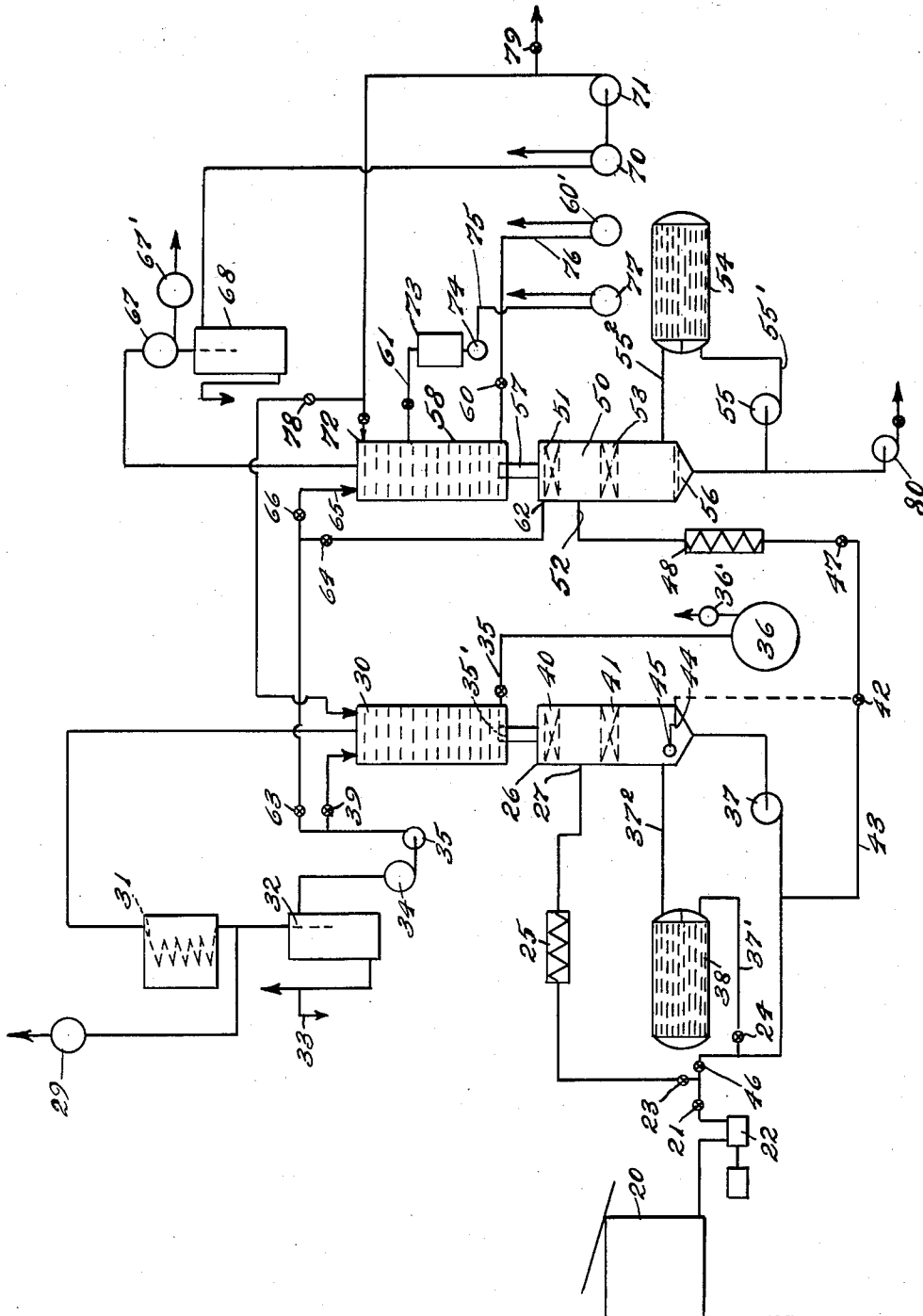


May 29, 1956

A. H. RADASCH
DISTILLATION OF COAL TAR

2,748,063

Filed Oct. 21, 1952



INVENTOR
Arthur H. Radasch.

BY *Richard S. ...*
ATTORNEYS.

1

2,748,063

DISTILLATION OF COAL TAR

Arthur H. Radasch, Upper Montclair, N. J., assignor to Alan Wood Steel Company, Conshohocken, Pa., a corporation of Pennsylvania

Application October 21, 1952, Serial No. 315,898

2 Claims. (Cl. 196-76)

This invention relates to an improvement in processes for distilling coal tar.

A purpose of the invention is to distill high temperature coal tar (tar produced in coke ovens, gas retorts and the like at a final coking temperature in excess of 750° C.) containing from 5 to 15 percent by volume and usually from 8 to 12 percent by volume of naphthalene, called "tar" herein, in two stages, to dehydrate the tar and remove light coal-tar solvent oil under vacuum in a first stage, to introduce the solvent oil from the first stage into a second stage, and to distill under vacuum in the second stage the residue from the first stage plus the added solvent oil.

A further purpose in the two-stage distillation of high temperature coal tar is to dehydrate and remove solvent oil in the first stage at a temperature between 80 and 140° C., thus avoiding or minimizing polymerization which may alter the compositions of the distillate products and distillation residue to be obtained in the second stage.

A further purpose in the distillation of high temperature coal tar is to avoid excessively low condenser temperatures incident to the presence of large amounts of water vapor in the second stage, which may increase the danger of plugging of columns and condensers with naphthalene, and may likewise be difficult to attain with available cooling water.

A further purpose in the distillation of high temperature coal tar is to minimize the danger of solidification of naphthalene in the fractionating column in the second stage at any temperature and particularly at low still-head temperatures by reintroducing solvent oil from the first stage into a second stage, thus altering the compositions in the fractionating column and in the condenser to maintain the oils fluid at both places.

A further purpose is to reduce the concentration of naphthalene in the overhead fraction of the second stage of distillation by reducing the tendency of water vapor in the second stage to carry over naphthalene oil through elimination of the major portion of the water in the first stage.

A further purpose is to distill coal tar that contains a substantial amount of naphthalene and a relatively low amount of solvent fraction by adding solvent to the tar, or to the vapors therefrom, during distillation and subsequently fractionating the vapors so that the naphthalene fraction produced on distillation may be removed from a fractionating column and the added solvent may be removed from a condenser at a temperature below the solidification point of the naphthalene fraction while carrying out the distillation under an absolute pressure between 10 and 80 mm. of mercury.

Further purposes appear in the specification and in the claims.

In the following description the terms, "solvent," "solvent fraction," and "solvent oil" are used interchangeably and mean the light fraction of coal tar distilling just below naphthalene. This fraction will usually distill 80 percent or more by volume to 200° C. and will contain not over 30 percent by volume, generally not over 10 or

2

15 percent, of naphthalene so that it will be entirely fluid at temperatures as low as 0° C. It will generally be made from coal tar, usually from the tar being distilled, and when so made will contain tar acids. However, a fraction of coke-oven light oil which is similar in character as regards distillation range and naphthalene content, but which lacks the tar acids, may be substituted for it and this substitution will frequently be made in practicing the invention, especially when starting up the process and when distilling tars that have a relatively low solvent content.

In the drawing I have chosen to illustrate only one of the numerous embodiments in which my invention may appear, selecting the form shown from the standpoints of convenience in illustration, satisfactory operation and clear demonstration of the principles involved.

The figure is a diagram useful in explaining the invention.

In my application, Serial No. 218,951, filed April 3, 1951, now Patent No. 2,673,833, for Distillation of Coal Tar and incorporated herein by reference, I have described a process for distilling coal tar under vacuum that may be operated in one stage, or in two or more stages. In my continued investigation of this process in a pilot plant I have found that, in most applications to a commercial plant, the one-stage process has several advantages over a two-stage process. Besides the fact that only one unit of each principal item of equipment is required, an additional advantage of the one-stage process is that the oils that are removed from the tar are held at temperatures above 100° C. for relatively short periods of time, thus minimizing the extent of heat polymerization of certain constituents in these oils.

However, it is sometimes difficult to condense the vapors when distilling some coal tars by the one-stage process owing to the fact that condensation temperatures are much lower under vacuum than for atmospheric distillation and unless special precautions are taken to assure absence of naphthalene in appreciable quantities from the vapors passing to the condenser, solid phase (naphthalene) may separate in the condenser and cause the plant to shut down. The problem is aggravated when excessive quantities of moisture are present in the tar.

The nature of the difficulty may be further explained by reference to the following table which gives the analyses of four tars. The method used consists of taking a 500 ml. sample and, after drying the sample, distilling it in a flask provided with a 10-ball Snyder fractionating column. The distillation is conducted in such a manner that a minimum of flooding occurs around the glass balls and all balls are acting vigorously and the rate of removal of distillate from the top of the column is uniform at the rate of approximately one drop per second. The volume of liquid distillate is noted when vapor temperatures measured by a thermometer placed at the head of the column read 200° C., 235° C. and 250° C., and the quantities of distillate so collected are calculated to percent by volume of the original tar sample.

TABLE I

Analysis of high temperature coal tars

Sample.....	A	B	C	D
Oil Fraction, percent by volume:				
Solvent, 0-200° C.....	2.76	2.05	0.94	0.20
Naphthalene—				
200-235° C.....	15.38	15.97	13.04	16.26
235-250° C.....	2.85	2.67	1.46	1.02
Ratio, (percent Naphthalene fraction)/(percent Solvent fraction).....	5.6	7.8	13.9	81
Setting Point of Entire Fraction to 250° C., °C.....	52.8	53.6	62.2	69.8

The yields of fractionated oils obtained by distillation of these tars in plant equipment are not identical with the above yields obtained in the laboratory but they approximate those yields fairly closely. Some of these tars, notably Sample D, contain a relatively small amount of solvent fraction. This is strikingly shown by reference to the next to the last horizontal line of the table which gives the ratio of the percentage by volume of naphthalene fraction (200–235° C.) to the percentage by volume of solvent fraction (0–200° C.). This ratio is a rough measure of the problem of separating these two oils by fractional distillation. Thus, when distilling tar D, more efficient fractionating equipment is required for the separation than for tar C, which in turn requires more efficient equipment than for tar A.

In operation, the solvent fraction is usually produced in the condenser, some of it being refluxed to the fractionating column. By interchange with components in the vapor, this reflux causes production of a naphthalene fraction in the column as is well known. Under conditions of poor fractionation, or upset in operation of the column, too much naphthalene may appear in the overhead stream and cause the condenser to plug.

The fractionating column must operate above the dew point for water since it will fail to function properly if liquid phase water is present. Hence, when distilling tar containing water, vapors leaving the column and passing into the condenser contain the water in vapor form. One effect of excessive water in the tar is seen from Table II. The amount of oil vapor that may be carried over into the condenser by the non-condensed water vapor and by the inert gases that may be present has been worked out for the following conditions: Pressure of operation, 35 mm. Hg; air leak into system, 25 pounds per hour; temperature entering condenser, 45° C.; oil vapor assumed to act as if it were a single component having a boiling point of 180° C. at atmospheric pressure; rate of distillation, 1000 gallons per hour.

TABLE II

Solvent carried at 45° C. and 35 mm. Hg by inert gas

Water in tar, percent by volume.....	0	2	4	6
Solvent carried, percent by volume of tar.....	0.3	2.4	4.5	6.6

The table shows that the carrying capacity of the gases for oil vapors depends in large measure on the water content of the tar.

None of the coal tars shown in Table I contains as much solvent fraction as is shown by the last two columns in Table II so that when tars that contain from 4 to 6% of water are distilled, the tendency will be for the gases to saturate themselves with the next heavier oil, which is the naphthalene fraction, and soon a point is reached where the naphthalene concentration in the condenser, which is generally operated below 45° C., is so high that the condensate will solidify and cause the plant to shut down. It is also seen by reference to the two tables that tar C and especially tar D should be nearly dry when distilled under vacuum.

I have found that these operating difficulties can be overcome by removing the water and solvent in a first or dehydration stage operated at an intermediate pressure and at low temperature and subsequently adding the solvent produced in the first stage to the second stage of distillation, which is referred to later as the distillation stage. I have also found that the same basic idea can be applied to the two-stage distillation process described in application Serial No. 218,951 in order to simplify the operation and the equipment required. That is, when the first stage is operated to dehydrate and remove both solvent and naphthalene oils, the solvent produced in the first stage may be added to the second stage of distillation and the vapors of the second stage fractionated in a column so that naphthalene oil of the second stage

may be produced in and withdrawn from the column at temperatures well above the setting point of the oil and the added solvent may be recovered from the condenser of the second stage. The improvement in operating technique embodied in the present invention is explained by reference to the figure.

Coal tar from a source 20, is fed through valve 21 by pump 22, through valve 23, through heater 25, and into flash chamber 26 at point 27. Valve 21 may be manually operated or may be an automatically controlled valve operated by the pressure differential across the valve, or by a temperature, such as the temperature at the outlet to the heater 25, as well known. The heater 25 may be of any suitable design, but will preferably be heated by steam at low pressure. The flash chamber 26 is maintained at 50 to 150 mm. of mercury absolute pressure and will usually be around 80 mm. pressure. In a preferred method of operation, the coal tar, at normal storage temperature of 50 to 60° C., will be heated in heater 25, to from 80° C. to 140° C., usually to around 100° C., that is, to a temperature sufficient to flash off most of the water at the pressure prevailing in the flash chamber. As is known, the removal of water from tar by distillation is accompanied by vaporization of some oil owing to the steam-distillation effect. The amount of oil so vaporized is roughly equal to the amount of water in the tar although, of course, it may be either more or less depending upon conditions of operation and on the nature of the tar. The oil evolved in a flash distillation of tar such as that described is a wide boiling oil and may contain much naphthalene. To prevent solidification of this oil, or any portion of it, when it reaches the condenser it is desirable that it be fractionated before it is condensed. A fractionating column 30 is provided above and connected to the flash chamber for this purpose. A condenser 31 is provided connected to the top of the fractionating column to condense water and oil, which then flow to the separator 32. A vacuum pump is provided at 29. Water overflows at 33 to be disposed of to the sewer or to be further treated as waste disposal rules require. Oil flows to the collector 34. A portion of it is returned by pump 35 through valve 39 as reflux to the column 30. The arrangement of the separator shown in the figure is for oil that is lighter than water but if the oil should be heavier than water the connections to the separator 32 will be reversed. When 2 or 3% by volume of oil is volatilized in the flash chamber together with most of the water, it will usually suffice if approximately half of the oil is returned to the column as reflux but, of course, no more solvent can be withdrawn as product than corresponds to the solvent content of the tar. Of course any other condensing arrangement that is equivalent to the above described method may be used. For example the single condenser 31 may be replaced by a partial condenser and a final condenser, and the oil fraction from the partial condenser may flow by gravity to the column as reflux, thus eliminating the necessity for the pump 35.

I have found that it is not necessary to get the tar bone dry in this first, or dehydration stage. It can be seen from Table II that when distilling in the distillation stage a tar containing a few tenths of a percent of water, the amount of oil vapors carried over into the condenser at 45° C. and at 35 mm. of mercury absolute pressure is not more than the solvent content of most coal tars as shown by reference to Table I, and if the vapors from the distillation are efficiently fractionated they can be handled without danger of solidification in the condenser. It is desirable, however, that the moisture content of the residue from the dehydration stage be not over 0.5%. I have found that high temperature coal tar with 0.1 to 0.5% by volume of moisture can be produced when the flash chamber of the dehydration stage is maintained at around 80 mm. of mercury absolute pressure and at 100° C. but the temperature and pressure ranges pre-

5

viously given permit sufficient latitude to allow for differences in tars, differences in operating conditions between summer and winter, differences in equipment, and the like.

Should it be desired to distill more oil in the dehydration stage, say up to 10% by volume or more, this can be done by heating the tar in the range from 140–200° C. while the flash chamber is maintained at 50 to 150 mm. of mercury absolute pressure. When more than 2 or 3% by volume of oil is distilled, it will be desirable to draw off a naphthalene oil at 35 below annular baffle 35' to the collector tank 36. A vacuum pump is provided at 36' to maintain the collector under vacuum if desired. In this case, the heat required for the distillation may be supplied to the residue in the flash chamber, either by jacketing the flash chamber and heating the jacket, or by withdrawing the dehydrated tar, as by pump 37, and circulating the tar through valve 24, pipe 37', heater 38, and returning the tar to the flash chamber by pipe 37². A preferred design of heater 38 is one which contains a multiplicity of parallel heating paths heating by condensing vapors such as steam. The circulation rate through the heater should be such that the Reynolds number characterizing the flow through the tubes is greater than 2100 and preferably greater than 7000 as previously described in application Serial No. 218,951.

The additional vaporization in the flash chamber causes more vapors to pass to the condenser. As before, no more solvent may be withdrawn as product than corresponds to the solvent content of the tar; the rest of the condensed oil vapors are returned to the fractionating column as reflux in order to produce the naphthalene fraction that is drawn off at 35.

The condenser 31 will receive the vapors at a temperature above the dew point for water. They will be cooled to as low a temperature as can be obtained by the cooling water available in order to condense them as completely as possible. In summer this temperature may not be below 40° C.; in winter and in plants where there is an adequate supply of cold water it will be lower, and may for example be 10° C., or even lower. In any case, the oil in the condenser should be such that it will not deposit crystals at the temperature of the condenser which will be 40° C., or below, but which cannot be below 0° C. in a water-cooled condenser. This means that the oil condensate will contain not over about 25 to 30% by volume of naphthalene.

Flash chamber 26 may contain packing 40 above the point 27 of tar feed for reducing entrainment in the vapors, and may contain packing 41 below the point of tar feed for the purpose of releasing water from the tar without foaming and for heating the incoming tar by hot vapors arising from below.

Dehydrated tar may be removed from the flash chamber by flowing it by gravity to the second stage, or it may be removed by a pump. For example, pump 37 may be used for this purpose. A valve 42 in pipe 43 operated by lever arm 44, controlled by the float 45, or any other similar or equivalent device may be used for controlling the flow automatically. Valve 24 on the recirculating line to the heater 38 may be closed when pumping to the second stage if desired. Valve 46 in a line connecting the discharge from the pump 37 to the line leading to the inlet to the heater 25 is provided so that the dehydrated tar may be heated by passing it through heater 25, thus providing greater flexibility in operation especially when heater 38 is not used.

The feed to the distilling stage flows through pipe 43 and valve 47 and may be further heated by heater 48 of suitable design before it enters the flash chamber 50. This flash chamber may be provided with baffles or packing 51 above the point 52 of feed entrance and with baffles or packing 53 below the point of feed.

Generally, the heat for this stage will be supplied by

6

the indirect heater 54. A preferred design of this heater contains a multiplicity of parallel heating paths through which tar flows at a Reynolds number greater than 2100, being forced through the heater by the pump 55 which receives tar from the pool 56 in the bottom of the flash chamber 50, flowing through pipes 55' and 55² and returning to the flash chamber. The temperature of the tar and vapors in the flash chamber will generally be between 160° and 200° C. and when the heating is done by steam, although it may go to 275° when, for example, it is heated by Dowtherm (diphenyl and diphenyl oxide). The pressure in the flash chamber will be between 10 and 80 mm. of mercury absolute, generally below 50 mm. The accumulation of distillation residue in the pool 56 is removed from the system by the pump 80.

Vapors released in the flash chamber pass through the connection 57 into the fractionating column 58 above the flash chamber. They will contain a high concentration of naphthalene and some creosote, that is, oils whose boiling points are above that of naphthalene.

The naphthalene oil and creosote may be drawn off from the bottom of the column combined as one fraction through valve 60 to receiver 60', or the creosote may be withdrawn through valve 60 and the naphthalene at a point higher in the column as at 61.

Since the tar feed to the distillation stage has been more or less completely stripped in the dehydration stage of its content of solvent, the vapors passing to the top of the column will contain a low concentration of solvent and a relatively high concentration of naphthalene. This means that unless other steps are taken the temperature prevailing at the top of the column and the temperature at which the condenser is operated must not be allowed to fall below predetermined values as explained in application Serial No. 218,951, since otherwise solid naphthalene may deposit at these points. On the other hand, if kept at a temperature sufficiently high to prevent deposition of solid naphthalene in the condenser, the inert gases leaving the condenser will carry along with them considerable quantities of uncondensed vapor of naphthalene and other oils. One way of recovering the oil vapors from these vent gases is by absorbing them in a heavy oil, as for example, a heavy petroleum or coal-tar oil having a distillation range above that of naphthalene. Another way is by condensing the vapors after they have been partially compressed to atmospheric pressure in a multi-stage steam jet ejector as described in my application Serial No. 317,930, filed October 31, 1952, for Method of Handling Gas.

Still another way is by application of this invention which entirely eliminates these troublesome naphthalene vapors and therefore obviates the necessity for any special treatment of the residual vapors from the condenser. If the solvent that has been removed from the tar in the dehydration stage is restored to the tar in the distillation stage, or to the vapors from the tar in this stage, the solvent will vaporize, being the most volatile component, and will pass preferentially to the top of the column and to the condenser of this stage. When reflux to the column is supplied from the condensate oil, which is a solvent containing little naphthalene, the condition of high-solvent and low-naphthalene concentration in the condensate can be maintained as well known from the principles of column operation. The vapors entering the condenser may then be cooled to as low a temperature as is practicable with substantially complete condensation and without danger from deposits of solid naphthalene. The quantity of solvent is preferably regulated to maintain the temperature of the vapor entering the condenser between 25° and 80° C. The naphthalene in the vapors of distillation is concentrated at a point in the column and is conveniently removed from this point. Thus my present invention consists of adding solvent to a stage of distillation of coal-tar, especially to coal tars of low solvent and relatively high naphthalene

contents, and fractionating the vapors of distillation to recover a naphthalene fraction and a fraction containing the added solvent. In its most usual application it consists of removing solvent and water from the tar to be distilled in a first or dehydration stage, separating solvent from the water and returning this solvent to the tar, or to the vapors from the tar, in a second, or distillation stage.

The solvent may be returned at any of several convenient points. Thus, it may be returned to the flash chamber 50 as at 62 through valves 63 and 64, and into the flash chamber where it will vaporize rapidly owing to its relatively high volatility. This method of introducing the solvent has the advantage of aiding the vaporizing of heavy components of the tar. The solvent may be returned to the top of the column 58, as at 65 through valves 63 and 66, as part of the reflux to the column, or the solvent and water condensate from condenser 31 may be combined with solvent and water from second stage condenser 67, flow to the single separator 68 and to the receiver 70, the solvent without the water to be picked up by pump 71 and returned as reflux at 72 at the top of the column. Some solvent may be returned through valve 73 to the top of fractionating column 30. The solvent may be added to the dehydrated tar of the first stage before it passes to the flash chamber 50. Various other ways that are but minor variations in application will occur to the user of this invention. A vacuum pump is provided at 67'.

The solvent, which in whole or in part, is restored to the tar in the distillation stage becomes part of the working contents of the fractionating column 58 and condenser 67, that is, it is the source of the solvent oils found in the vapors and oils appearing in the upper part of the column and in the condenser. The amount of solvent that is returned as reflux to column 58, including that produced in condenser 31, is regulated by the valves in the reflux pipes so as to accomplish the desired fractionation between naphthalene and solvent, that is, so that the naphthalene fraction is produced in the column to be drawn off at 60 or 61 as described above and so that the oil vapors passing to the condenser 67 contain not more than 25 percent of naphthalene by volume and will be fluid when they are condensed. That part of the solvent not used or required for reflux may be drawn off through valve 79 as product. A part of the production of solvent, considering the overall process of distillation, may be taken from the collector 34.

In application of this invention, it will be found desirable to make certain that the minimum quantity of solvent is dissolved in the naphthalene fraction that is removed through valve 60 or at 61. This may be assured by stripping the solvent from the naphthalene oil fraction either by direct action of steam or by reboiling part of the naphthalene fraction after it has been withdrawn from the main column 58, either of which methods is well known practice in the industry. For this purpose, a stripping column 73, with a reboiler 74 are provided. Naphthalene oil runs off at 61 and into the column 73 which may be either a packed or plate-type column. As it runs down the column, it meets rising vapors from the reboiler 74. The vapors containing the more volatile or solvent components of the naphthalene stream flow back into the main column 58. The naphthalene oil collecting in the reboiler 74 will be stripped of its solvent content and may be allowed to overflow through line 75 into the receiver 77.

Similarly a stripping column could be used on the creosote fraction after it is drawn off through the valve 60, for the purpose of removing low boiling components like naphthalene from it.

The application of this invention is quite different from using an absorber following a condenser for the purpose of absorbing vapors, mostly naphthalene, before the gases pass to the steam jets or vacuum pumps. When using

an absorber, the solvent for the naphthalene and other oil vapors is generally an oil that is less volatile than naphthalene and more often than not, the absorbent will be a petroleum fraction, which is of non-coal-tar origin. In that case the absorbent must be kept separate from other liquids in the system and any oils dissolved in the absorbent must be recovered from it in a separate and subsequent operation. In the present invention the light fraction produced in the dehydration stage is mixed back into the tar in the distillation stage, or into the tar vapors, to become an indistinguishable part of the latter and the light fraction is again fractionated from the vapors in a part of the fractionating column that is already in use for this purpose. This makes it possible to remove naphthalene where it exists in high concentration in the oil which is at a point that is some plates removed from the top of the column, and where its temperature is between 80° and 150° C. which is well above its setting point which is usually between 50° and 70° C. The concentration of naphthalene is progressively reduced in the oils on the plates above the point of naphthalene removal so that by the time the vapors reach the condenser the concentration of naphthalene does not exceed about 25 or 30 percent by volume in the low boiling oils that are condensed in the condenser. Generally it will not be over 15 percent by volume in these oils. The oil in the condenser will not deposit crystals at 0° to 40° C.

As examples in the application of this process the following may be cited:

Example 1.—The tar used contained 2.8 percent by volume of water. The analysis of the dry tar showed it to contain 1.64 percent solvent (0–200°) and 15.17 percent naphthalene oil (200–235° C. by volume. This was heated and flashed in a chamber maintained at 99° C. under 96 mm. of mercury absolute pressure. Dehydrated tar produced contained 0.1 percent by volume of water. Oil produced was 1.11 percent by volume of the dry tar feed. It had a specific gravity of 0.959 at 15.5° C., contained 6.8 percent by volume of tar acids and 90 percent by volume of it distilled below 203° C.

Example 2.—The tar used contained 3.9 percent by volume of water. The analysis of the dry tar showed it to contain 1.82 percent of solvent and 15.69 percent naphthalene oil by volume. This was heated in a preheater and flashed into a chamber maintained at 112° C. and 80 mm. of mercury absolute pressure. Heat was also applied to the residue in a recirculation type heater. The dehydrated tar made contained 0.05 percent by volume of water. The oil had a specific gravity of 0.954 at 15.5° C., contained 4.1 percent tar acids and 10.9 percent naphthalene by volume, and 90 percent by volume of it distilled below 208° C.

The oil was mixed with the dehydrated tar, and fed through a preheater and into a flash chamber maintained at 167° C. and 33 mm. of mercury absolute pressure, the residue being heated by circulation through a tubular heater. Oil distilled in this stage was 1.98 percent solvent and 16.78 percent naphthalene oil by volume. The solvent oil had a specific gravity of 0.996, contained 20.6 percent of tar acids, 5.2 percent of naphthalene by volume and distilled 90 percent by volume at 193° C. The naphthalene oil was produced in two portions, one portion having a setting point of 57.0° C. and the other a setting point of 51.6° C.

In the distillation stage the vapors passing to the condenser were at 38° C. and 18 mm. of mercury absolute pressure and the temperature leaving the condenser was 13° C. These conditions could not have been maintained if the solvent had not been added to the system in the second stage. Without the presence of solvent a temperature of 60 to 65° C. would have been needed in the condenser to keep the oil liquid at all times and at that temperature the amount of naphthalene vapors carried beyond the condenser would have been consider-

able and would have required special steps for its elimination and recovery.

If the original wet tar had been distilled under the same total pressure conditions, the temperature entering the condenser to maintain the same concentration of oil vapors would need to be about 15° C. instead of 38° C. This would have called for correspondingly low temperatures near the top of the column with consequent danger from solidification of naphthalene within the column.

In view of my invention and disclosure variations and modifications to meet individual whim or particular need will doubtless become evident to others skilled in the art, to obtain all or part of the benefits of my invention without copying the process shown, and I, therefore, claim all such insofar as they fall within the reasonable spirit and scope of my claims.

Having thus described my invention what I claim as new and desire to secure by Letters Patent is:

1. The process of distilling coal tar containing a substantial amount of naphthalene and separating it into fractions, which comprises continuously feeding the coal tar containing moisture into a first stage maintained at an absolute pressure of 50 to 150 mm. of mercury and at a temperature of between 80 and 140° C. and there flashing water and oil into vapor and producing a dehydrated tar containing less than one-half of one percent of water by volume, condensing water and oil vapors to produce a coal tar solvent fraction that does not deposit crystals at temperatures between 0° and 40° C., continuously removing dehydrated tar from the first stage and feeding it into a second stage maintained at an absolute pressure of 10 to 80 mm. of mercury and at a temperature of 160 to 275° C., continuously feeding coal tar solvent fraction produced in the first stage and in liquid form into the second stage at a point and in a manner such that vapors from the coal tar solvent will mix with vapors produced from the dehydrated tar in the second stage, fractionating the vapors in the second stage in a fractionating column to produce a naphthalene fraction having a setting point in excess of 50° C. and an overhead fraction that does not deposit crystals at tem-

peratures between 0° and 40° C., producing a pool of residue of distillation and removing accumulation of residue from the second stage.

2. The process of distilling coal tar containing a substantial amount of naphthalene and separating it into fractions, which comprises continuously feeding the coal tar containing moisture into a first stage maintained at an absolute pressure of 50 to 150 mm. of mercury and at a temperature of between 140° C. and 200° C. and there flashing water and oil into vapor and producing a dehydrated tar containing less than one-half of one percent of water by volume, fractionating the vapors to produce a naphthalene fraction containing a part of the naphthalene present in the original tar, condensing water and oil vapors to produce a coal tar solvent fraction that does not deposit crystals at temperatures between 0° and 40° C., continuously removing dehydrated tar from the first stage and feeding it into a second stage maintained at an absolute pressure of 10 to 80 mm. of mercury and at a temperature of 160 to 275° C., continuously feeding coal tar solvent fraction produced in the first stage and in liquid form into the second stage at a point and in a manner such that vapors from the coal tar solvent will mix with vapors produced from the dehydrated tar in the second stage, fractionating the vapors in the second stage in a fractionating column to produce a second naphthalene fraction at a temperature above 80° C. and an overhead fraction that does not deposit crystals at temperatures between 0° and 40° C., producing a pool of residue of distillation and removing accumulation of residue from the second stage.

References Cited in the file of this patent

UNITED STATES PATENTS

1,814,989	Wessell	July 14, 1931
1,957,484	Zimmerli et al.	May 8, 1934
2,029,883	MacCubbin et al.	Feb. 4, 1936
2,260,072	Wilton	Oct. 21, 1941
2,366,900	Weir	Jan. 9, 1945
2,673,833	Radasch	Mar. 30, 1954