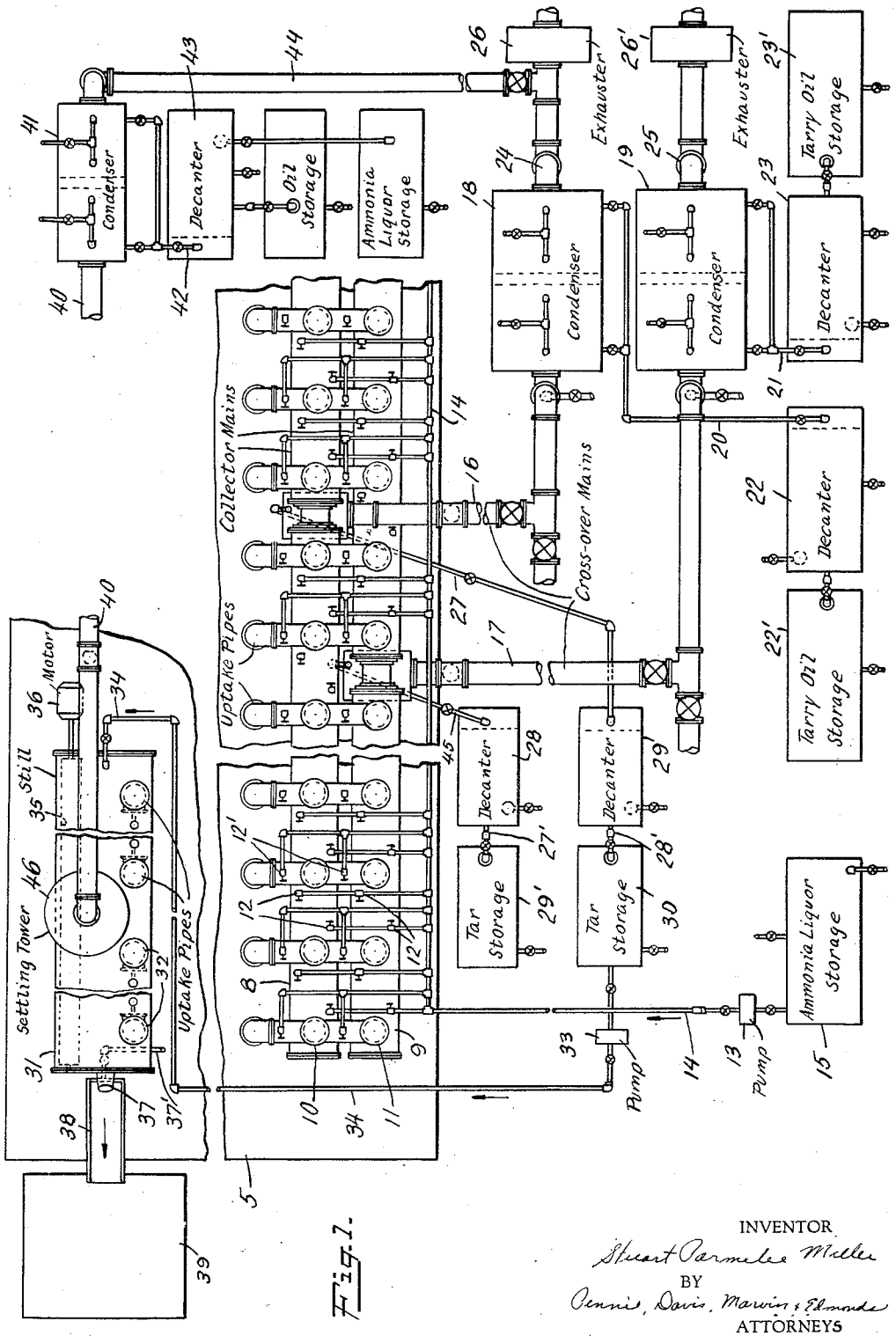

Fig. 1.

INVENTOR
Stuart Parmelee Miller
BY
Dennis, Davis, Marvin & Edmonds
ATTORNEYS

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Fig. 3.

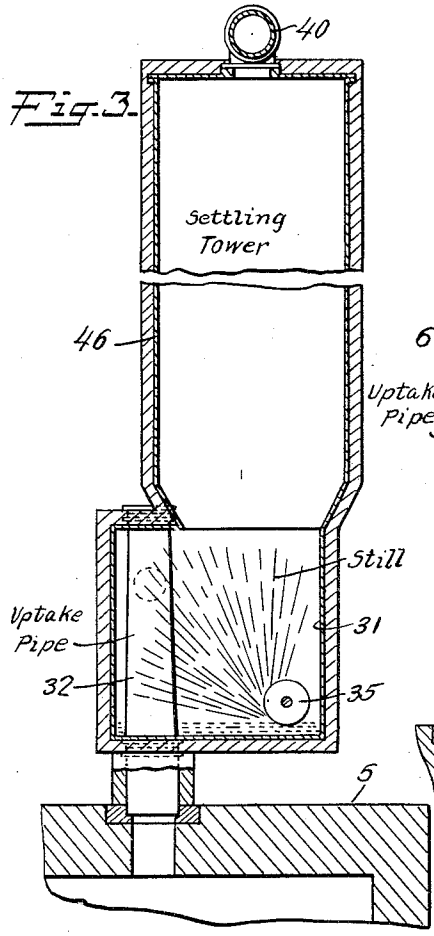


Fig. 2.

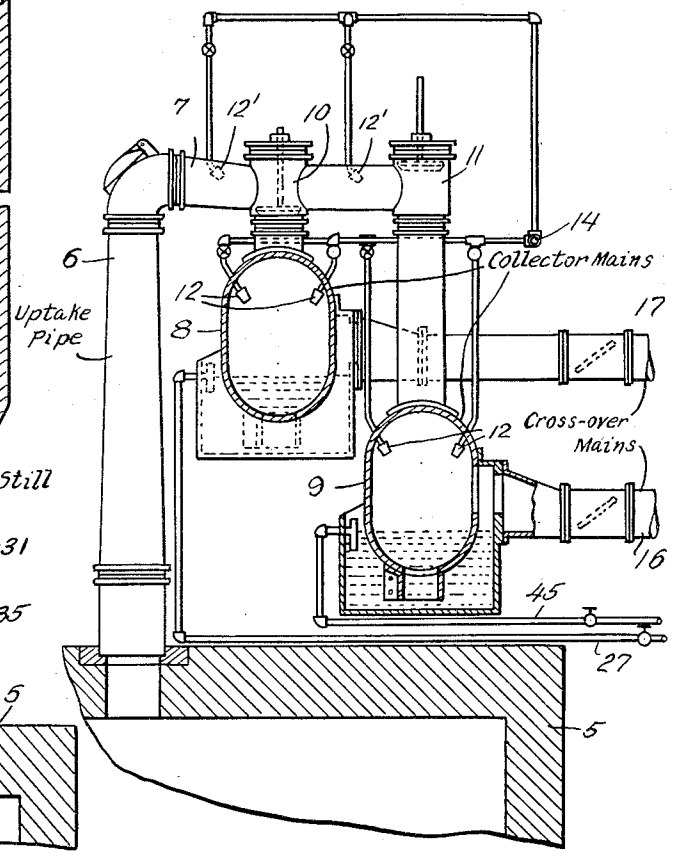
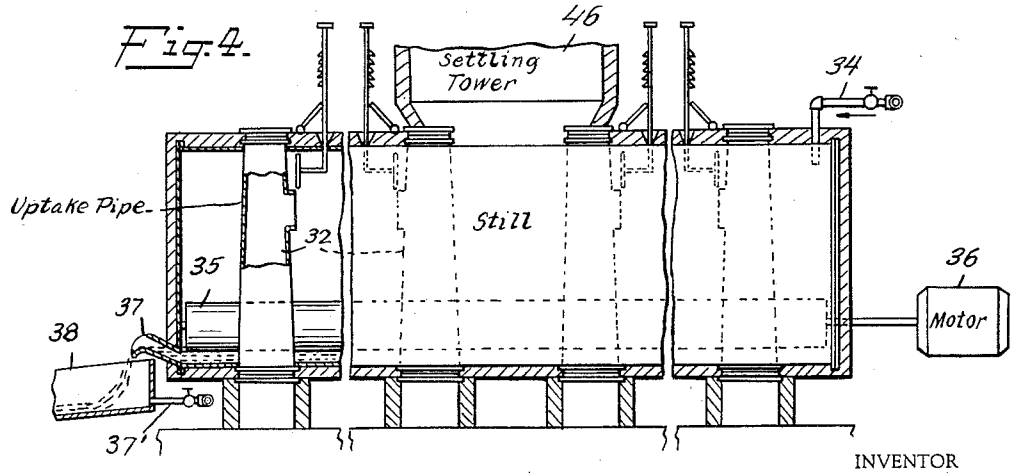


Fig. 4.



INVENTOR
Stuart Parmelee Miller
BY
Dennis Davis, Marvin & Edmonds
ATTORNEYS

Patented Feb. 9, 1932

1,844,896

UNITED STATES PATENT OFFICE

STUART PARMELEE MILLER, OF ENGLEWOOD, NEW JERSEY, ASSIGNOR TO THE BARRETT COMPANY, OF NEW YORK, N. Y., A CORPORATION OF NEW JERSEY

PRODUCTION OF HIGH CARBON PITCHES, ETC.

Application filed December 29, 1927. Serial No. 243,307.

This invention relates to the production of so-called "high carbon" pitches having predetermined characteristics.

In the ordinary operation of by-product coke ovens the gases produced by the coking operation pass from the individual ovens through uptake pipes and goose-necks to a collector main common to the ovens of the battery The gases which leave the ovens at high temperature, e. g., 600° to 700° C. or higher, are cooled ordinarily as rapidly as possible by the application of sprays of ammonia liquor or ammonia liquor and tar in the goose-necks and collector main. The rapid cooling causes the separation of a heavy tar in the collector main. Further cooling is effected in the cross-over main which connects the collector main to the condensing system and in the condensing system and an additional quantity of light tar or tarry oils is thus separated. The collected heavy and light tar is ordinarily mixed and shipped from the coke oven plant to a tar distillation plant for distillation and separation of the oils and the production of pitches therefrom.

The pitches produced by distillation of the tar do not contain ordinarily a sufficient proportion of free carbon to make them acceptable for certain uses for which pitches having a high free carbon content are required, such as certain road and roofing pitches requiring a high proportion of free carbon. It is customary, therefore, to prepare such products by mixing gas-retort or similar tars containing a large proportion of free carbon with ordinary coke oven tar, the mixture being then distilled to produce a pitch having the required free carbon content. The quantity of gas retort tar and similar material having the desired characteristics is, however, relatively limited and the demand for pitches having relatively high free carbon content is increasing. It will be understood by those skilled in the art that the terms "high carbon" and "free carbon" do not necessarily refer to carbon. They refer to those constituents of pitch, tar, etc., which remain as an insoluble residue when the pitch or tar is diluted with benzol, or other solvent such as carbon bisulfide. The so-called "free carbon" consists doubtless of some carbon per se but largely of hydrocarbons of relatively high molecular weight.

It is an object of the present invention to provide a method and apparatus whereby new pitches having high free carbon content, making them suitable for various purposes requiring the presence of free carbon, may be produced directly at coke oven plants, thus avoiding the necessity for using gas retort and similar tar and simplifying the procedure in producing high free carbon pitches.

It is a further object of the invention to provide new pitches of high free carbon content at gas retort plants, including pitches of higher carbon content (for the same melting point) than the highest carbon pitches now ordinarily obtainable from gas retort tar.

During the operation of a coke oven or retort the gases discharged contain a greater or less proportion of tar constituents depending upon the stage of the coking or gas making operation. In the earlier part of the operation the gases contain a relatively larger proportion of tar constituents and the tar separated from such gases is relatively low in free carbon as compared with that from the later stage of the system. During the latter part of the operation, however, the tar yield decreases but the proportion of free carbon in the tar recovered is higher. Thus, for example, during approximately the last third or quarter of the coal distillation in each individual oven the tar which is separated from the gases contains a high proportion of free carbon.

In carrying out the invention, advantage is taken of the distinctive characteristics of the tar carried by the gases escaping from the ovens during different periods in the op-

eration thereof by providing two or more collector or hydraulic mains which may be connected to part or all of the ovens of the battery and by diverting the gases from the ovens during the required periods in the operation thereof into one or the other of the collector mains, to separate therefrom in one of the mains tar of high carbon content, and such tar is then kept separate and separately distilled to give a high carbon pitch. Thus, the rich gases which are delivered from the ovens during the earlier part of the coking operation may be delivered to one main, and during the latter part of the operation the ovens may be connected to another main so that the gasses carrying tar with high free carbon content will be treated separately to recover tar having the desired characteristics, and the high carbon tar thus separated from the gases is then separately distilled to give a high carbon pitch.

In a companion application, Serial No. 243,305, I have described the production of low carbon pitch by separately collecting the gases from the early portion of the coking period, cooling the gases to separate tar therefrom, and separately distilling the low carbon tar to produce low carbon pitch. In carrying out the present invention, the period of operation during which the gases are separately collected for the production of a high carbon tar is regulated in accordance with the carbon content of the tar and of the pitch to be produced therefrom, and the total gases from the remaining portion of the coking operation can be separately collected and the tar recovered therefrom separately distilled to give a lower carbon pitch. In such cases, a high carbon pitch of regulated high carbon content and a lower carbon content pitch can be simultaneously produced. In order to produce simultaneously both a high carbon pitch of regulated high carbon content and a low carbon pitch of low carbon content, provision must be made for regulating independently the period of operation during which the high carbon tar gases and the low carbon tar gases are separately collected. The method according to which such high and low carbon pitches are simultaneously produced, together with an intermediate tar or pitch is covered by separate application Serial No. 243,309. The present invention relates to the production of the high carbon pitches regardless of whether the lower carbon tar separately collected from the gases is subjected to distillation or employed for other purposes, but the present invention can in some cases, be advantageously combined with the production of low carbon pitches in accordance with said companion applications.

The high carbon tars separated from coke oven gases near the end of the coking period have a materially higher specific gravity and higher viscosity as well as higher carbon content than the corresponding total tars or the lower carbon tars, and the oils contained in such tar differ from the composite oils contained in the total tar or in the lower carbon tar. Accordingly, when the high carbon tars are subjected to distillation, there is produced not only a new high carbon pitch but also a new distillate. Both the pitch and the distillate are characterized by high specific gravity. The distillate, because of its high specific gravity, is particularly valuable for certain purposes such as the production of creosoting compositions where a relatively high gravity is desirable. The new high carbon pitches are valuable as improved substitutes for high carbon pitches produced from blended tars.

The production of the high carbon tar, from which the high carbon pitch is produced, can be carried out in two-main systems such as have been employed for the production of "rich" and "lean" illuminating gases. It is known that the gases from the early period of the coking operation, after the separation of tar therefrom, are of high B. t. u. value and high illuminating value, and such gases are referred to as "rich" gases. It is also known that the "lean" gases from the latter part of the coking operations, after the separation of tar therefrom, are of low B. t. u. value and are low in illuminants. The tar separated from such gases is, however, collected together as a composite tar. In using such two-main systems, I provide for keeping the tar from the separate mains and separate condensing systems separate from each other so that the high carbon tar from the lean gases will be collected and subjected to distillation separately from the tar from the rich gases. Moreover, the regulation of the operation, and the time during which the rich and lean gases are separately collected, is determined not by the B. t. u. or calorific value or illuminating value of the gases after the tar has been separated therefrom, as in present two-main systems, but is regulated to obtain a tar of the desired high carbon content from which pitch of high carbon content can be produced by distillation.

In the operation of two-main systems, the gases in each of the collector mains may be cooled independently and to a regulated extent by the application of sprays of ammonia liquor, or by the circulation of ammonia liquor and tar, or of tar alone, in much the usual manner. By suitable regulation of the temperature of the gases in the collector main, it is possible to effect the condensation of a relatively heavy tar of high carbon content in one of the collector mains and a relatively heavy tar of lower carbon content in the other. The extent to which the cooling and condensation is carried out in the

collector mains can be regulated to give a greater or less amount of tar therein, but ordinarily the greater part of the high carbon tar will be collected from the collector mains. The gases leaving the collector main can be further cooled in the cross-over main and an additional amount of high carbon tar collected at the end of the cross-over main before the gases enter the condensers. In condensers of the multi-stage type, a plurality of lighter high carbon tars can be separately collected in the condensers. In this way, a fractional condensation and collection of the high carbon tar is possible, and a plurality of separate heavier and lighter tars are produced which can if desired, be combined together to give a total high carbon tar for distillation; or these separate high carbon tars can be separately subjected to distillation for the production of separate products therefrom. The heavy high carbon tar from the collector main, for example, can be distilled with a minimum of distillation to give a high carbon pitch and a relatively low oil yield; while the lighter tar from the condensers will give a relatively much larger proportion of distillate oil and a relatively low amount of high carbon pitch residue.

The distillation of the high carbon tar to produce the new high carbon pitch can be carried out in stills of various types, including those commonly employed for tar distillation. The ordinary externally heated tar stills cause a substantial decomposition of oil constituents during the distillation with resulting increase in the carbon content of the pitch produced, so that the pitch contains a considerably increased carbon content over that contained in the tar distilled. For the production of the higher carbon pitches, the high carbon tars can advantageously be subjected to distillation in ordinary externally heated tar stills, thereby still further increasing the carbon content over that contributed by the high carbon tar itself. Such distillation can be carried out in externally heated stills at the coke oven plants, or the tar can be shipped to tar distillation plants and there separately distilled. When such externally heated tar stills are employed, the oil yield will be reduced because of the decomposition which takes place during the distillation.

Instead of distilling the high carbon tars in externally heated stills, they can advantageously be distilled at the coke oven plant by bringing them into direct and intimate contact with hot coke oven gases at a sufficiently high temperature to effect the distillation. Such distillation can be carried out with the relatively lean gases produced during the later portion of the coking operation in which case the tar separated from the gases and added to the pitch will be tar of the same or similar carbon content, to that produced by the distillation. In some cases, the total gases from several ovens can be employed throughout the coking period in which case the pitch added to the product from the gases may be somewhat lower in carbon content than that produced from the tar distilled. Some decomposition and increase in carbon content of the pitch will take place if the distillation with the hot coke oven gases is carried out at a sufficiently high temperature and with sufficiently prolonged heating of the tar and pitch at a sufficiently high temperature. In distilling the tar to produce a high melting point pitch, for example, around 300° F. melting point, or higher, appreciable decomposition and increase in carbon content will take place. The decomposition, however, will not be as great as that in externally heated tar stills, and a considerably increased yield of distillate will be produced.

The distillate produced from the high carbon tar will be of a distinctive character. It will be of relatively high specific gravity, relatively low in tar acids, and containing constituents differing to a greater or less extent from the constituents contained in the distillate from the low carbon tar produced during the early stages of the coking operation. The distillate from the high carbon tar can advantageously be kept separate and employed as a clean or relatively clean oil for special purposes, such as the production of creosoting compositions where a high gravity is required. By separately distilling the heavy high carbon tar from the collector main and the lighter high carbon tars from the condensers, separate distillates can be obtained and separate high carbon pitch products.

The distillation of the tar at the coke oven plants can advantageously be carried out in a suitable still in which the tar is intimately mingled with the hot coke oven gases. For example, the tar can be sprayed or atomized or otherwise introduced into a short collector main or other still which can be connected to a number of the coke ovens of the battery so that the hot gases therefrom are conveyed through the short main in intimate contact with the tar. The gases from several adjacent ovens can be employed, whether the gases are being given off from the early or later period of coking, or only the ovens can be employed which are near the end of the coking period so that the gases will contain high carbon tar similar to that being distilled. The amount of heat available in hot coke oven gases is several times that required for the distillation of such an amount of tar as is recovered from the gases. Consequently, it will be necessary to utilize the hot gases from a relatively few ovens to accomplish the distillation of all of the high carbon tar produced from the battery.

The gases from the selected ovens are employed at a sufficiently high temperature to effect the distillation of the tar. The tar may be introduced, for example, through spray nozzles into the gases or, in place of or as an adjunct to the spray nozzles, the short collector main may be provided with suitable agitators, such as rotating discs or a rotating cylinder or other agitating device, adapted to be rotated at high speed and to dip into the body of tar or pitch in the bottom of the short main and to bring about intimate and thorough dissemination and atomizing of the tar and pitch into the hot gases passing through the main, or the tar or pitch may be agitated by introduction of compressed coke oven gas beneath the surface. The amount of distillation can be varied by changing the rate at which the tar to be distilled is supplied and the time during which it is exposed to the hot gases, as well as by the intimacy of the contact and the amount and temperature of the gases employed. The tar to be distilled can be supplied to one end of the main and the pitch produced withdrawn from the other and the pitch can be recirculated if desired to increase the extent of the distillation. The atomizing or spraying devices are advantageously such as will break up the tar or pitch being distilled into fine particles and scrub the gases therewith and keep the walls of the distillation vessel washed down with the tar or pitch spray.

Where the heavy high carbon tar and the lighter high carbon tar are separately subjected to distillation by means of the hot coke oven gases, separate stills and separate condensing systems are provided, which permit independent and separate regulation of the distillation of the respective high carbon tars, so that the pitches produced and the distillates produced can be independently regulated; although the distillates can be collected together from the separate distilling operation, or fractionally condensed, to bring similar oils from the separate distillations together. The pitches produced from the separate distillations can also be blended if desired, or can be kept separate, where separate pitches are desired.

The pitches produced may be of higher or lower melting point. For certain road-treating purposes, a high carbon pitch having a melting point around 105° to 110° is required, and having a carbon content of above 15%. Such high carbon pitches of relatively low melting point can be readily produced by collecting together the gases from a sufficient portion of the latter period of coking to give a sufficiently high carbon tar so that upon distillation it will give a high carbon pitch of the desired melting point. Similarly, for higher melting point pitches, such as roofing pitches, of around 145 to 150° F. melting point, where a high carbon content is required, the tar can be so selected as to have the proper carbon content to give on distillation pitches of the required carbon content. Pitches can be produced according to the present invention of much higher carbon content than may be required for certain purposes; and such pitches can advantageously be blended with tar or other pitches to give composite blended products, in accordance with my companion application Serial No. 243,308.

While I have referred particularly to coke oven plants, and to the production of high carbon pitch at such plants, the invention is applicable to other coal distillation plants, such as gas retort plants, etc. At horizontal or inclined gas retort plants, where the tar is a relatively high carbon tar, much higher than coke oven tar, the present invention makes possible the production of pitches of usually high carbon content, by separately collecting the higher carbon tar from the later part of the gas making operation, and subjecting it to distillation, e. g., by direct contact with hot gas retort gases, much as coke oven tar is distilled by direct contact with hot coke oven gases. Similarly, pitches can be produced at other gas retort plants, such as intermittent vertical gas retort plants, which are higher in carbon than ordinary pitches from such gas retort plants, and which more nearly resemble pitches from coke oven tar. Along with the production of the high carbon pitches, new and distinctive disillate oils will likewise be produced.

The process of this invention may be carried out at a by-product recovery system at a coke oven or other coal distillation plant having a plurality of separate gas collecting systems with provision for keeping separate the tars from the gases so as to recover and keep separate the high carbon tar. For distillation of such tar at the coal distillation plant, separate means may be provided for conveying the high carbon tar to the still and for distilling such tar by direct contact with the hot coal distillation gases.

As an example of the application of the invention it may be desirable to produce a high free carbon pitch having a melting point, for example, of about 105° F. (as determined by the cube-in-water method). Such a pitch is suitable for use as a road pitch. Pitch of this character can be produced as hereinbefore indicated at coke oven plants, by separating the gases as they are discharged from the coke evens during the latter part of the coking operation and by cooling such gases to separate the tar constituents which are distilled thereafter by contact with hot coke oven gases. If the proportion of free carbon in the pitch thus recovered is too high for the particular requirements it can be reduced by utilizing ordinary coke oven gases as the distilling media, or a relatively "richer" gas can be selected and the carbon content of the

pitch will be reduced, or, on the other hand, if the content of free carbon is not too high the distillation can be conducted with "lean" gases from the ovens, thus ensuring the maintenance of the desired proportion of free carbon in the product.

It is possible similarly to produce pitches adapted for other purposes as, for example, roofing pitches having a melting point of from 145° to 150° F. (by the water-bath-melting-point method). Such pitches can be produced directly at the coke oven plant with a sufficiently high free carbon content by separating the gases from the later portion of the coking period, by cooling such gases to separate the tar therefrom, and by distilling such tars by intimate contact with hot coke oven gases, either the ordinary gases from the ovens or "lean" gases as may be necessary in view of the desired free carbon content of the product.

It will be understood that in the practice of the invention the cooling of the gases in the separate collector mains and condensing systems will be regulated by supplying more or less ammonia liquor or other cooling agent thereto for the purpose of separating tar with the desired proportion of oil constituents therein and that by regulation of the amount of tar and oil constituents which are condensed in the collector mains and by variation of the amount of distillation to which the tar is subsequently subjected, the characteristics of the pitches obtained can be varied. The proportion of free carbon in such pitches is determined by the character of the tars distilled and to some extent by the distillation, the pitches obtained from the high carbon tar being relatively high in free carbon content; whereas the pitches obtained by the distillation of low carbon tar which is separated from the "rich" gases are relatively low in free carbon content. It will be understood that the recovery of pitches having both low and high free carbon content can be conducted simultaneously in the operation of the same battery and that pitches thus produced can be utilized for any purpose to which they are adapted, although the invention is not limited to such simultaneous production of low carbon pitch, and may be carried out whether or not the lower carbon tar is distilled or employed for fuel or other purposes.

The present invention is of more or less general application to coke oven and other coal distillation plants and to the recovery of pitches and oils from the gases discharged therefrom. It will be further described with reference to the accompanying drawings which show apparatus adapted for the practice of the invention, but it is intended and will be understood that the invention is not limited thereto

Fig. 1 is a plan view of a portion of a coke oven battery illustrating the application of the invention thereto;

Fig. 2 is an enlarged sectional view of one of the collector mains; and

Figs. 3 and 4 are similar views of the short collector main or still in which the tar is distilled.

Referring to the drawings, 5 indicates the battery of ovens which are connected by uptake pipes and goose-necks 6 and 7 to collector mains 8 and 9. Valve boxes 10 and valves 11 therein are disposed in the goose-necks to permit the direction of the hot gases from the ovens into one or the other of the collector mains as may be desired. Thus, by operating the valves "rich" and "lean" gases from the several ovens of the battery can be collected separately in the collector mains 9 and 8 respectively, wherein they are cooled to the desired extent by ammonia liquor, for example, introduced through spray nozzles 12 which are supplied through a pump 13 and pipe 14 from a source of the cooling liquid such as a tank 15. Ammonia liquor may also be sprayed in through nozzles 12′ and will cool the gases and keep the valves 11 clean.

From the collector mains the gases carrying oil vapors and some tarry constituents are delivered through cross-over main 16 and 17 to condensers 18 and 19 wherein the gases are subjected to cooling to separate tarry oils therefrom. The oils may be withdrawn through pipes 20 and 21 and delivered to decanters 22 and 23 wherein ammonia liquor is separated from the oils. The oils and ammonia liquor may be delivered to suitable receptacles 22′, 23′ and 15 for storage and subsequent use. The gases may be delivered through pipes 24 and 25 to exhausters 26 and 26′ which maintain the pressure balance in the system and may pass thence through suitable equipment (not shown) for the recovery of ammonia, light oils, etc.,

In my process the rich and lean gases are kept separate to allow the collection of high and lower carbon tars. I do not divide the gases on the basis of their relative calorific values but on the basis of the carbon content of the contained tars or pitches. Where a total fuel gas is usually collected, the gases may after leaving the condensers 18 and 19 be blended and may be handled by a common exhauster.

The tar and ammonia liquor from the separate collector mains 8 and 9 may be withdrawn through pipes 45 and 27 and delivered to decanters 28 and 29 in which the ammonia liquor is separated from the tar. The ammonia liquor may be returned for further use as a cooling medium or treated in any suitable manner for the recovery of ammonia therefrom. The tars may be delivered through pipes 27′ and 28′ to storage receptacles 29′ and 30 from which they may be withdrawn as required for further treat-

ment such as distillation to produce pitches.

The distillation of the lean gas tar in production of high carbon pitch is effected preferably in a suitable still such as a short collector main or still 31 which may be disposed at the rear of the coke oven battery and is connected to a suitable number of ovens through uptake pipes 32 which deliver the hot gases to the short collector main. The tar to be distilled, for example, tar derived from "lean" gases and, therefore, rich in free carbon, is delivered by a pump 33 through a pipe 34 to one end of the stills. In the box 31 a rotating cylinder 35 is provided, which is rotated at a high speed by the electric motor 36. The cylinder dips into the body of tar or pitch at the bottom of the still and the rapid rotation causes an intimate and thorough dissemination and atomizing of the pitch and tar into the hot gases passing through the still, and cause the desired distillation. The pitch produced is withdrawn through outlet 37 which in Figs. 1 and 4 is shown at the opposite end of the still from the tar inlet although this arrangement is not essential.

The pitch may be withdrawn and carried away thru a pipe or thru an outlet 37 and gutter 38 such as shown in Figure 4.

In general the softer pitches will be withdrawn thru pipes and the harder pitches such as those of 300 to 400° F. melting point and upwards will be withdrawn thru outlets like 37 and gutter like 38. In case the gutter 38 is used the pitch may conveniently be granulated by allowing it to drop into a stream of water from line 37'. The water and granulated pitch will be delivered by the gutter to settling basins 39 from which the pitch may be recovered. In case softer pitches are being made the open gutter is objectionable since it allows excessive fuming of the hot liquid pitch. For that reason a closed pipe line is more desirable.

The hot gases which are partially cooled by contact with the tar and pitch in the short collector main or still are enriched in oil constituents by the distillation effected therein, and are effectively scrubbed by the tar and pitch spray. The gases pass out of the still into settling tower 46, during the passage up which the pitch globules and spray entrained in the gases by the roll spray settle out and are returned back to the still 31, and such scrubbed gases can be withdrawn through a pipe 40 and delivered to a condenser system 41, etc., which may be located in any suitable place, wherein the gases are cooled for the purpose of recovery of the oil constituents. The settling tower 46 may where desired be equipped with baffling devices or with tower packing, not shown, for further assisting in removal of entrained material from the gases. The oil thus separated can be withdrawn from the condensers through a pipe 42 and delivered to a decanter 43 in which it is separated from ammonia liquor. The oil can be stored in a suitable receptacle and utilized for any desired purpose. The cooled gases can be delivered by a pipe 44 to the exhauster 26 and mingled with the other lean gases from the battery, being subjected therewith to the usual treatment for the recovery of ammonia, light oils, etc., therefrom. If rich and lean gases, based on calorific values, are not normally collected and kept separate the gases from 41 may be blended with the rich and lean gases from coolers 18 and 19 to yield a total gas which may then go to the equipment normally used for recovery of ammonia, light oils, etc.

It will thus be seen that the present invention provides for the separate recovery of high carbon tar from coke oven gases from near the end of the coking period and the distillation of such tar by itself to produce therefrom a new high carbon pitch, which differs from high carbon pitches heretofore produced, for example, by blending high carbon gas retort tar with coke oven tar and distilling the blended product. It will further be seen that the invention provides for producing pitches of regulated high carbon content as well as of regulated melting point, and that the distillation can be carried out either in ordinary externally heated tar stills or with special advantage at the coke oven plants, by stills heated directly by the hot coke oven gases into which the tar to be distilled is sprayed or atomized or otherwise brought into intimate contact. It will further be seen that the invention provides an improved method and apparatus as well as improved products, making possible the direct production at coke oven plants of new high carbon tars and new oils having special properties and advantages and being directly merchantable as commercial products, or for use in making commercial products by blending with other products.

While the invention has been more particularly described in connection with coke oven operation and the production of new and valuable pitches at coke oven plants, the invention includes the similar production of new and distinctive high carbon pitches in a similar manner at other coal distillation plants such as horizontal and inclined gas retort plants, intermittent vertical retort plants, etc.

I claim:

1. The method of producing pitch of high free carbon content from coke oven and other coal distillation gases, which comprises collecting and withdrawing the gases produced during the later stages of the coal distillation in the ovens or retorts, independently of the gases produced during the earlier stages of distillation, keeping the gases from the later stages of distillation separate and cooling the

same to separate tar of high free carbon content therefrom, keeping the resulting tar separate and distilling the same to produce pitch of high free carbon content therefrom.

2. A new high carbon pitch having a melting point of around 105 to 110° F., and a carbon content of around 10 to 15%, such pitch being a coke oven tar pitch resulting from the distillation of high carbon tar separated from coke oven gases during the later portion of the coking operation.

In testimony whereof I affix my signature.

STUART PARMELEE MILLER.