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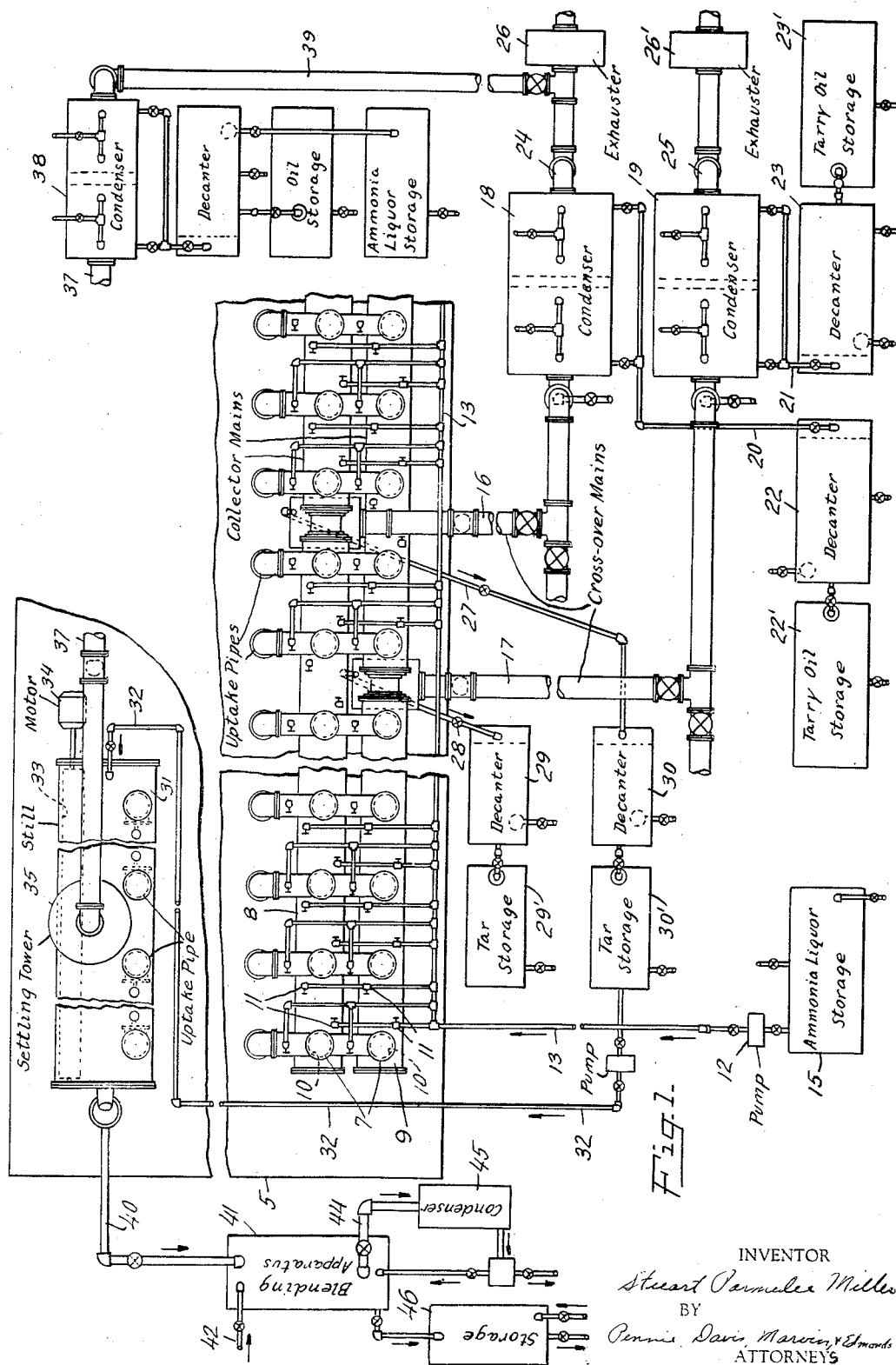
S. P. MILLER

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LOW CARBON PITCH COMPOSITIONS

Filed Dec. 29, 1927

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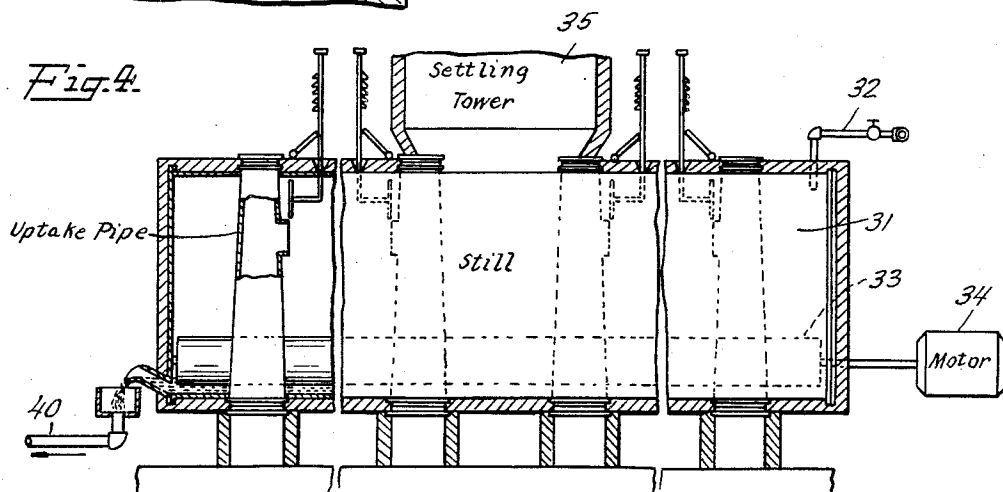
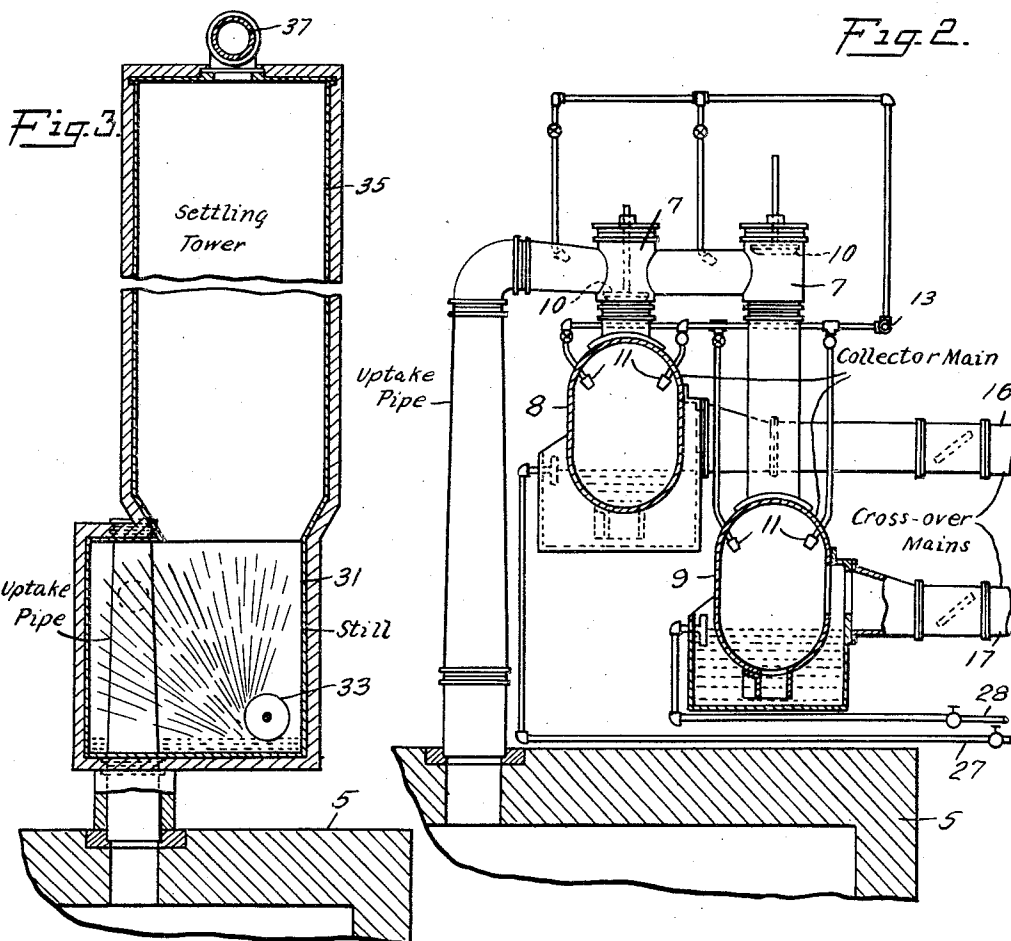
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LOW CARBON PITCH COMPOSITIONS

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3 Sheets-Sheet 2



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LOW CARBON PITCH COMPOSITIONS.

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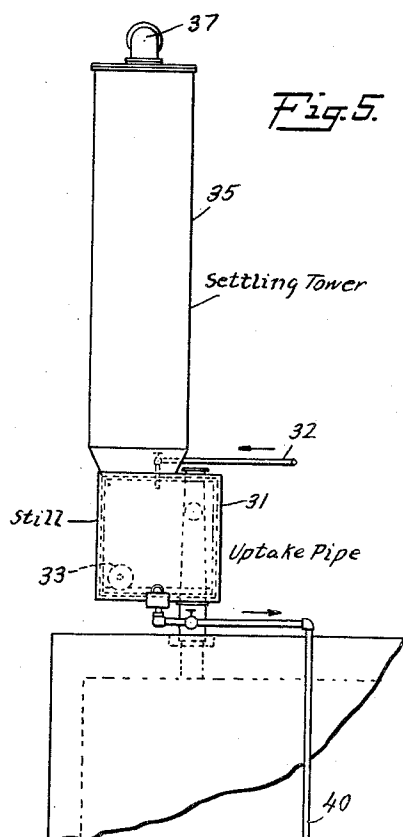
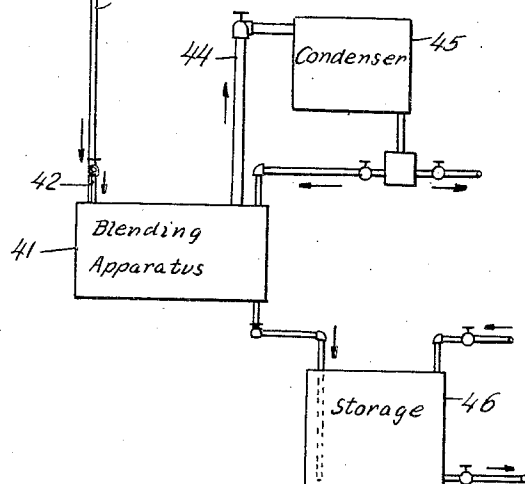
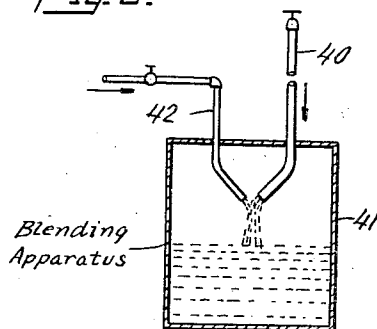


Fig. 6.



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UNITED STATES PATENT OFFICE

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LOW CARBON PITCH COMPOSITIONS

Application filed December 29, 1927. Serial No. 243,306.

This invention relates to the production of low carbon pitch compositions. More particularly, the invention relates to the production of new pitch compositions at coke oven and other coal distillation plants and from coke oven and gas retort tars.

In the ordinary operation of by-product coke oven and gas retort plants, the gases from the ovens or retorts are cooled to separate tar therefrom and the tar is shipped to tar distillation plants and there distilled to produce distillate oils and pitch residues.

The pitches produced by the distillation of coke oven tar, for example, have a content of free carbon too high to permit them to be employed for certain purposes where a low content of free carbon is required, and, for the production of low carbon pitches, it is customary to employ tars from other sources, particularly water-gas tar, either alone or blended with coke oven tar before the mixture is subjected to distillation.

The present invention provides for the production of new composite pitch compositions of low carbon content directly at coke oven and other coal distillation plants and from coke oven or gas retort tar.

In referring to content of carbon or free carbon in tar and pitch I use these terms in their ordinary significance to mean the ingredients remaining insoluble on digestion of the tar or pitch with benzol or other similar solvent, and to include not only elemental or molecular carbon but also insoluble inorganic impurities and insoluble hydrocarbons of high molecular weight.

According to the present invention, a low carbon tar is separated from the coke oven or other coal distillation gases given off from the individual coke ovens or gas retorts during the early period of the coking or gas-making operation and this low carbon tar is subjected to distillation to form a relatively high melting point but low carbon pitch, and this pitch is blended with additional tar to form a composite blended pitch product of regulated low carbon content.

In my companion application, Serial No. 243,305, filed December 29, 1929, I have described the production of low carbon pitches

by separately collecting low carbon tar from the gases given off during the early portion of the coking or gas-making operation at coke oven or gas retort plants and distilling such low carbon tar to produce low carbon pitches of varying melting point and of low carbon content.

The present invention is distinguished from the invention of said companion application by the production of low carbon pitches of higher melting point than desired in the final product, and the blending of such high melting point low carbon pitches with additional tar or softer pitch to form a composite blended product.

The production of the low carbon tar and pitch and the distillation of the low carbon tar to make the pitch can advantageously be carried out in accordance with the process of my said companion application, but the distillation is carried to a point such that the pitch produced is of a much higher melting point than desired in the final product.

While the low carbon pitch can be distilled in various kinds of tar stills, including the ordinary externally heated tar stills, it can more advantageously be distilled by bringing it into direct and intimate contact with hot coke oven or other coal distillation or gasification gases, since such distillation can readily be carried out at the coke oven or gas retort plant or at the water gas plant and is accompanied with much less decomposition of oil constituents and less increase in carbon content than when the distillation is carried out in externally heated tar stills. The distillation of the tar at the coke oven or gas retort plant by means of hot coal distillation gases is also advantageous in that it permits the tar to be continuously distilled and pitch of regulated melting point produced and the hot pitch to be utilized directly for blending with the tar or softer pitch, which tar or softer pitch may also be produced and employed in a heated condition.

The extent to which the distillation is carried can be varied, and will depend upon the nature of the composite products to be produced. Pitches of intermediate melting point

can be produced of melting points for example, around 165° to 225° F., determined by the cube-in-air melting point method, but pitches can advantageously be produced of lower melting point and likewise of much higher melting point, around 250 or 300° F. or even around 400° F. or higher, since the low carbon tars lend themselves to distillation, in contact with hot coal distillation gases, to produce pitches of such high melting point. The production of high melting point pitches has the advantage that it gives a high yield of heavy oils which are recovered as valuable products of the process.

In my companion application, Serial No. 243,307, filed December 29, 1929, I have described the production of high carbon tar from the gases given off near the end of the coking or gas-making operation and the distillation of such tar for the production therefrom of high carbon pitches; and in my companion application Serial No. 243,308, filed December 29, 1929, I have described the distillation of the high carbon tars to produce high carbon pitches of higher melting points than that desired in the final products, and the blending of such high melting point high carbon pitches with tar for the production of composite pitch products of regulated high carbon content. The improved products of the present invention can be produced at the same time as the processes described in my said companion applications, although the present invention is of broader application and the low carbon pitches can be produced in cases where a high carbon pitch is not simultaneously produced.

In the practice of the invention at coke oven plants, for example, the plant may advantageously be provided with two collector mains, both united to the same coke ovens, and with valves for connecting each oven with one of the other mains so that the gases can be collected from the various ovens in one main during the desired initial period of operation in order to collect therein the gases from which low carbon tar is condensed, leaving the gases from the later period of operation to be collected in the other collector mains to give tar of higher carbon content. The tars from the respective collector mains as well as from the separate condensing systems, are kept separate and the low carbon tar is subjected to distillation by itself, using either the heavy tar from the collector main or the lighter tar from the condensers or combining the heavy and the lighter tars to form a composite low carbon tar which is then subjected to distillation.

The higher carbon tar simultaneously separated from the other collector main may be employed as tar or may itself be subjected to distillation for the production of high carbon pitches therefrom. The operation of the collector mains and of the condensers may be

conducted in accordance with the usual practice by circulating ammonia liquor, or ammonia liquor and tar, through the collector mains, and by cooling the condensers either directly or indirectly to separate the lighter tars or tarry oils from the gases; but in regulating the operation of such systems, the gases will be collected in one or the other of the collector mains, depending upon the carbon content desired in the tar, and only such portion of the gas from the early period of operation will be collected together as will give a tar of sufficiently low carbon content so that upon distillation it will give a low carbon pitch suitable for use for blending with additional tar or soft pitch in the practice of the invention. The gas may be collected, for example, only during the initial third or quarter of the coking operation, although in some cases it may be collected during the first half or more of the coking operation, depending upon the carbon content required in the low carbon pitch composition.

The distillation of the low carbon tar is advantageously carried out in short collector mains or stills into which the gases from a plurality of ovens are directly led at a high temperature and into which the tar is introduced and brought into intimate contact with the gases. The tar may be sprayed into the gases, or the operation can be promoted by providing mechanical agitating devices such as rotating discs, or rotating rolls or rotating paddles operating at a sufficiently high speed to effectively atomize or spray the tar and pitch into the gases to secure rapid and effective distillation, and to wash down the surfaces of the main. This distillation can be carried out continuously, to produce continuously high melting point low carbon pitch, which is drawn off at a high temperature and advantageously blended while still hot with the tar to produce the composite composition.

The tar or softer pitch which is blended with the high melting point pitch may itself be a low carbon tar or pitch such as that from which the pitch was produced, and in this way a pitch of minimum carbon content can be produced. Where such a minimum carbon content is not required, a tar of higher carbon content can be employed, such as ordinary coal tar comprising the total tar from the coke oven operation, or even in some cases high carbon tar to a greater or less extent. So also, I do not exclude the blending with the new high melting point low carbon pitches of other tars than coke oven tars, since in some cases low carbon tars such as, for example, water-gas tar, or vertical retort tar, or low carbon pitches from water-gas or vertical retort tars, may be included in the composition. The blended pitch composition will vary in properties and characteristics depending upon the nature of the tar employed,

as well as the proportions of the tar and pitch, and the melting point and carbon content and other properties of the pitch itself.

The blending of the high melting point pitch with the tar is advantageously accomplished by introducing continuous streams of the hot thinly fluid pitch and of the tar to be blended therewith into a mixing chamber. The tar itself may be hot or may be preheated to dehydrate it or to remove lighter oils therefrom. The heavy tar from the collector mains may be employed while still hot, or other tar may be separately preheated or distilled to a greater or less extent to give a heavier tar, or soft pitch, partly freed from lighter oils, for the blending operation. By introducing continuously streams of the pitch and tar into a suitable receptacle, such as a nearly empty tank, the streams will blend with each other and produce a uniform blended product. Such a blending operation is desirable if the tar which is introduced contains water, since the temperature of the pitch will ordinarily be so high as to cause rapid vaporization of the water and foaming of the mixture, and the use of such a large receptacle permits the water vapor and other light oils to be removed conveniently, and avoids any objection from foaming. The heat of the hot thinly fluid pitch results in giving a heated mixture, and promotes the mixing operation, as well as the dehydration and in some cases the partial distillation of the tar. The blending may of course be done batchwise.

The vaporized water and oil constituents, vaporized during the mixing of the pitch and tar, can be withdrawn continuously from the mixing receptacle, and the oil and water then condensed, and the oil separated from the water by providing a suitable condenser. The blended products can be withdrawn continuously from the mixing receptacle and delivered to suitable storage containers. The heat of the hot pitch will under most circumstances be sufficient to give a resulting hot mixture sufficiently fluid to permit it to be drawn off and handled as a liquid product, without difficulty in handling, although the composite product after cooling may be of a solid character. The tar or soft pitch may be heated, if necessary, before blending to secure fluidity of the final fluid.

The proportions in which the high melting point low carbon pitch and the tar are admixed can of course be varied, depending upon the melting point of the pitch and the melting point and other properties of the blended product. For low carbon pitches having a melting point of around 160-170° F. (by the cube-in-air melting point method) such as are desired for saturating fibre conduits for telephone cables, the distillation of the low carbon tar can be carried to a point considerably above that of the pitch desired,

for example, to 225° F. or even to 300° F. or higher melting point, and the resulting pitch then blended with tar, either raw tar or tar which has been subjected to preliminary distillation to remove more or less of the lighter oils, to give a blended product having the proper melting point and other properties.

Such blended low carbon pitch compositions are distinguished from low carbon pitches produced by the distillation of low carbon tars without blending. The blended pitches contain a proportionally greater content of the heavier pitch constituents, such as those remaining after the distillation of the tar to produce the high melting point pitch. Such compositions contain relatively less of the intermediate products which are removed from the pitch during its production; and such composite pitches commonly contain somewhat more of the lighter oils present in the tar and which are removed during the distillation to produce a low carbon pitch of similar melting point to that of the blended product.

The present process is likewise differentiated from the process of my companion application, Serial No. 243,309, filed December 29, 1929, by the carrying out of the distillation to produce a low carbon pitch of higher melting point than that desired, with simultaneous production of a high yield of heavy distillate oils, and the blending of the high melting point pitch with additional tar, which has not been subjected to distillation, or which has been distilled only to a limited extent. The present process enables a much smaller amount of tar to be distilled, but distilled to a much higher melting point pitch and with increased yield of high boiling point oils, and it enables a considerable amount of tar to be employed without subjecting it to distillation for the production of pitch therefrom.

As an example of the practice of the invention, the gases from the first third, quarter or half of the coking period of a coke oven plant can be cooled in the separate collector main or mains by ammonia liquor, or ammonia liquor and tar, to separate a heavy low carbon tar from the collector main, and the gases can then be further cooled in the condensers to separate a lighter tar of low carbon content therefrom. The heavy tar alone or the lighter tar alone or both tars admixed together, can then be subjected to distillation in a separate short collector main or distillation tank, into which the hot coke oven gases from several of the ovens are directly introduced, and in which the tar is brought into intimate contact with the hot gases. The distillation can be carried up to the point of producing, for example, a pitch of 165° to 225° F. melting point or even up to a point of producing a pitch of 300° F. melting point or 400° melting point or even

higher in some cases. The production of the higher melting point pitches is accompanied with some decomposition and increase in carbon content, but with the low carbon content of the tars employed, the pitches, even of high melting point, are still relatively low in carbon content. For the production of the higher melting point pitches, it is advantageous to omit spray nozzles in the distillation box or main and to employ a mechanical agitating device, such as rotating discs or rotating rolls or paddles, rotating at a sufficient speed to atomize the tar or pitch into the gases to effectively scrub the gases and bring the tar and pitch into intimate contact therewith.

The resulting high melting point pitch of low carbon content can then be drawn off continuously and supplied continuously to a mixing tank into which a supply of tar is also continuously added, and in which the blending of the pitch and tar takes place to give a resulting hot mixture of a homogeneous character which then may be drawn off to storage tanks or to other place of shipment or of use.

The invention can be applied in connection with the by-product recovery systems of existing coke oven plants particularly those which have two collector mains and two condensing systems for the separate production of "rich" gas and of "lean" gas for fuel purposes. In such systems, as now operated, the tars from the respective mains are collected together as a composite product, and the separate collection of the rich and lean gases depends upon the B. t. u. or illuminating value of the gases after the tar has been separated from them. The present invention provides for the separate collection of the tar from the rich and lean gases, and the regulation of the rich and lean gas mains is conducted from the standpoint of the character of the gas after the tar has been separated. In such plants, a separate distillation main or still is provided for bringing the tar into intimate contact with the hot coke oven gases at a sufficiently high temperature, and a mixing device or chamber is provided for mixing the resulting high melting point pitch with additional tar to form the composite product.

The provision of separate collector mains and separate condensing systems such that the high and low carbon tars can be separately collected and kept separate, and so that the heavier and lighter tars can be separately collected and kept separate, enables the low carbon tars in part or in whole to be distilled for the production of the high melting point low carbon pitches, and gives a variety of tars for blending with the resulting pitches, these tars including the heavy low carbon tar itself, the lighter low carbon tar, and the heavier and lighter high carbon tars, or a blend or

mixture of these in any desired proportions.

The present invention is of particular value for the production of low carbon pitch composition at coke oven plants from coke oven tar but in its broader aspects it is applicable similarly to other coal distillation plants. Gas retort plants can be operated in a similar manner with segregation of the gases given off during the early portion of the gas making operation in the individual retorts and separation of a relatively low carbon tar from such gases, with distillation of such low carbon tar to produce low carbon pitch and blending of such pitch with additional tar. The low carbon pitches from horizontal or inclined gas retort plants will be higher in free carbon than the low carbon pitches similarly produced at coke oven plants and will more nearly resemble ordinary coke oven tar pitches. The low carbon pitches from intermittent vertical retorts will be unusually low in carbon content. The various pitches can be blended in varying proportions with different tars, or even with other pitches to give blended products of regulated low carbon content. The oils produced by distillation of the low carbon pitches will vary with the low carbon tars distilled and themselves form valuable products of the process. The process makes possible the production of increased yields of higher boiling oils from the tar utilized, while leaving increased amounts of low boiling oils in the undistilled tar blended with the high melting point pitch.

The invention will be further described with reference to the accompanying drawings which show apparatus adapted for the production of the new pitch compositions.

In the accompanying drawings

Fig. 1 is a plan view of a portion of a coke oven battery;

Fig. 2 is a cross-sectional view of one of the collector mains;

Figs. 3 and 4 are enlarged sectional views of the short collector main or still; and

Figs. 5 and 6 are details in section of the blending apparatus.

Referring to the drawings, 5 indicates a battery of ovens which are connected by uptake pipes and goose-necks to valve boxes 7 which communicate with collector mains 8 and 9. Valves 10 are arranged in the valve boxes to permit the direction of the hot coke oven gases into one or the other of the collector mains depending upon the stage of the coal distillation operation in the particular oven to which the valve box is connected. Thus, by operating the valves "rich" and "lean" gases from the several ovens of the battery can be collected separately in the collector mains wherein they are cooled to the desired extent by ammonia liquor introduced through spray nozzles 11 which are supplied through a pump 12 and pipe 13 from a source of ammonia liquor such as a tank 15.

From the collector mains the gases carrying oil vapors and partially condensed tarry constituents are delivered through cross-over mains 16 and 17 to condensers 18 and 19 in which the gases are further cooled to separate the oil constituents. The tarry oil together with ammonia liquor can be withdrawn through pipes 20 and 21 and delivered to decanters 22 and 23 wherein the ammonia liquor is separated from the oils and delivered to storage tanks for subsequent recovery of ammonia therefrom. The oil can be delivered to suitable storage receptacles 22' and 23' and the ammonia liquor can in part be returned if desired for further use as a cooling medium either in the collector mains or in the condensers. The excess will be worked up for recovery of ammonia therefrom. The gases escaping from the condensers are delivered through pipes 24 and 25 to exhausters 26 and 26' and thence to equipment (not shown) for the separation of ammonia, light oils, etc.

The tar and liquor which separated in the collector mains can be withdrawn through pipes 27 and 28 to receptacles 29 and 30 in which tar is separated and withdrawn to receptacles 29' and 30'. The tar from the "rich" gas main contains a relatively low proportion of free carbon and this tar is subjected to distillation in a suitable still, such, for example, as a short collector main or still 31 which is disposed preferably at the rear of the ovens and is connected to a plurality of ovens so that hot coal distillation gases pass continuously through the short collector main or still. The tar to be distilled is introduced into the main or still 31 at one end through pipe 32. The main or still is provided with a cylinder or roll 33 adapted to be rotated at a high rate of speed by electric motor 34. The roll dips into the tar and pitch in the bottom of the main or still and the rapid rotation atomizes the tar and pitch, in the form of finely disseminated spray, into the gases passing through the main or still. In thus mingling with the hot gases the tar is distilled to produce pitch and the volatile constituents are separated and join the gases which pass up through setting tower 35, during which passage the tar spray and globules entrained in the gases settle out and are returned to the main or still 31. The gases may be withdrawn continuously through a pipe 37 and delivered to a condenser 38 located at any convenient place wherein the oil constituents can be separated from the gases. Thereafter the gases may be delivered through a pipe 39 to the exhauster and mingled with the remaining gases from the ovens of the battery.

The pitch produced in the short collector main or still can be withdrawn continuously through a pipe 40 and delivered to the blend-

ing apparatus 41. The tar or other material to be blended with the pitch is similarly introduced through a pipe 42, the two pipes being directed so that the streams are mingled as they enter the chamber. The tar and pitch thus blend to produce a product having the desired characteristics which can be withdrawn continuously through a pipe 43 and delivered to suitable storage receptacles 46.

Owing to the relatively high temperature of the pitch the tar which is blended therewith will be heated and any moisture present therein, together with volatile oil constituents, will be vaporized immediately upon contact between the tar and pitch. The vapor can be withdrawn as it is released through a pipe 44 and delivered to a condenser 45 wherein it is cooled. The oil thus recovered may be mixed with oil obtained by the cooling of the other gases from the system or it may be returned to the pitch in 41.

In a similar manner the new pitch compositions of the invention can be produced at other coal distillation plants, particularly gas retort plants, where a similar segregation of the gases and production of low carbon tar and pitch can be carried out with blending of the high melting point low carbon pitch and additional undistilled or partly distilled tar to form various blended pitch compositions of regulated carbon content and other properties. It will be understood that various changes may be made in the details of the operation and in the apparatus and process and in the character of the composite products produced and that the invention makes possible a wide range of products which can be regulated to a greater or less extent by regulating the carbon content of the tar distilled, the extent to which the distillation is carried, the kind of tar blended with the pitch and the proportions of the blended ingredients, etc.

I claim:—

1. New blended pitch compositions, comprising low free carbon pitch of high melting point blended with coal tar, such pitch resulting from the distillation of low carbon tar separated from the gases produced during the early period of operation of the ovens of a coke oven or gas retort plant.

2. New low carbon pitch compositions, comprising a blend of low carbon coal tar and of high melting point pitch resulting from the distillation of such tar, the low carbon tar being obtained from coke oven or other coal distillation gases during the early period of the coking or gas making operation.

In testimony whereof I affix my signature.
STUART PARMELEE MILLER.

CERTIFICATE OF CORRECTION.

Patent No. 1,844,899.

Granted February 9, 1932, to

STUART PARMELEE MILLER.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 1, line 49, and page 2, lines 16 and 23, for the year "1929" read 1927; page 3, lines 93 and 94, for "Serial No. 243,309, filed December 29, 1929" read Serial No. 243,305, filed December 29, 1927; and that the said Letters Patent should be read with these correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 17th day of May, A. D. 1932.

(Seal)

M. J. Moore,
Acting Commissioner of Patents.