DISTILLATION OF COAL TAR

BY

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Presented by

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Approved

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DISTILLATION OF COAL TAR

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C. C. WILSNACK AND S. E. LUNAK.
DISTILLATION OF COAL TAR.

Coal tar is a by-product obtained in the manufacture of coal gas and coke. In the process of gas-making the constituents of the tar escape from the gas retorts in the form of vapour, with a little solid free carbon in an extremely finely divided state; they are precipitated in the hydraulic main, in the condensers, scrubbers, etc., in a liquid state, at the same time as the ammoniacal liquor is formed. The tar formed in the hydraulic main is, of course, poorer in the more volatile products than that formed in the condensers and scrubbers, and is consequently much thicker than the latter; but it is usual to mix all these classes of tar in the cistern where they collect, along with the ammoniacal liquor. The process of obtaining tar and ammonia as by-products in coke-making is similar to that in the making of coal gas.

The theory of the formation of tar in the distillation of coal is very uncertain. From the researches of Berthelot we know that most or-
ganic bodies, also those of the fatty series, furnish aromatic compounds when exposed to a very great heat, especially when brought into contact with a red hot tube. But, on the other hand, those same aromatic products are decomposed by higher degrees of heat, and ultimately furnish free hydrogen, along with free carbon, or else compounds so rich in carbon that they have been hitherto indistinguishable from it. But the different stages of these processes are extremely complicated and the attempts at explaining the formation even of the better known constituents of coal-tar by means of some plain reactions of a few simple compounds, such as acetylene do seem to be very convincing.

Coal tar is an extremely complex mixture of chemical compounds, some of which have not even been isolated as yet. Thus very little is known of the indifferent oils occurring in its distillation, between the phenols and naphthalene on the one side, and anthracene oil on the other; neither do we know all the com-
pounds existing in crude anthracene, and still less those constituting the pitch. Whether some of the constituents of coal-tar were already present in the coal and are therefore simply evolved by the ordinary process of distillation, must be left an open question. It can hardly be doubted that coal contains aromatic compounds; some contend that it is entirely made up of such, and contains no free carbon at all. But it is another question whether any of these compounds are volatile, without change, at the temperature existing in the gas retorts.

The first public gas-plant was erected in London in the year 1813, in Paris 1815, in Berlin 1826. The tar formed in the manufacture of coal-gas necessarily forced itself upon the notice of the gas-manufacturer, since it could not be thrown away without causing a nuisance. It was probably from the first burnt under the retorts; but the method of doing this without giving very much trouble was not understood then. It was also used as
a paint for wood or metals. It was quickly perceived that in this respect tar is improved by boiling it down to some extent; and as early as 1815 it was found that, if this boiling down is carried out in closed vessels or stills, a volatile oil is obtained which may be employed as a cheap substitute for spirits of turpentine. But this does not seem to have been carried out to any great extent, and coal-tar remained for more than a generation from the first introduction of gas-lighting, a nuisance. The tar was used for various purposes in the crude state and it was not until 1845 that the presence of benzene was discovered in tar. At that time benzene was the only product of the distillation, the residue in the still being used for roofing. The discovery of aniline colors in 1856 gave tar distilling a sudden impetus. The discovery of alizarin, a dye obtained from anthracene, soon followed and this made it necessary to make a complete distillation of the tar. Plants are now in operation in Europe and the
United States where the complete distillation of tar is made on a commercial scale. The primary distillation in most plants is the same; the secondary distillations vary according to existing conditions. The following is a description of the method used by the authors and is a process that can be used on a commercial scale.

**PRIMARY DISTILLATION.**

The first step in the analysis of coal tar is to subject it to fractional distillation according to the following scheme.

1. First runnings up to 110°C.
2. Light oil up to 210°C
3. Carbolic Oil (phenol & napthalene) to 240°C
4. Creosote Oil up to 270°C
5. Anthracene Oil above that.

1000 cc of the tar were placed in a small copper still and heated by gas, the gas being measured with a meter. The quantity of the gas used was noted for each fractionation.
The specific gravity of the tar used was .971.

Gas used for the five fractions are as follows:

<table>
<thead>
<tr>
<th>Fraction</th>
<th>Liters</th>
<th>Calories</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10-1/2</td>
<td>86.1</td>
</tr>
<tr>
<td>2</td>
<td>40</td>
<td>328</td>
</tr>
<tr>
<td>3</td>
<td>12</td>
<td>98.4</td>
</tr>
<tr>
<td>4</td>
<td>6</td>
<td>49.2</td>
</tr>
<tr>
<td>5</td>
<td>16</td>
<td>131.2</td>
</tr>
</tbody>
</table>

The method of procedure for obtaining primary and secondary fractions is discussed in the following pages in the order of their boiling points.

First Runnings:

The product distilling up to 110° is known as the first runnings. This consists of a large amount of water, some ammoniacal liquor and some of the light oils which have been carried over. In commerce the distillate is allowed to stand for sometime until all the oil has come to the top. This is drawn off and added to the light oil fraction. The ammoniacal liquor is
sent to the plant for the recovery of ammonia.

The amount obtained was 10.5 grams which represents a little over 1% by weight of the original amount. The product was seen to consist of nothing but water so it was thrown away.

**Light Oil:**

The product obtained between the temperature of 110° to 210° is known as light oil and the weight was 369.5 grms or 38%. This oil contains benzene, toluene, xylene, some of the higher homologues, together with phenols and naphthalene. The light oil is put into a separatory funnel with 100 cc of 30% NaOH and thoroughly agitated for several minutes. The sodium hydroxide combines with the phenols which are thus removed when the soda solution is drawn off. 160 cc of solution were drawn and added to the alkaline washings from the carbolic oil fraction.

After washing the oil with water, 10 cc of sulphuric acid Sp.C 1.84 were added, the temperature was kept low so that the acid could not act on the benzine, toluene and xylene. The purpose
of the acid was to dissolve thiopen, olefins and in general to remove any bodies upon which it acts. Also naphthalene and phenols are converted into sulphuric acids, remaining dissolved in the acid. The action of sulphuric acid upon the valuable bodies, that is benzine and its homologues, is very insignificant in the cold if the quantity of acid is moderate, therefore the acid should not be used in excess of that needed for purification.

The acid tar formed when the sulphuric acid is added was drawn off and the oil washed several times with water and finally with dilute NaOH to remove all of the acid. The 10 cc of acid combined with 20 cc of the oil so that 85 cc was lost altogether in the washings.

The acid tar causes a great deal of trouble to tar distillers killing as it does all fish in rivers and perceived at great distances by its stench. Many attempts have been made to utilize the acid tar even without profit, but as yet no satisfactory treatment seems to have been discovered. It is usually neutralized with lime
and allowed to sink into the ground.

In commerce the washed oil is now subjected to fractional distillation by steam according to the following plan. Up to $110^\circ$ the distillate is called 90% benzol since that amount by volume distills below $110^\circ$. It contains about 70% pure benzine 24% toluene and some xylene. If collected up to $140^\circ$ C, the distillate is known as 50% benzol and contains about 46% of pure benzene. Between $140^\circ$ C and $170^\circ$ a distillate called solvent naphtha is obtained. This consists mainly of xylenses, cumenes, etc., and is used as a solvent for resins and rubber. Only rarely is a complete distillation into benzene, toluene and xylene aimed at.

Since the object of this investigation is to obtain the percent of each constituent present in coal tar a complete distillation was made with the following results. The distillation was made in a fractionating column and the amount of gas used in each fractionation noted.
<table>
<thead>
<tr>
<th>Name of Substance</th>
<th>Boiling point</th>
<th>Specific Gravity</th>
<th>Amount Obtained</th>
<th>% Amt. Gas used</th>
<th>Calories</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene C₆H₆</td>
<td>82</td>
<td>.879</td>
<td>48</td>
<td>6.5</td>
<td>79</td>
</tr>
<tr>
<td>Toluene C₇H₈</td>
<td>112</td>
<td>.872</td>
<td>50</td>
<td>6.5</td>
<td>60</td>
</tr>
<tr>
<td>Xylene C₈H₁₀</td>
<td>140-145</td>
<td>.888</td>
<td>97</td>
<td>12.7</td>
<td>150</td>
</tr>
</tbody>
</table>

Some naphthalene was obtained in this distillation which was added to the naphthalene in fraction 3. The distillation was carried on until the temperature began to rise above 150° when the flame was cut off and the contents added to the creosote fraction.

A better name for xylene is xylo1 which consists of a mixture of the three isomeric xylenes, metaxylene, paraxylene and orthoxyylene, the first named being the only one of technical importance. Their separation is based on the behavior of these substances towards nitric acid, concentrated sulphuric acid and fuming sulphuric acid. Metaxylene is reacted on by nitric acid so the mixture of three substances was heated with dilute nitric acid. The acid combines with paraxylene and orthoxyylene and the metaxylene can easily be separated from them. It was then dis-
tilled with steam when a mixture of water paraffine and metaxylene was obtained. This was treated with concentrated sulphuric acid when the metaxylene dissolved, leaving the paraffine unaltered. The acid was next neutralized with sodium hydroxide and the metaxylene distilled when it was obtained in a pure state.

Benzine, toluene and xylene are used mainly in the manufacture of colouring matters. Nitrobenzene is made from the purest benzine and is used as a substitute for oil of bitter almonds in cheap perfumery. Crude benzol is very frequently used as a solvent both in the manufacture of colours and otherwise. Its greatest importance is its ability to dissolve fatty matter, upon which is based its extensive use as a detergent. Benzol also dissolves many resins, gutta-percha, alkaloids, etc. In the manufacture of rubber goods, the three xylenes are used extensively as solvents.

Third Fraction.

Carbolic Oil and Naphthalene.

The third fraction was collected be-
between the temperatures of 210° and 240° C. A condenser was used at first but removed when 218° was reached. It was necessary from now on to keep the pipe through which the distillate flowed warm as the product coming over had a tendency to solidify and choke up the pipe. This fraction when completely distilled amounted to 256.6 grams and had a specific gravity of 1.008. At approximately 65° C, the mass solidified into a canary yellow compound. The odour of coal tar was so predominant that it was impossible to detect the presence of another compound by smell. The distillate contained essentially naphthalene and phenol with a small quantity of creosote oil.

Naphthalene is very volatile, the temperature of boiling water being sufficient to volatize it. For this reason we found it present in all of the distillates. Knowing this fact, the quantitative estimation was left as the very last operation so that the naphthalene in the other distillates could be added and the total determined.
The original fraction of 256.6 grams was placed in a large separatory funnel and the whole placed under hot water at about 85° C. At this temperature the compound remained in the liquid state. To determine the amount of NaOH is to be added a certain amount of oil is put in a graduate and the soda solution gradually added. The whole is carefully shaken after each addition. The two layers separate out. The volume is noted and if on the next addition of alkali, it is not lessened, the addition has been sufficient.

Fifty cc. of sodium hydroxide sp.gr. 1.15 was then heated to 92° C. and added to the solution in the funnel and the whole thoroughly shaken for a few minutes. Upon permitting the solution to remain undisturbed for about ten minutes, keeping it under the hot water tap, two layers separate out, one of sodium phenolate, uncombined sodium hydroxide and a very small quantity of light oils. The heavy layer was drawn off and the rest poured into a shallow dish and allowed to solidify.
An attempt was made to remove the residual phenols as a compound of sulphonic acid but this failed as the sulphuric acid sp. gr. 1.7 combines with naphthalene forming alpha and beta naphthalene sulphuric acid above 70°, this being below the temperature at which the washing was carried on, the substance necessarily being in the liquid state to insure intimate reaction. An apparatus devised to keep the temperature of the naphthalene between 65° and 70° C. will make this separation possible. To the solidified naphthalene in the dish all the naphthalene from the other fractions, light oil, creosote and anthracene, was added and the whole placed into a centrifugal separator. The purpose in doing this was to enable the sulphuric acid and naphthalene to come intimately in contact and at the same time keep the temperature low so that the two would not react chemically. To keep particles of naphthalene from leaving through the pores in the dish a thin strip of
lion was placed over the openings. Twenty cc. of acid sp. gr. 1.7 was used in this operation. The substance was then thoroughly washed with distilled water temperature 40–50° until very nearly all traces of acid were removed. The three washings alkaline, acid and water were set aside for the determination of phenol. To complete the separation of solid from liquid as much as possible the impure naphthalene was placed in a press and subjected to a pressure. The separated liquid was likewise added to the three quantities for the determination of phenol. To complete the purification of naphthalene it was melted and put into a distilling flask and subjected to the first secondary distillation. The gas used was 117 liters = 959 calories. The distillate still having a pale yellowish tinge made it necessary to redistill it. The oil remaining behind in each case (B.P. above 218°) was added to the creosote fraction. After the second distillation which required 85 liters of gas or 697 calories, an attempt was
made to sublime it but this failed as the apparatus did not prove very efficient, the surface upon which the crystals were to form not being cool and large enough. It was impossible to set up a larger apparatus in the laboratory. The product after the second distillation was as pure as could be obtained under the circumstances. The appearance of the crystals upon close examination showed that a very faint yellow tinge was still present and the odour of coal tar predominant showing that a small quantity of oil still existed within the mass of naphthalene crystals. To make a quantitative determination of this product even this small quantity of oil had to be removed. To accomplish this, the crystals were put upon a porous plate and squeezed. The plate absorbed the oil completely leaving the pure white crystals, M.P. 78.1°. Total amount 147.62 grams or 15.22%.

Naphthalene obtained in this manner turns pink upon standing due very likely to the formation of rosalic acid or compounds of
phenol of which a very slight trace may be present. To obtain naphthalene which will not colour after standing it is advisable to treat in the following manner: The naphthalene obtained was melted and thoroughly mixed with manganese dioxide (5% or 8 grams). The substance was kept just above its melting point by allowing hot water to run on the separatory funnel containing the mixture and sulphuric acid sp.gr. 1.7 added (10% or 15 grams). The whole was kept in a liquid condition and agitated for 35 minutes. The naphthalene was then placed in a flask and distilled for the third time. Gas used for this distillation was 52 liters or 436 Calories.

DESCRIPTION OF A NAPHTHALENE STILL.

The diagram on the following page gives an illustration of a small naphthalene still which could be used for either commercial or laboratory purposes. At the present day the only still that separates naphthalene and the oil, described in the foremost book
treating on this subject, consists of a barrel filled with twigs placed over the vessel containing the mixture. A fire applied under-neath volatilizes the desired product which passes up the barrel depositing in crystalline form on the twigs, etc. This is at best but a very crude method due to the incomplete recovery, the naphthalene having a marked tendency to cling to wood. An improvement over this apparatus can be found in that suggested in the diagram. "A" is the receptacle for the oil and naphthalene which is introduced through an opening "B" which can be tightly closed with the cap "b". A six inch pipe fitting tightly to the opening provided for it in the receptacle serves to conduct the vapors to the condenser. The length of this pipe which is made of wrought iron is 3-3/4 feet. The holder "A" is made of boiler plate 3/16" in thickness. A plate is fastened to the top of the holder which is fastened on with bolts which can readily be taken out enabling the top to be lifted off so that the receptacle can be cleaned when necessary.
A cock "D" permits the oil to be taken out after all of the naphthalene has vaporized. "E" is a cast iron box 1/4" x 19" x 3' which serves to hold the water that causes volatilization to take place. The water is introduced through a 1-1/2" pipe "F" while "G" allows the steam to escape to the air. "H" is a tap for drawing off the water when it is found necessary to remove it. Like the holder, this water box has a removable top fitting tightly at all joints which when removed allows the removal of the receptacle "A". A temperature of 115° C. can be obtained within this tank when the water containing calcium chloride is heated up to its boiling point. Gas flames issuing from numerous holes in a circular bent pipe supply the heat for this operation.

The receiver or condenser is a cylindrical boiler plate tank 3-1/2 x 5-1/2 feet with double walls, the space between being 1-1/2 inches. The pipe "G" has a hood "I" made of wire gauze 200 mesh to the inch which prevents the crystals from falling back into the pipe. A
circular shaped pipe "K" similar to that used for heating the water is placed directly over the condenser. This pipe carries the water which issuing from the numerous holes strikes the top plate of the receiver. The path of the water as indicated by the arrows is down between the double wall and out of the cock "M". A small platform at the bottom conducts the warm water to the outlet. As shown in the diagram, the outer walls project about 1-1/2 inches above the top and reach to within the same distance of the bottom platform. A cover-plate "N", 9 inches in diameter, at the top tightly fits into an opening which has its edges raised 1-1/3" to prevent the water from leaking through into the condenser. The copper coil pipe "O", 3/4" in diameter, admits water at "P" and delivers it at the opening "R". The coil is wound in a conical shape rather than in a cylindrical form, because in the former case the cooling surface intercepts the passage of the hot vapors whereas if the latter arrangement were employed the vapors would rise to the
top and the condensation would not be as rapid. The naphthalene crystals which cling to the pipe can be removed by opening the cover "N" and striking the coil lightly with an iron rod. The opening "P" permits the withdrawal of the crystalized product.

SEPARATION OF PHENOL.

Phenol distilled over between the temperatures 120° - 218°. Above that temperature the distillates are free from it. It will be remembered that we have three quantities with which to work, the alkaline, acid and water washings of naphthalene and the alkaline washings from the light oils, all of which contain some phenol. The fraction 130° - 190° of light oil is the only one washed with sodium hydroxide to free it from carbolic acid. All of the alkaline solutions were put together after having first made the water washing of naphthalene to convert any phenol present into the phenolate. The acid washing was neutralized with a saturated solution of sodium hydrate and
then made alkaline with sodium hydrate (Sp.gr. 1.15). The four liquids were of a milky appearance especially the alkaline washings from the naphthalene and the light oils. The other two were slightly turbid in appearance and contained a thin layer of oil which was separated and added to the light oil. The products were then put into a small distilling flask and distilled with a condenser up to 218°. The amount of gas used was 38 liters or 311 Calories, the residue a few cc. of naphthalene and oils was discarded. The distillate a liquid of milky appearance was then fractionated into two parts up to 180° and from 180° - 218°. The amount of gas for both distillations 33 liters - 270 calories. During this process the condenser was used and great care was taken to have the receptacle fit very closely to the end of the condenser. The fraction 180° - 218° was placed in a freezing mixture with the object of crystallizing out the phenol. A few crystals were obtained in this manner but it could hardly be assumed that all of the carbolic acid
would separate out in this manner. Commercially this process is actually pursued, but as this is a quantitative determination, this method could not be followed. A few cc. of bromine were added to the fraction up to 180° and an immediate precipitation of dibrom phenol, a light yellow substance, took place. The second fraction 180° - 218° was then treated in a similar manner but no precipitation occurred upon the addition of bromine. Apparently as the second fraction was more concentrated than the first, the non precipitation must have been due to its being too concentrated. The inference proved to be carried as upon diluting this fraction the formation of dibrom-phenol rapidly took place. The precipitate was filtered, washed with water, dried over calcium chloride and weighed.

Amount of dibrom phenol = 30.39 gr.
" " phenol = 11.30 "
γ " " = 1.16

CREOSOTE OIL.

Sp.gr. = 1.07
The creosote oil was distilled over between the temperatures 240 - 370°. It contained essentially naphthalene, anthracene, phenol, anilene, methyl naphthalene and phenanthrene. The naphthalene was the only product separated. This was returned to the third fraction.

Creosote oil was obtained from the light oil, carbolic oil and anthracene fractions. These were all put into one receptacle and treated for naphthalene. The naphthalene was removed by decanting off the supernatant oil and placing the crystals in a press. After this operation they were squeezed out on a porous plate in the manner previously described. As creosote oil in this state finds great commercial application, nothing further was done with it. This product contains essentially naphthalene, methyl naphthalene, anthracene, phenanthrene, and the hydrocarbons coming in between these; phenol, cresol, etc.; aniline and similar bases. Naphthalene is soluble in creosote oil, hence the product which was sepa-
rated out was only the undissolved portion. The authors are, however, inclined to think that but very little of this substance remained in solution as the product upon distillation showed no signs of solidifying, characteristic of naphthalene distillation.

Professor George Lunge in his treatise on Coal Tar and Ammonia gives the following uses of the creosote oil.

1. Passing through red hot tubes to obtain illuminating gas and more readily saleable hydrocarbons.
2. Pickling timber.
4. Preparation of varnishes.
5. Burning for heating purposes.
7. Preparation of cheap mineral oils in lieu of Linseed oil.
8. Burning for lamp black.
9. Lighting.
11. As an antiseptic.

In the production of illuminating
gas a retort made of fire brick, heated from below is used. It is heated up to a very bright red heat; and a continuous jet of heavy oil run into it by a swan neck pipe. The volatile products are condensed by an ordinary hydraulic main. In this main most of the undecomposed oil was condensed; the remainder, together with naphthalene and the more volatile hydro carbons newly formed, were condensed in cooling pipes. The uncondensable products are led away to be used for lighting.

It was been known for a long time that timber saturated with creosote oil will keep from rotting when exposed to moisture. For this purpose an apparatus has been devised which causes the wood to take up nearly one gallon of oil per cubic foot. The timber is placed on an iron bogie frame, which is run upon rails into the cylinder, whose front is then firmly closed. The air is now exhausted till the air gauge indicates about 1/6 or 1/8 atmosphere; communication is now opened by a cock with a tank holding the creosote oil, heated to a temperature of
about 50° C. The oil at once rushes into the vacuum and deeply penetrates into the pores of the wood. When no more oil is sucked up, a joice pump is started which presses more oil into the cylinder, till a pressure of 8 to 10 atmospheres has been obtained. This is kept up until the wood is saturated to the extent stated above.

In using creosote oil for lighting purposes, the oil is caused to flow slowly through a jet. Steam under pressure is now forced through the same jet, drawing with it a certain quantity of air. The steam acts on the oil forming a gaseous mixture which burns with a very white flame, not attained by ordinary lamps, because in them, when enough air is admitted the flame becomes hotter but not more luminous. The steam should be as dry as possible; and the condensed water must be allowed to run away.

The preparation of creosote oil for lubricating purposes consists essentially in the removal of the phenols which promote friction. This is done by adding dry, slaked lime.
and rosin oil. The mixture is stirred for some time to assure complete reaction between the lime and phenol, after which it is allowed to stand. The supernant oil is drawn off and used.

The other uses mentioned are not nearly as important as those described and will therefore not be discussed.

ANTHRACENE OIL:

Anthracene oil, or "green oil" as it is called, forms the fraction coming over above 270°. This fraction consists of a brownish green oil with a few greenish yellow crystals adhering to the sides of the flask. The distillation was carried on up to coke and 93.9 grams of distillate were obtained. This fraction contains a great many bodies of high boiling point, the most valuable being anthracene from which the dye alizarin is made. Anthracene is the most valuable product in coal tar and some distillers prefer obtaining nothing but anthracene from the tar.

It is present only in small quantities, the per-
Percentage varying between 0.25 percent to 0.7 percent.

The laboratory method of treating the fraction consists of cooling it and separating the crystals from the oil. The crystals are then mixed with one-third of their weight of potassium carbonate and distilled from an iron retort. Sixteen liters of gas were necessary for the distillation which represents 131 calories. This removes certain impurities, among them carbazole \((C_6H_4)\_2NA\) which is present in considerable proportions in crude anthracene and is thus converted into a non-volatile potassium derivative \((C_6H_4)\_2NK\). The distillate consists almost wholly of anthracene and phenanthracene, which is separated by means of carbon disulphide in which only phenanthracene is soluble. On re-crystallization from benzene the anthracene is obtained pure. The amount of anthracene obtained was 2.7 grams which is almost 0.3%. There was a loss of 6.2 grams, this perhaps being the carbazole which combined with the potassium. The remaining oil, 90 grams in weight, was added to the
creosote fraction.

The working up of the anthracene oil in commerce consists essentially in the separation of the solid hydrocarbons from the liquid ones by cooling and pressing. The latter go back to the heavy oil or creosote fraction or are redistilled.

The anthracene oil is put into shallow iron pans not over 18 inches deep and cooled. Then the crystallization is complete, the pasty mass, consisting of about 10 percent of pure anthracene is forced into bag filters; here the oil is pressed out. In some plants centrifugal machines or filter presses are used which work a good deal better as some of the crystals are forced through the canvas in bag filtering. The pressing is always done in a cold room so that the anthracene will be prevented from going into solution again. The crystals are then pressed in hydraulic presses heated by steam. This raises the proportion of anthracene up to 30-35%. The pressed anthracene is now ground up and treated with solvent naphtha which removes the phenanthra-
cone and other bodies. The anthracene is again pressed and then dried, when it is sold to the dye manufacturers. It contains about 70% of pure anthracene.

Anthracene crystallizes in colorless, glistening leaflets, with a fine fluorescence; it melts at 213°, and boils at 351°; it dissolves readily in boiling benzol.

**PITCH:**

After the anthracene oil was distilled the heat was increased and a dark heavy oil came off. The oil is very combustible and great care must be taken so that it will not catch fire. 16.7 grams of oil were obtained. When all the oil had been distilled the still was allowed to cool and the coke left in the still scraped out and weighed. Its weight was 19.7 grams or about 3% of the total. The gas necessary for the distillation up to coke was 160 litres which represents 1512 calories.

All of the primary distillations were made in a small copper still having a total ca-
capacity of about 2-1/2 liters. The products obtained were then redistilled in glass distilling flasks. As a complete distillation was aimed at, a great deal of difficulty was experienced in preventing losses, the different oils having a tendency to stick to the sides of the flask which always made a loss of a few grams. This was partly overcome by receiving the products in a flask from which the distillation could be carried on directly so that it would not be necessary to transfer the product to another flask.

It would probably be well to state the prices of the different coal tar products before ending this paper. From this list of values and the time and labor taken to obtain the different fractions, it can readily be calculated which constituent in the tar would be most profitable to work up commercially:

<table>
<thead>
<tr>
<th>Product</th>
<th>Specification</th>
<th>Price</th>
</tr>
</thead>
<tbody>
<tr>
<td>Anthracene Oil 22° Tw.</td>
<td>per gal.</td>
<td>.02-1/2</td>
</tr>
<tr>
<td>Benzole 50/90% f.o.b.</td>
<td></td>
<td>.18</td>
</tr>
<tr>
<td>&quot; 90%</td>
<td></td>
<td>.20</td>
</tr>
<tr>
<td>Cryst. Carbolic Acid 34/35° C.</td>
<td>per pound</td>
<td>.14</td>
</tr>
<tr>
<td>&quot; 39/40° C.</td>
<td></td>
<td>.15</td>
</tr>
<tr>
<td>Liquid &quot; 97/99%</td>
<td>gallon</td>
<td>.22</td>
</tr>
<tr>
<td>Crude &quot; at 600 F.</td>
<td></td>
<td>.42</td>
</tr>
<tr>
<td>Creosote</td>
<td></td>
<td>.01-3/4</td>
</tr>
<tr>
<td>Light Oil</td>
<td></td>
<td>.07</td>
</tr>
<tr>
<td>Naphtha crude 20% at 120° C.</td>
<td></td>
<td>.08-1/2</td>
</tr>
<tr>
<td>&quot; solvent 90% at 160° C.</td>
<td></td>
<td>.243</td>
</tr>
<tr>
<td>&quot; 95% &quot; 190°</td>
<td></td>
<td>.263</td>
</tr>
<tr>
<td>Pitch,</td>
<td>ton</td>
<td>7.30</td>
</tr>
</tbody>
</table>
On the following page will be found a summary of the results obtained as described in the previous pages of this paper. The amount of each product and the heat necessary expressed in large calories for each distillation may be found on this page.