No. 755,309. PATENTED MAR. 22, 1904.

A. NIKIFOROFF.

MANUFACTURE OF THE BENZOLS AND THEIR HOMOLOGUES.

APPLICATION FILED SEPT. 8, 1902.

NO MODEL. 3 SHEETS—SHEET 1.

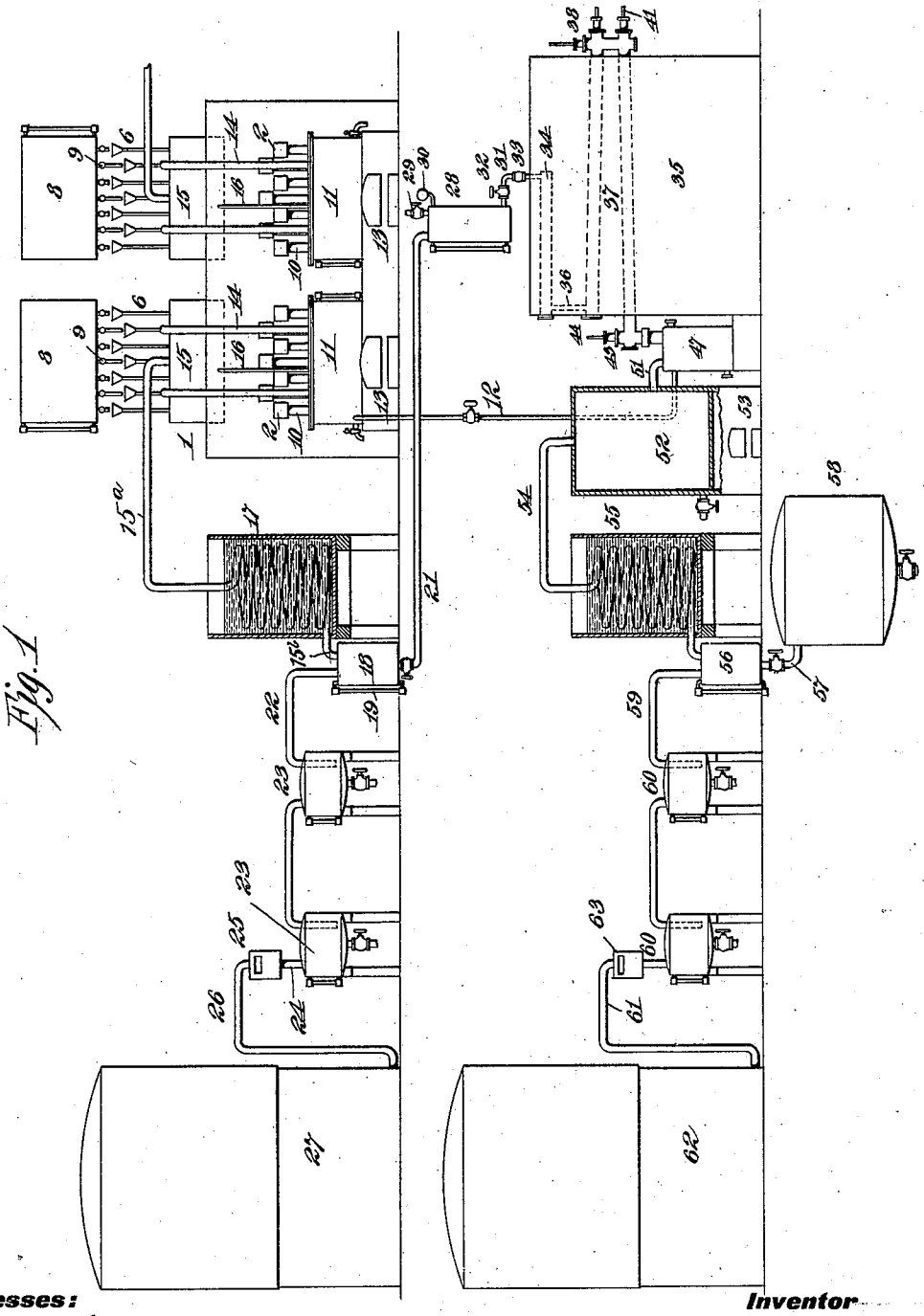

Fig. 1

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NO MODEL. 3 SHEETS—SHEET 2.

Fig. 3

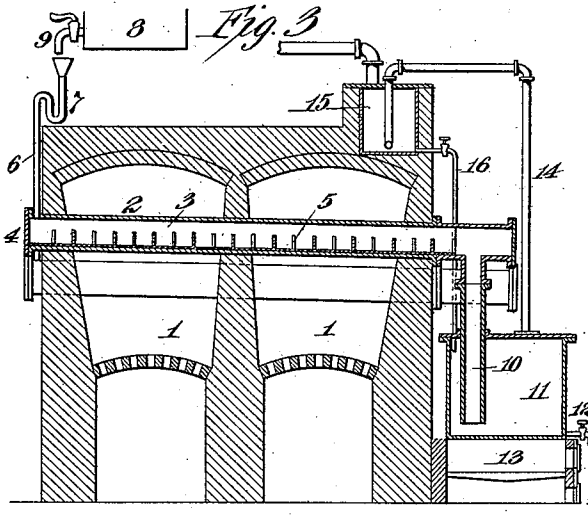


Fig. 2

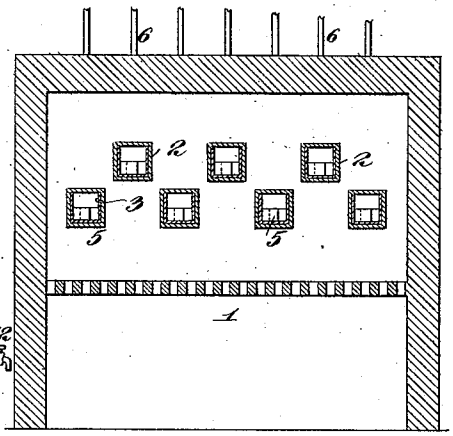


Fig. 4

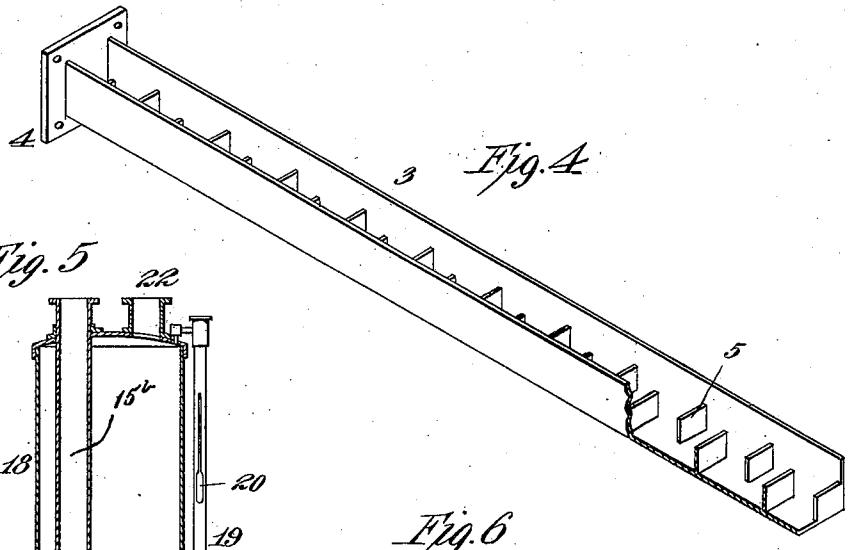
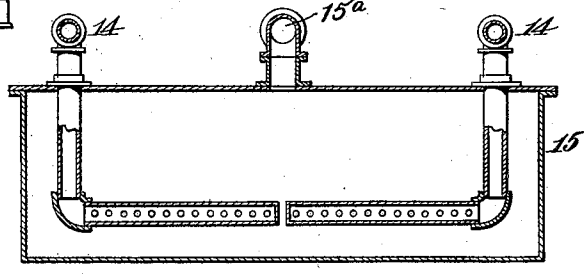


Fig. 5

Fig. 6



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NO MODEL. 3 SHEETS—SHEET 3.

Fig. 7

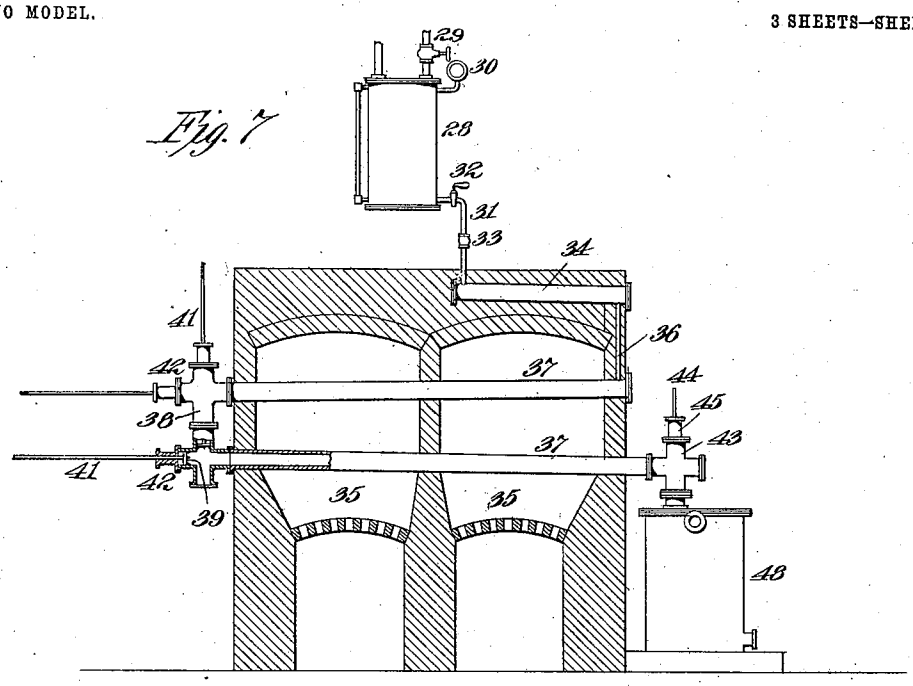


Fig. 8

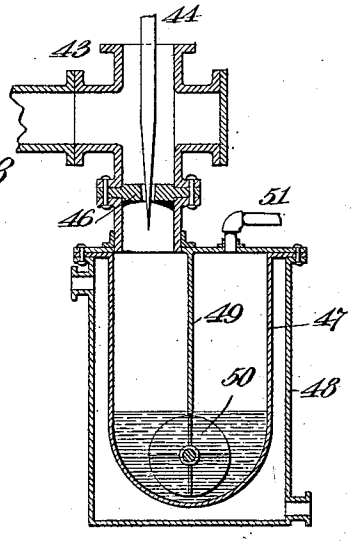


Fig. 9

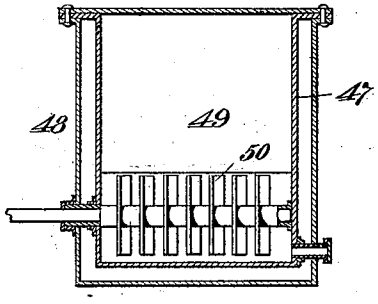
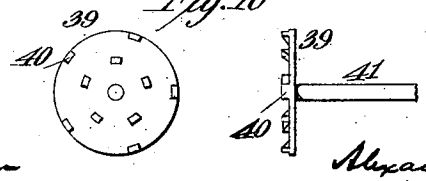


Fig. 10



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No. 755,309. Patented March 22, 1904.

UNITED STATES PATENT OFFICE.

ALEXANDER NIKIFOROFF, OF MOSCOW, RUSSIA.

MANUFACTURE OF THE BENZOLS AND THEIR HOMOLOGUES.

SPECIFICATION forming part of Letters Patent No. 755,309, dated March 22, 1904.

Application filed September 8, 1902. Serial No. 122,531. (No specimens.)

To all whom it may concern:

Be it known that I, ALEXANDER NIKIFOROFF, a subject of the Emperor of Russia, and a resident of Moscow, Russia, (postal address, Rojdestvenka 8,) have invented certain new and useful Improvements in the Manufacture of the Benzols and Their Homologues, of which the following is a specification.

My invention relates to improved processes for manufacturing benzol, its homologues and compounds of the aromatic series from crude petroleum, petroleum residue, heavy oils resulting from the distillation of bituminous coal and the like, and from the residuum resulting from the manufacture of oil-gas—*i. e.*, the final liquid by-products or tailings, including any other source of heavy liquid or solid hydrocarbon from which hydrocarbons of the aromatic series may be derived, all of which will be hereinafter referred to as "raw material."

My invention is based on the fact that crude petroleum and the heavy hydrocarbons above referred to, besides containing hydrocarbons of the paraffin series, also contain hexahydrocyclic compounds. I find that by subjecting to the combined effect of high temperature and pressure the volatilized products resulting from the decomposition of the raw materials referred to and having a boiling-point below approximately 200° centigrade I am enabled to effect the dehydrogenation of the more or less simple hydrocarbons and to effect the decomposition with subsequent dehydrogenation of the more or less complex hydrocarbons, resulting in the formation of benzol and of a whole series of its derivatives, such as toluol, xylol, cumol, naphthalene, anthracene, &c. In this way I am enabled to carry my process into effect expeditiously and economically and when the original raw material is free from sulfur the resulting benzol obtained is entirely free from thiophene, the presence of which is unavoidable and detrimental in connection with benzol as obtained by other processes heretofore suggested and which requires elaborate treatment for its removal.

Benzol, toluol, and other hydrocarbon combinations of the aromatic series obtained by my process are identical in their useful properties with corresponding combinations secured by other and less advantageous processes at the present time. Thus, for instance, the benzol obtained by my process is capable of being fully nitrated into nitrobenzol, while the anthracene can be converted into alizarin with perfect dyeing properties.

My object generally is to provide a new and useful process for the treatment of heavy hydrocarbons for the production therefrom of benzol, its homologues and compounds of the aromatic series which shall be simple, effective, and capable of operation under conditions of the highest economy.

Broadly stated, my improved process consists, first, in subjecting the heavy hydrocarbon to decomposition, whereby the products vaporizing substantially below 200° centigrade are removed, so that at the very commencement of the operation I provide a product capable of being effectively operated upon by the subsequent treatments and eliminate the heavy tarry residuums, which would otherwise clog the apparatus and retard the operations; second, in subjecting the distillate thus secured, volatilizing substantially below 200° centigrade, to the effect of high temperature under a pressure above the atmospheric, whereby some of the hydrocarbons are dehydrogenated, while the complex hdrocarbons are first decomposed and subsequently dehydrogenated, and in separately recovering the aromatic distillate thus secured, and, third, in subjecting the separate aromatic distillates obtained as explained, if necessary, to the effect of fractional distillation or other treatment in any well-known way for the recovery of benzol or any of its derivative aromatic compounds, all as will be more fully hereinafter described and claimed.

In order that the invention may be better understood, attention is directed to the accompanying drawings, forming part of this specification, in which—

Figure 1 is a diagrammatic view illustrating a suitable apparatus for carrying the process into effect; Fig. 2, a transverse sectional view of the retort-furnace for subjecting the raw material to a preliminary decomposition; Fig. 3, a longitudinal sectional view of the same

through one of the retorts; Fig. 4, a separate sectional perspective of one of the troughs employed in each of the decomposition-retorts; Fig. 5, a sectional view of the tank for receiving the useful distillate secured in the preliminary decomposition operation; Fig. 6, a view, partly in section, of the separating-tank employed for separating the useful distillate from the products of distillation or decomposition obtained in the first decomposition and whose boiling-point is approximately above 200° centigrade and below 250° centigrade; Fig. 7, a separate sectional view through the retort in which the second operation is performed for subjecting the useful distillate of the first decomposition to the combined effect of high temperature and pressure; Fig. 8, a separate sectional view illustrating the wash-tank for washing the distillate of the second operation and showing also the needle-valve for regulating the pressure in the high-temperature retorts; Fig. 9, a longitudinal sectional view of the wash-tank, and Fig. 10 detail views illustrating the preferred form of scraper for removing sediment from the high-temperature retorts.

In all of the above views corresponding parts are represented by the same numerals of reference.

The decomposition-furnace comprises the furnaces 1 1 and a plurality of inclined retorts 2, made, preferably, of cast-iron. Within each retort is a removable trough 3, whose flange 4 forms one end of the retort and is bolted in place. Each of these troughs is provided with staggered baffle-plates 5, so that the hydrocarbon in flowing down the same will be caused to take a tortuous course. When the troughs become objectionably clogged with the heavy residuum resulting from the first decomposition, they may be removed and cleaned, as will be understood. The several decomposition-retorts 2 are supplied with raw material through feeding-pipes 6, having traps 7 to form a seal. These feeding-troughs receive material from a tank 8, having a plurality of valved nozzles 9 corresponding with the several feeding-pipes. A discharge-pipe 10 leads from each decomposition-retort and extends into a tank 11, having a valved draw-off pipe 12 and heated in any suitable way, as by a furnace 13, so that its temperature shall be approximately 250° centigrade. It is to be understood that this furnace is used only in beginning operations. Afterward its use will not be necessary. Leading from the tank 11 is a vapor-pipe 14, entering a separator 15 and equipped at its lower end with a perforated extension (see Fig. 6) located close to the bottom of the separator, as shown. The separator 15 is arranged above the retort-furnace, and its temperature is maintained at from 175° to 200° centigrade by the heat of the furnace or in any other way. A valved pipe 16 connects the bottom of the separator 15 with the tank 11, as shown. A vapor-pipe 15[a] leads from the top of the separater 15 to a condenser 17 and thence, as indicated at 15[b], to a receiving-tank 18, provided with a glass gage 19, having a hydrometer 20 therein, by means of which the specific gravity of the distillate deposited in the receiving-tank 18 can be observed at any time. Leading out of the bottom of the receiving-tank 18 is a valved draw-off pipe 21, and out of the top of that tank is a vapor-pipe 22, connected with a series of solvent-tanks 23, as shown, supplied with any suitable solvent, such as acetone or methylic alcohol. From the final solvent-tank a vapor-pipe 24 leads to a gas-meter 25, by which the volume of substantially permanent gas leaving the solvent-tanks may be measured. A vapor-pipe 26 conveys the uncondensed vapors to a gas-holder 27 of any suitable construction.

The valved draw-off pipe 21 from the receiving-tank 18 leads to a supplying-tank 28 for furnishing to the high-temperature retorts the distillate useful for the production of hydrocarbons of the benzol series. This supply-tank is furnished with compressed air, compressed carbonic-acid gas, or other neutral gas through a valved pressure-pipe 29, and the pressure is indicated on a gage 30. Leading from the supply-tank 28 at its bottom is a pipe 31, furnished with a cut-off valve 32 and with a check-valve 33, the latter permitting the liquid to flow out of the supply-tank, but preventing a flow of the liquid in the opposite direction. The pipe 31 leads to a supplemental retort 34 in the top of the furnaces 35 and by means of which the useful distillate will be vaporized, so as to be supplied to the high-temperature retorts in a vaporized condition, whereby I am enabled to obtain a more intense and uniform decomposition in the latter retorts.

From the auxiliary retort 34 a pipe 36 leads to the high-temperature retorts 37, connected together in series, in order that the vapors may be subjected to the effect of high temperature and pressure for a relatively prolonged period. The retorts 37 are connected by a pair of four-way castings 38, as shown, in which are mounted a plurality of scrapers 39, (see Fig. 11,) having teeth 40 and carried on rods 41 passing through stuffing-boxes 42. By operating these scrapers I provide for the effective cleaning of the high-temperature retorts as well as the connections between the same, as will be understood. A door 38[a] at the bottom of the casting allows of its being cleansed. The discharge end of the lowermost retort 37 is furnished with a four-way casting 43, in the vertical passage of which is mounted a needle-valve 44, passing through a stuffing-box 45. The tapered lower end of this needle-valve coöperates with the tapered opening in a disk 46, so that by moving the needle-valve more or less within the opening the diameter of the latter may be regulated to thereby provide

for the desired retardation of the flow of the products of decomposition through the same, with a consequent regulation of the pressure within the high-temperature retorts. Connected with the discharge from the high-temperature retorts is a washer (see Fig. 8) comprising a receptacle 47, having a curved bottom, as shown, and contained in a water-jacket 48, by which the receptacle may be always kept cool. Arranged within the receptacle 47 is a central vertical partition 49, dividing the same into two chambers, and below this partition is an agitator 50 of any suitable construction, the shaft of which is rotated from any desired source of power. From the washer a vapor-pipe 51 leads to a tank or vessel 52 for containing naphthalene-oil and anthracene-oil and which is heated in any suitable way—as, for example, by a furnace 53 beneath it—so that its temperature shall be approximately from 200° to 225° centigrade. A vapor-pipe 54 leads from the top of the vessel 52 to a condenser 55 and thence to a receiving tank or vessel 56, preferably corresponding in construction with the tank 18 and which therefore does not require detailed illustration. A valved draw-off pipe 57 leads from the bottom of the tank 56 to a receiving vessel 58 for receiving the aromatic distillate. Leading from the top of the vessel 56 is a vapor-pipe 59, connected with a series of solvent-tanks 60, supplied with a suitable solvent, such as acetone or methylic alcohol, and leading from the final solvent-tank of the series is a vapor-pipe 61, supplied with a gas-meter 63 and connected to a suitable gas-holder 62.

In the carrying out of my improved process for the treatment of any suitable raw material—such, for example, as crude Balakhani petroleum—I proceed substantially as follows: The crude material is supplied to the tank 8 and from the latter fed to the several decomposition-retorts 2 through the supply-pipes 6. In flowing through the troughs 3 the passage of the liquid material is obstructed by the baffle-plates 5, so that it partakes of a tortuous course, and consequently is subjected to the effect of the heat for a relatively prolonged period. In these decomposition-retorts the crude material is subjected to a temperature of from 500° to 525° centigrade, and the material will be thus evaporated and partly decomposed. The volatile products of the decomposition or evaporation pass through the vapor-pipe 10 into the tank 11, which is maintained at a temperature of approximately 250° centigrade. Any vaporized products volatilizing at a temperature above 250° centigrade will thereby condense and accumulate in the tank 11, from which they may be withdrawn from time to time as desired. The liquid collected in tank 11 corresponds in its character to the residuum obtained in the distillate of crude kerosene-oil, and its use, beyond the small portion employed for charging the washer 47, is for fuel or for the manufacture of lubricating-oil. The vapors having a boiling-point below 250° centigrade pass through the vapor-pipe 14 and enter the separating-tank 15, maintained at a temperature of from 175° to 200° centigrade. In this separating-tank I condense the vapors whose boiling-point is 200° centigrade or more. The more volatile vapors, boiling below approximately 200° centigrade, pass through the vapor-pipe, and their condensable portion is condensed by the condenser 17 and accumulates in the receiving-tank 18. Any non-condensable gases will be conducted through the several solvent-tanks 23 and their soluble constituents removed by the solvent therein. The solution thus secured can be subjected to any desired distilling process for the separation of the solvent from the dissolved hydrocarbon. The hydrocarbon recovered from the solvent-tanks contains volatile parts of useful distillate—*i. e.*, principally a small quantity of benzol—resulting already at this period of the decomposition. From the solvent-tanks 23 the permanent gas passes through the gas-meter 25, by which its volume is measured, and enters the gas-holder 27, from which it may be removed for use. The gas obtained in this manner is a final product, and in the example given with Balakhani petroleum will amount to about 12.85 per cent. of the amount of material used and finds ready use for heating, lighting, and other industrial purposes.

I find in practice that in the tank 15 is collected and condensed a small proportion of distillate vaporizing under a temperature of 200° centigrade and having useful properties in this art. This residuum consists of a mixture of a material somewhat similar to the residuum as in tank 11, with a portion of the condensed vapors which in the course of operation condense before reaching the condenser 17. In order that these volatile products can be recovered, it is only necessary to withdraw the contents of the tank 15 into the vessel 11 through the valved pipe 16, (see Fig. 3,) whereby the relatively high temperature of the latter (250° centigrade) will again evaporate and distillate and carry it over into the tank 15. This may be done from time to time as most convenient to the operator.

The troughs 3 after a few hours of continued use accumulate porous coke, and in order that this may be removed the several troughs are withdrawn, preferably one at a time, and are replaced by fresh troughs. By removing the troughs one at a time, as explained, I am enabled to operate the apparatus continuously.

I find in practice that by maintaining an approximately constant temperature in the retort-furnaces I am enabled to keep the process of decomposition under perfect control by merely adjusting the supply of raw material to the retorts in such a way as to keep

the specific gravity of the condensed distillate in the tank 18 and the quantity of gas measured by the meter 25 approximately constant, so that in this way the process of decomposition is an extremely simple one.

The useful condensed distillate from the first decomposition collected in the receiving vessel 18 and which amounts in the given example to about fifty per cent. of the entire amount of petroleum operated on is now subjected to the second operation of the process—namely, to the effect of high temperature under a pressure above atmospheric in the high-temperature retorts. It will be noted that by subjecting the raw material to a preliminary decomposition I obtain in the first instance a useful distillate capable of being acted upon directly for the production of benzol, its homologues, and compounds of the aromatic series and have eliminated the heavier residual hydrocarbons, which would otherwise not only clog the subsequent apparatus, but would unduly overload the same. Heretofore, so far as I am aware, in all attempts which have been made to obtain benzol, &c., from crude petroleum the entire bulk of raw material has been subjected to a decomposing operation, necessitating the use of retorts charged with refractory materials, such as as coal, coke, iron, pumice-stone, and the like. These retorts have become quickly clogged, so that the previous experiments have been slow and generally impractical. Thus the expedient of subjecting the raw material to a preliminary decomposition, although simple, is from the standpoint of utility highly important. The valve in the draw-off pipe 21 is now opened and a charge from the tank 18 enters the supply-tank 28. The valve is now closed and pressure is admitted to the supply-tank through the pressure-pipe 29, so that when the valve 32 is opened material from the supply-tank will be forced into the high-temperature retorts. The temperature maintained in the retorts 37 varies from 900° to 1,000° centigrade, and the temperature maintained in the auxiliary retort 34 is obviously somewhat lower, but is always sufficient to effect a vaporization of the liquid hydrocarbon. Consequently the liquid hydrocarbon entering the auxiliary retort 34 will be vaporized, so that it enters the high-temperature retorts 37 in a vaporized condition, whereby I am enabled to obtain a more intense and uniform decomposition in the latter. The subjection of the vapor in the retorts 37 to high temperature results in the generation of a pressure which can be regulated by the needle-valve 44, as explained. In the example given a pressure of about fifteen pounds above atmospheric is maintained. Should this pressure ever overbalance the pressure in the tank 28, the check-valve 33 will be closed, so as to automatically protect the supply-tank 28 from the effect of any accidental excessive pressure which might take place—for example, if the tapered opening in the disk 46 became clogged. This opening can, however, be ordinarily kept perfectly free by means of the needle-valve 44.

The gases and vaporized products of decomposition leaving the high-temperature retorts are conducted to the washer 47, which is supplied with the residuum from the first decomposition drawn from the tank 11 through the pipe 12 or any other distillate boiling above 250° centigrade obtained by any process to just submerge the lower end of the partition 49, and thereby form a seal between the two chambers. In this washer the products of decomposition are cleaned of any particles of fine soot which may become mechanically entrained therewith. From the washer the vaporized products of decomposition are conducted to the tank 52, maintained at a temperature of from 200° centigrade to 225° centigrade and in which the materials condensing at that temperature are collected, principally and essentially anthracene oil and naphthalene oil. The condensable vapors whose boiling-point is below approximately 200° centigrade are condensed by the cooler 55 and accumulate in the tank 56, from which they may be removed and accumalated in the tank 58. This condensed distillate I refer to as the "aromatic" distillate and in the example given amounts to about 21.50 per cent. of the original material. The uncondensed vapors from the tank 56 pass through the solvent-tanks 60 and any soluble constituents are removed therefrom, the substantially permanent gas passing through the gas-meter 63 to the gas-holder 62, from which it may be used when desired. The gas thus obtained in the example given is about 16.15 per cent. of the original material, is a final product, and may be used for the same purposes as the gas obtained with the holder 27, either alone or mixed with the gas obtained by the first decomposition.

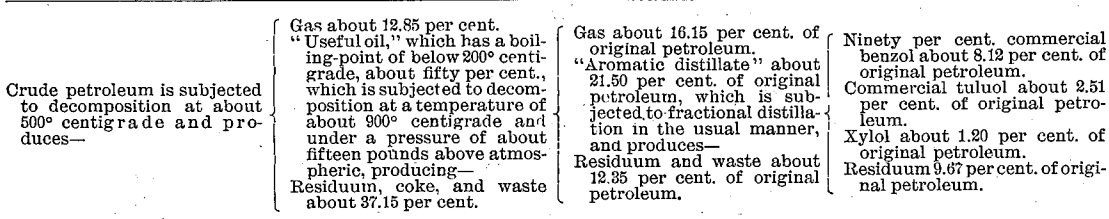
As a result of the process of decomposition described effected at high temperature and under a pressure above atmospheric I secure products which can be subjected to any desired subsequent treatment for the obtaining of useful hydrocarbons by any of the well-known processes. For instance, the products condensed in the tank 52, having a boiling-point above approximately 200° or 225° centigrade, can be subjected to any well-known process for obtaining naphthalene or anthracene, the aromatic distillate received in the tank 58 can be subjected to fractional distillation in well-known ways to obtain benzol and its immediate derivatives, while the solution in the solvent-tanks 60 can be diluted with water and regenerated, if acetone or methylic alcohol is used as absorbents, for the recovery of aromatic distillate. If absorbents are used which are not soluble in water, the solution can be subjected to fractional distillation.

The aromatic distallate, either alone or in addition to the product in the tank 58, can be subjected to fractional distillation for further refinement.

In the example given—viz., operating upon Balakhani petroleum—the following percentages of the original amount have been obtained: 8.12 per cent. of ninety per cent. commercial benzol, 2.51 per cent. of commercial tuluol, and 1.20 per cent. of xylol.

The following diagram indicates in schematical form a general outline of the entire process:

Example of Process Applied to Balakhani (Caucasian) Petroleum.



It is to be understood that when my improved process is used in connection with other petroleums, or when other petroleums and other substances—such as petroleum residue, heavy oils resulting from the distillation of bituminous coal, and the like—are used as raw materials the temperatures and pressures will be varied, as determined by experiment.

As I have described my process as applied to crude Balakhani petroleum with a temperature at the first operation of about 500° centigrade and the second operation of about 900° centigrade under a pressure of about fifteen pounds above atmospheric, I desire it understood that such temperature and pressure are cited merely as an example. I believe that my process may be successfully applied to some substances by employing a temperature in the second operation of as low as 750° centigrade and that the pressure can be varied within very wide limits.

It will be understood that if my improved process is to be used in connection with the decomposition of solid hydrocarbons the latter may be first fused and then admixed with a sufficient quantity of raw material, as defined above, to constitute a viscous liquid capable of flowing through the decomposition-retorts.

The apparatus described in the specification is not claimed herein, but forms the subject-matter of an application for patent filed January 14, 1903, Serial No. 139,024.

Having now described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The process of treating raw materials, substantially as set forth, for the production of an aromatic distillate capable of yielding benzol, which consists in subjecting the raw material to a preliminary decomposition to separate hydrocarbons having a boiling-point of approximately 200° centigrade or under, and in then subjecting the distillate so secured to the effect of a temperature of about 750° centigrade and under a pressure above the atmosphere, as and for the purposes described.

2. The process of treating raw materials, substantially as set forth, for the production of an aromatic distillate capable of yielding benzol, which consists in subjecting the raw material to the effect of decomposition under a temperature of approximately 500° centigrade, in collecting the vaporized distillate having a boiling-point of approximately 200° centigrade or under, and in subjecting such distillate to the effect of a temperature of about 750° centigrade under a pressure above the atmosphere, as and for the purposes described.

3. The process of treating raw materials, substantially as set forth, for the production of an aromatic distillate capable of yielding benzol, which consists in decomposing the raw material under the effect of heat, in condensing the volatile distillate having a boiling-point of 200° centigrade or under, and in subjecting such distillate to the effect of a temperature of about 750° centigrade under a pressure above that of the atmosphere, in collecting the distillate of the second decomposition whose boiling-point is approximately 200° centigrade or over, and in separately collecting the aromatic distillate of the second decomposition whose boiling-point is 200° centigrade or under, as and for the purposes set forth.

4. The process of treating raw materials, substantially as set forth, for the production of an aromatic distillate capable of yielding benzol, which consists in decomposing the raw material under the effect of heat, in condensing the volatile distillate having a boiling-point of 200° centigrade or under, in subjecting such distillate to the effect of a temperature of above 750° centigrade under a pressure above that of the atmosphere, in collecting the distillate of the second decomposition whose boiling-point is approximately 200° centigrade or over, in separately collecting the aromatic distillate of the second decomposition whose boiling-point is 200° centigrade or under, and in subjecting the uncondensed vapor of the second decomposition to the effect of a solvent, as and for the purposes set forth.

5. The process of treating raw materials, substantially as set forth, for the production of an aromatic distillate capable of yielding benzol, which consists in decomposing the raw material under the effect of heat, in condensing the volatile distillate having a boiling-point of 200° centigrade or under, in subjecting such distillate to the effect of a temperature of above 750° centigrade under a pressure above that of the atmosphere, in collecting the distillate of the second decomposition whose boiling-point is approximately 200° centigrade or over, in separately collecting the aromatic distillate of the second decomposition whose boiling-point is 200° centigrade or under, in subjecting the uncondensed vapor of the second decomposition to the effect of a solvent, and in finally collecting the uncondensed permanent gas, as and for the purposes set forth.

6. The process of treating a hydrocarbon derived from raw material, substantially as set forth, and whose boiling-point is 200° centigrade or less, which consists in subjecting such hydrocarbon to the effect of a temperature of about 750° centigrade under a pressure above atmospheric, and in subsequently condensing and collecting the aromatic distillate, as and for the purposes set forth.

7. The process of treating a hydrocarbon derived from raw material, substantially as set forth, and whose boiling-point is 200° centigrade or less, which consists in subjecting such hydrocarbon to the effect of a temperature of above 750° centigrade under a pressure above that of the atmosphere, in collecting and condensing the aromatic distillate, and in passing the uncondensed distillate through a solvent, as and for the purposes set forth.

8. The process of treating a hydrocarbon derived from raw material, substantially as set forth, and whose boiling-point is 200° centigrade or less, which consists in subjecting such hydrocarbon to the effect of a temperature of above 750° centigrade under a pressure above that of the atmosphere, in collecting and condensing the aromatic distillate, in passing the uncondensed distillate through a solvent, and in subsequently collecting the permanent gas, as and for the purposes set forth.

9. The process of treating a hydrocarbon derived from raw material, substantially as set forth, and whose boiling-point is 200° centigrade or less, which consists in subjecting such hydrocarbon to the effect of a temperature of above 750° centigrade under a pressure above that of the atmosphere, in collecting and condensing the vaporized distillate whose boiling-point is above 200° centigrade, and in separately condensing the aromatic distillate whose boiling-point is above 200° centigrade, as and for the purposes set forth.

10. The process of obtaining benzol from raw materials, substantially as set forth, which consists in subjecting the raw material to the effect of high heat, in condensing the distillate having a boiling-point of 200° centigrade or less, in subjecting such distillate to the effect of a temperature above 750° centigrade under a pressure above that of the atmosphere, in condensing the aromatic distillate derived from the second decomposition, and in fractionally distilling the aromatic distillate so obtained, as and for the purposes set forth.

11. A step in the process of obtaining benzol from a hydrocarbon whose boiling-point is 200° centigrade or less, derived from the decomposition of crude materials, substantially as set forth, which consists in subjecting the hydrocarbon to the effect of a temperature above 750° centigrade under a pressure above that of the atmosphere, in collecting and condensing the aromatic distillate, and in fractionally distilling such distillate, as and for the purposes set forth.

12. A step in the process of treating raw materials, substantially as set forth, preparatory to the decomposition thereof for the production of an aromatic distillate from which benzol can be immediately derived, which consists in subjecting a current of the raw material to contact with a surface heated to 500° centigrade whereby the material is gradually raised in temperature, and in collecting the products of decomposition thus obtained and separating those whose boiling-point is approximately 200° centigrade or less, from those whose boiling-point is above 200° centigrade, as and for the purposes set forth.

In witness whereof I have hereunto set my hand in presence of two witnesses.

ALEXANDER NIKIFOROFF. [L. S.]

Witnesses:
NAUSU MINTZ,
STEFAN VON OTTERETZKY.