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DISTILLATION OF TAR AND RECOVERY OF PRODUCTS THEREFROM

Filed April 20, 1927

2 Sheets-Sheet 1

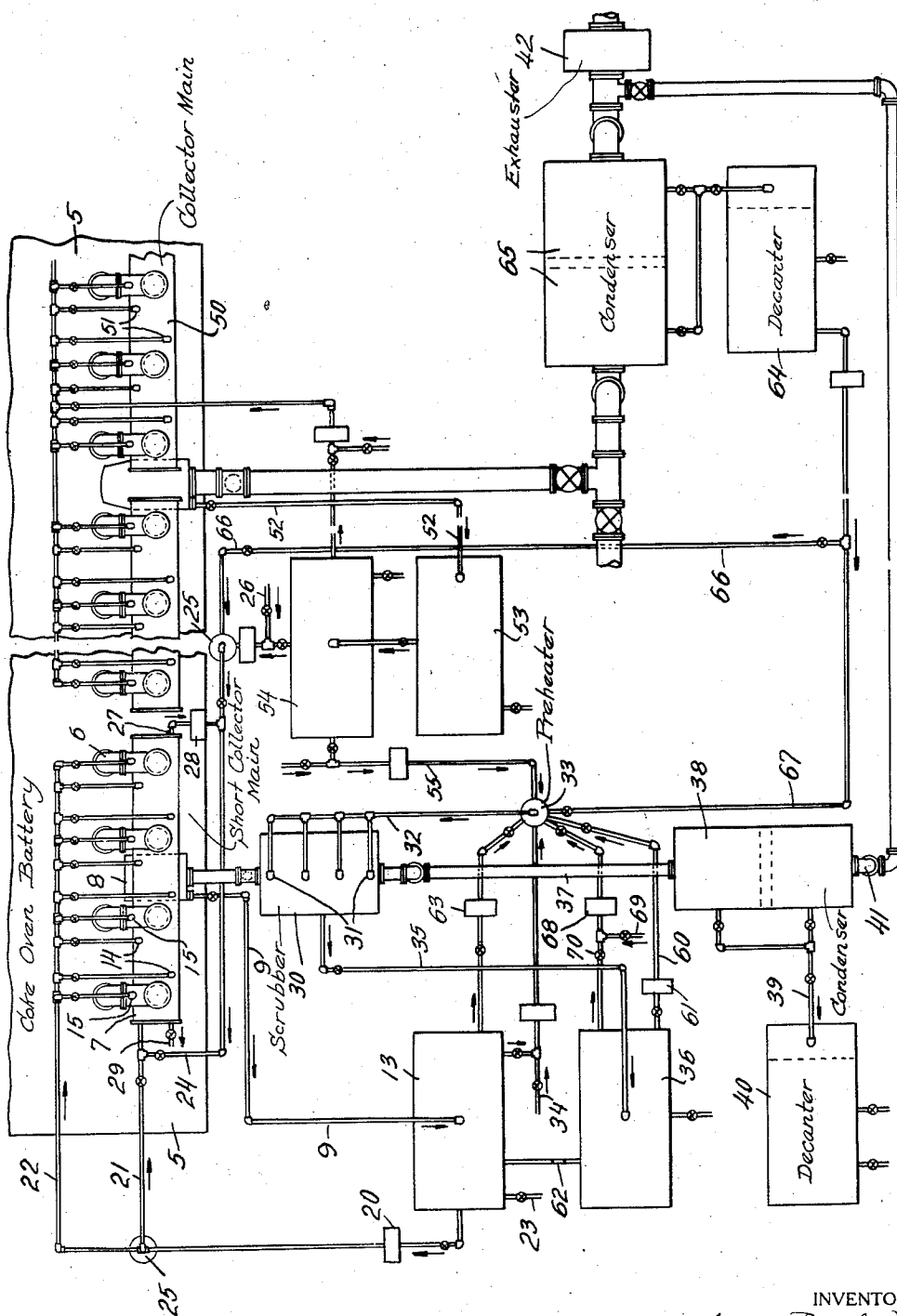


Fig. 1.

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Fig. 2.

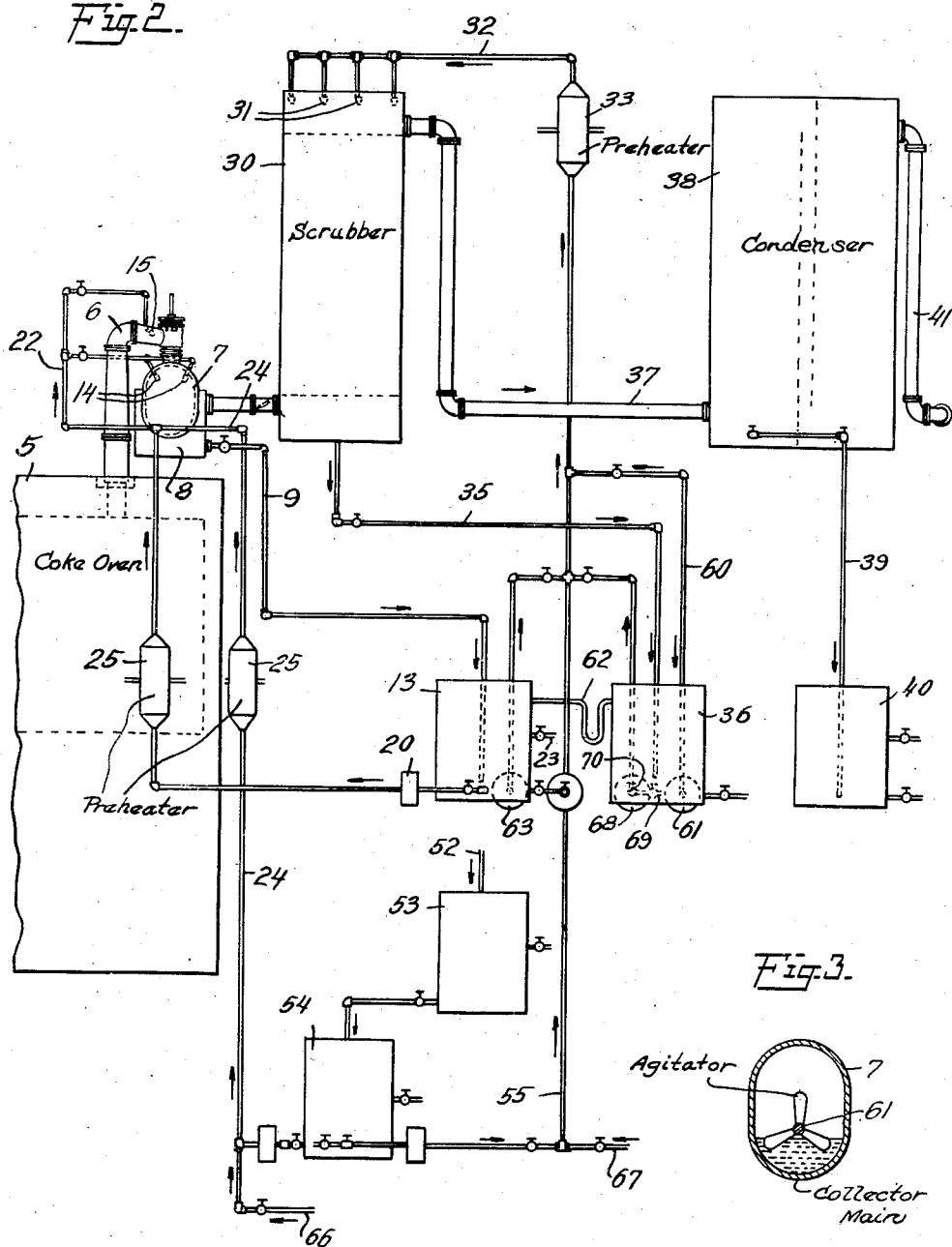
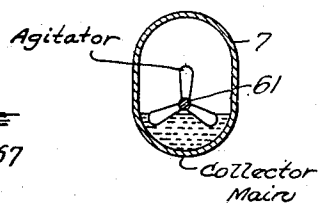


Fig. 3.



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DISTILLATION OF TAR AND RECOVERY OF PRODUCTS THEREFROM

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This invention relates to improvements in the distillation of tars and oils, particularly to the utilization of the heat available in the gases produced in coal carbonization plants to distill coal gas condensates, and to the production of oils and tars containing only a small percentage of insoluble impurities. The invention will be described especially with reference to its application to coke ovens.

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 commonly known as "foul" gases leave the ovens at high temperature, e. g., 600° to 700° C. or higher and carry a considerable proportion of volatilized and entrained tars and oils as well as solid particles of coke, coal and carbon and other impurities. Ordinarily, the gases are cooled 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 heat in the gases being thereby dissipated and lost. The rapid cooling causes separation of tar containing heavier oils in the collector main. Further cooling is effected in the cross-over main which connects the collector main to the condensing system. An additional quantity of tar carrying both heavier and lighter oils is recovered in the cross-over main and in the condenser. The collected tar is ordinarily shipped from the coke-oven plant to a tar distillation plant for distillation and separation of the oils and the production of pitches of varying qualities. Handling losses, freight charges and distillation costs, including fuel and capital and maintenance expenses for the special equipment required for distillation add to the cost of production of the tar distillation products.

It is the object of the present invention to provide a method for the distillation of tar at a coke-oven plant or other coal carbonization plant, the utilization of the heat of the coke-oven gases for that purpose, and particularly the production of salable oils directly by such distillation together with pitches having the

various qualities and characteristics required by the trade.

According to the usual practice, the condensates obtained in the operation of coke ovens are collected as tar and contain considerable amounts of dust, coke braize and free carbon. It is one of the purposes of this invention to treat the hot coke oven gases in such a manner as to allow the subsequent direct recovery of salable distillate oil fractions and salable pitch, thus avoiding the necessity for distillation of tar as ordinarily carried out.

On cooling coke oven gases, according to the ordinary methods, a fog or mist of tar particles is produced. Due to the extremely small size of these particles, the tar fog persists in large quantities in the gases even after cooling to atmospheric temperatures. Hence, oily fractions recovered by stepwise cooling of the gases have been so contaminated with tar that the fractions have not been salable as oils but have been utilized as tars. It has been necessary to distill them for production of salable oils. The residue from the distillation has been pitch.

According to this invention, salable oils may be recovered directly from the gas stream. The heat of the gases from a coal carbonization process is utilized in volatilizing tar or oil, the volatile portions of which are recovered as marketable products at a subsequent step of the process. By enriching the gases in condensable constituents which are subsequently recovered as salable products, the percent of entrained tar fog and solid impurities in the gases, using the condensable constituents as a basis for figuring the percentage, is less than the percent of entrained tar fog and solid impurities in the same gases not thus enriched with condensable constituents, using the condensable constituents in the gas as a basis for figuring the percentage.

The process of this invention comprises two operations. The first operation involves enriching the gases with condensable constituents, and the second operation comprises scrubbing the enriched gases. Scrubbing removes entrained tar and solid impurities to a substantial degree. Both operations com-

bine to reduce the percentage of impurities in the products and make possible the recovery of marketable products with a low percentage of insoluble impurities direct from the gas stream.

According to the present invention, the coke oven gases while at high temperature are utilized to distill tar or oils brought directly in contact with them. The gases are greatly enriched in condensable constituents and are simultaneously somewhat cooled. The gases, while still at high temperature, are then passed through any one of a number of suitable scrubbing devices in which they are brought into intimate contact with tar or oil. In the first operation, i. e., the distillation operation, a portion of the tar fog normally present in the gases is thrown down with the tar or oil brought in contact with the gases and is removed as the residue from the distillation. Further tar fog is removed in the second or scrubbing operation.

The material used in the distillation operation may be a tar, a pitch or an oil. These materials may be those recovered from the coke ovens, or they may be materials from another or other sources. Likewise, in the second or scrubbing operation tar, oil or pitch from the coke ovens may be used, or materials from another or other sources may be employed. The amount of distillation, and hence the extent of the cooling of the gases, may be regulated by controlling the amount, temperature of and character of the tar, pitch or oil distilled. Likewise, the character of the residue from the distillation and the degree and character of gas enrichment will vary with amount, temperature of, and character of tar, pitch or oil distilled. In the scrubbing operation, the gases may be scrubbed with tar, pitch or oil. Depending upon the character of the scrubbing material, its temperature and its amount, the gases will be (1) cooled, (2) held at the same temperature, or (3) heated; the degree and character of enrichment will be decreased, held constant or increased. In some cases, the degree of enrichment may be held constant but the character of the enriching medium may be entirely altered as in the case in which the hot gases are scrubbed with a low boiling oil. In such a case, high boiling constituents in the gases will be precipitated and will be replaced by lower boiling products, the extent of this replacement being determined by the regulation imposed on the system.

The gases leaving the second process, i. e., the scrubbing, will upon stepwise cooling yield fractions so low in tar or free carbon that they are salable as oils, rather than as tars. According to the preferred form of the present invention, the scrubbing is effected by a scrubbing device located in the hottest part of the cross-over main near the exit from the collector main. At this point the gases are

hot and by the scrubbing solid matter and entrained tar can be removed without condensing any considerable amount of the heavy oils and tars. By regulating the temperature of the gases at this point and by regulation of the temperature of the spray of the scrubber, the quality of the tar or pitch recovered from the scrubber may be regulated. If the gases are very hot and a hot spray is used, a heavy pitch is recovered and heavy constituents of the gases from which free carbon, etc. have been removed are thrown forward into the coolers where heavy constituents, as well as lighter constituents, are recovered in a readily marketable condition.

The temperature of the gases may be regulated by regulating the spraying to which the gases are subjected prior to their entrance into the cross-over main. According to the usual practice, the gases are sprayed with water or ammonia liquor in the collector main. Because of the high latent and specific heats of water and ammonia liquor, it is desirable for the purposes of this invention to spray with tar or tar constituents in the collector main instead of the usual aqueous solution, inasmuch as by this procedure the temperature of the gases is not lowered to the same extent as would result from spraying with the same quantity of an aqueous solution. Likewise, the vapors are enriched by volatilization of the volatile contents in the tar or oil sprayed into the collector main. By decreasing the amount of tar or oil sprayed into the collector main, or by preheating the spray, the temperature of the gases entering the cross-over main may be raised above the usual temperature. By this procedure the quantity of heavy constituents condensed in the collector main is kept at a minimum. Condensation of the constituents can likewise be retarded by diluting the vapors with some inert gas.

The heat of the gases leaving the coke oven is sufficient to distill a quantity of tar or oil many times that produced by the oven itself. In this invention, these additional tars or oil may be volatilized by being added to the system as fluids with which to flush the collector main. They may be added to the main in excess of the quantity required to flush the main and if desired, recirculated through the main until the lighter constituents have been volatilized. They may be introduced into the gases through spray nozzles. By preheating these fluids, the capacity of the system for recovering pure distillates is increased.

In order that the heat of the gases passing through the collector main may be utilized to the greatest extent, the materials to be distilled should be brought into intimate contact with the gases.

It may be advisable to provide some means for agitating the non-volatilized constituents which flow through the main. This may be accomplished by providing a rotating

shaft running the length of the main on which are paddles or disks or other spray devices. Agitating means as cone sprayers may be inserted through the top of the collector main. Suction T's (McDaniel type ejectors) may be located in the main with their suction ends beneath the level of the tar or pitch therein. When a gas under pressure is supplied to such devices, the tar or pitch or oil in the main will be thrown into the stream of gases in the main and distillation of these products will be accomplished.

When it is desired to utilize the features of this invention to the utmost, the heat of the coke oven gases will be used most efficiently to produce the maximum of enrichment of the gases that can be obtained. In order to accomplish this, the heat of the coke oven gases is utilized to the fullest extent by treating the gases with volatile oils or tars, instead of by cooling the gases with water or ammonia liquor as is the customary practice.

The scrubbing medium in the second step of the invention may be made to serve a double purpose. It serves to remove entrained tar and solid particles from the gas. In addition to this, by volatilization of the light constituents of the scrubbing medium the gas may be further enriched, and a heavier tar, oil, or pitch may be produced at the scrubber. The volatilized constituents may subsequently be recovered as salable materials by cooling of the enriched gases.

Depending upon the quality of the oil, tar and pitch which it is desired to recover, the nature of the recoverable constituents added to the process may be varied. Where several batteries of coke ovens are operated in one plant, it is possible to equip one battery with means such as is described in this application and to supply to this one battery the tars and pitches or a portion of the tars and pitches obtained in the operation of the other batteries; or it is possible to equip only a portion of the ovens of one battery with a separate collector main which leads to a separate cross-over main, in which a tar scrubber is provided, and by equipping only a portion of the ovens of the battery in this way utilize the oils, tars and pitches, or a desirable fraction of the oils, tars and pitches produced in the remainder of the battery in the operation of the few ovens equipped with the separate collector main and scrubber.

By adding only the lighter fractions of tar and oils recovered from the operation of other ovens to the gases from ovens equipped with the scrubber, a greater portion of light oils will be recovered, and, conversely, by adding a preponderance of heavier fractions a greater quantity of oils and tars or pitches of high boiling point will be recovered. In-

stead of coke oven by-products, tar such as gas house tar, vertical retort tar, or water gas tar, etc., or tars or oils from other sources can be distilled, and when blended or composite pitches or distillates are desired, several different kinds of tars, oils or pitches may be admixed in suitable proportions, and oil, tar or pitch of the desired composition may be obtained directly. For example, horizontal gas retort tar high in free carbon will yield a composite pitch higher in free carbon than the pitch from coke oven tar together with a marketable composite distillate.

The tar, oil or pitch used in the collector main or scrubber may be preheated in any suitable manner to reduce it to a thinly fluid state and thus facilitate spraying into the hot coke oven gases and increase the capacity of the gases for distillation.

It is possible to utilize the invention for the distillation of contaminated oils to recover therefrom clean salable products.

The enriched gases are washed in the scrubber and this may be accomplished at a period in the recovery process when the heavier oils are still in the vapor phase. It is generally advantageous to keep the tar in the scrubber at a temperature above the dew point of the gas for the oil which it is desired to recover subsequently in the condenser system. If cool tar, or tar lower in temperature than the gas stream, is used, low boiling as well as high boiling constituents may be thrown down into the scrubbing medium, the gases may be impoverished in condensable constituents and smaller quantities of salable products will be subsequently recovered than will be the case when a scrubbing medium of higher temperature is used.

By controlling the temperature of the gases and the temperature and amount of the tar supplied to the scrubber and the temperature and amount of the constituents added in the collector main, the amount and the character of salable oils subsequently recovered, as well as the nature of the product produced at the scrubber, can be varied.

The enriched and somewhat cleaned gases produced by this process may be condensed in any desirable manner. It is a feature of this invention that by operating with the collector main hot and using a hot tar scrubber salable heavy distillates may be recovered. Hitherto, the recovery of such salable heavy distillates directly has not been accomplished.

Although the invention has been described in connection with a coke oven system and the drawings are confined to coke oven operation, it is to be understood that features of this invention are applicable to other operations in which gases containing entrained tar fog, dust or free carbon are produced, and it is intended and will be understood that

this invention is not limited to the specific details of the apparatus as illustrated in the drawings or to a coke oven system, but is of broad application.

5 In the drawings,

Fig. 1 is a plan view of a coke oven recovery plant showing the use of a tar scrubber in connection with a short collector main;

10 Fig. 2 is an elevation, partly in cross-section, through the short collector main, of the plant shown in Fig. 1; and

Fig. 3 is a cross-section of the collector main showing one form of agitating means.

15 The drawings show a battery of ovens 5, four of which are connected through the usual uptake pipes 6 with a short collector main 7 having center box 8 and drain pipe 9 to a tar receptacle 13. The tar from the receptacle 13 may be recirculated through the short collector main by pump 20 which returns the tar either through the pipe 21 into the end of the collector main or through the pipe 22 through the sprays 14 and 15. The ammonia liquor may be decanted through the outlet 23 before recirculation, if desired. Tar from the balance of the ovens not connected with the short collector main may be delivered to this short collection main through the pipe 24, or tar from an outside source may be delivered through the pipe 26. Each of these lines is equipped with a pre-heater 25, so that the tar may be preheated if desired.

25 The gases from the short collector main enter the bottom of the scrubbing tower 30 situated in the hottest part of the cross-over main and pass up through the tower counter-current to the tar or oil which is delivered through the sprays 31. The sprays may be fed through pipe 32 from the tar storage 13, or they may be fed from an outside source through pipe 34. A preheater 33 is provided for preheating the tar which is sprayed into the scrubber.

35 In the scrubber solid particles and entrained fog are removed and collected together with the non-volatilized fraction of the spray at the bottom of the scrubber and delivered through the pipe 35 to the storage 36. The purified gases pass from the scrubber through the pipe 37 to the condenser 38. Although an ordinary direct cooled condenser such as commonly used at coke ovens is here shown, any satisfactory type of condensing means may be used. The condensed distillate is removed through the pipe 39 and delivered to the decanter 40.

40 The greater part of the battery which is not connected with the short collector main and the tar scrubber is operated according to the usual practice. The hot gases pass from the coke ovens to the collector main 50, which is of the usual type and which is supplied with ammonia liquor or water or a mixture of ammonia liquor or water and tar through

sprays 51. The tar recovered in the collector main is drawn off through the pipe 52 to the decanter 53. The tar is drawn off into a collector 54 and from there may be supplied to the scrubber through the pipe 55 or the tar from the decanter may be used to flush the short collector main 7, in which case it is delivered through the pipe 24.

70 Fig. 3 is a cross-section of the collector main 7 showing a shaft 61 to which are attached paddles for agitating the tar in the collector main to thereby aid in the volatilization of the light constituents of the tar. According to the preferred method of operation, tar from the decanter 54 is conducted to the short collector main 7 in which it is brought into intimate contact with the hot gases passing through the collector main either by spraying the tar into the collector main or by any suitable agitating device or by a combination of such means. The light constituents of the tar are volatilized and the non-volatile constituents are drawn off through the line 9 as residue, e. g., pitch or heavy tar. This may be recirculated through the collector main if a heavier product is desired in which case some of the heavy product is continually drawn off from the receptacle 13 to suitable storage. The scrubber 30 is supplied from the decanter 54 through the line 55 or tar or oil of a different character is supplied to the scrubber depending upon the quality of the product which it is desired to recover from the scrubber. If a large quantity of tar is supplied to the scrubber and it is desired to further distill the light constituents from this material, it may be recirculated to the scrubber through the line 60 by means of the pump 61 or may be allowed to overflow through the pipe 62 to the receptacle 13, from which it may be pumped to the collector main by the pump 20.

105 Since the temperature of the collector main is above the temperature of the scrubber, the highest boiling products will normally be recovered from the collector main and the circulation of tar or oil will be through the scrubber and thence to the collector main, if the same material is to be treated in both the scrubber and the collector main, although in special cases it is possible to use the product recovered in the collector main in the scrubber by removing it from the tank 13 by the pump 63 and spraying it into the scrubber.

120 Instead of withdrawing the tar from the center box of the long collector main through the line 52 and conveying it thence to the scrubber and collector main, the tarry oil recovered in the decanter 64, which is drawn off the condensers 65, may be conveyed to the short collector main through the line 66 or to the scrubber through the line 67.

The volatilized constituents, together with the gases from the coke oven, pass up through

the scrubber 30 where entrained solid particles and tar fog are removed to a considerable degree. The vapors containing only a small fraction of entrained tar fog and solid

particles are withdrawn from the scrubber and pass through the line 37 to the condenser 38, and thence through the line 41 by means of the exhauster 42 to the usual apparatus for recovery of ammonia and light oils.

When it is desired to accurately control the boiling point of the constituents contained in the gases coming off of the scrubber through the line 37, this may be done by recirculating any suitable scrubbing medium from the container 36 through the scrubber by means of the pump 68. By controlling the temperature of the scrubbing medium by means of the heat interchanger 33, which may be either a preheater or a cooler as the circumstances demand, and by regulating the composition of the scrubbing medium, for example, by adjusting the valve 70, which controls the outflow from the tank 36, and admixing with the scrubbing medium the necessary quantity of low-boiling constituents which may be supplied by any suitable tar or oil recovered in the coke oven operation, or supplied from an outside source 69, the scrubber 30 will operate to condense higher boiling constituents than it is desired should be thrown over into the condensers. The lower boiling constituents supplied through the sprays will be volatilized so that by controlling the temperature, character and amount of the scrubbing medium, substantially all constituents with a boiling point above that desired in the products recovered in the condenser will be condensed in the scrubber and drawn off through the line 35, and the excess heat of the gases entering the scrubber will be absorbed as latent heat in the low boiling constituents contained in the scrubbing medium which are volatilized. By controlling the temperature and composition of the scrubbing medium in this manner, not only will the nature of the gases passing off through the line 37 be controlled, but it is also possible at the same time to control the properties of the product withdrawn from the bottom of the scrubbing device.

I claim:—

1. The method of distilling tar or pitch, which comprises bringing the material to be distilled into intimate contact with hot fresh coke oven gases while at a sufficiently high temperature to bring about effective distillation thereof and produce pitch, subsequently scrubbing the gases with the pitch while still at a high temperature to give clean gases containing oil vapors from which relatively clean oils can be subsequently condensed and to give a pitch residue from the scrubbing operation.

2. The method of distilling tar, which comprises collecting tar from the gases from the greater part of a coke oven battery in which coal is being coked by cooling the gases evolved therein, subjecting this tar to distillation by bringing it into direct contact with coal distillation gases from the ovens of another part of the plant, thereby distilling the tar, and scrubbing the gases and vapors resulting from the tar distillation and cooling the resulting gases and vapors to produce relatively clean oils and pitch.

3. The method of distilling tar, which comprises collecting coal distillation gases from a plurality of the ovens or retorts of a coal distillation plant in which coal is being distilled, bringing the tar into intimate contact with hot coal distillation gases from the plant in an amount and at a temperature sufficient to distill the tar and produce pitch, scrubbing the resulting gases and vapors to remove entrained tar and pitch particles, and cooling the scrubbed gases to give relatively clean oils.

4. The method of distilling tar or pitch and recovering distillate which comprises distilling coal tar or pitch by bringing it into intimate contact with hot coal distillation gases while at a substantially high temperature to bring about effective distillation, to produce pitch and to enrich the gases in oil vapors without substantial gasification, scrubbing the gases with coal tar or pitch while they are still at a high temperature to remove entrained impurities and then cooling the scrubbed gases so as to condense distillate oil therefrom, together with oils originally present in vapor form in the coal distillation gases.

5. The method of distilling tar in hot coal distillation gases from a coke oven battery in the ovens of which coal is destructively distilled which comprises conveying distillation gases from ovens of the plant to a common

Variations of the process and apparatus here disclosed are possible within the scope of this invention. The invention depends primarily upon the distillation of tar by means of hot coke oven gases, for example, in the collector main, and scrubbing of the resulting gases containing the volatilized oils to remove solid particles and entrained tar mist. The scrubbing is accomplished while the gases are still hot. In this way distillates, both light and heavy, which are relatively free from dust, free carbon and other insoluble material, are obtainable in a direct manner. Although the features of this invention have been described in connection

gas collector main while still at a high temperature, flushing the collector main with a quantity of coal tar sufficient to prevent the accumulation of pitch in the main, spraying the tar into the gases in the collector main so
5 as to distill the tar and produce pitch without substantial gasification of the tar, removing from the main gases and vapors resulting from the distillation of the tar and scrubbing them with tar or pitch while they
10 are still at a high temperature to separate suspended particles from them.

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