July 29, 1924. 1,503,093

R. M. CATLIN

SHALE DISTILLATION APPARATUS

Filed Aug. 4, 1920 3 Sheets-Sheet 1

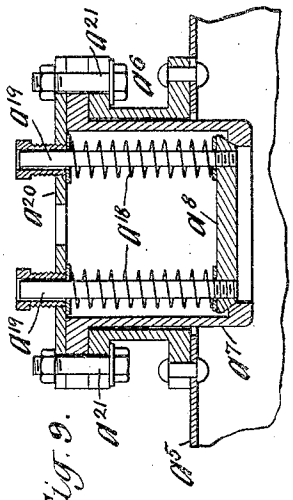


Fig. 9.

Fig. 1.

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Filed Aug. 4, 1920 3 Sheets-Sheet 2

Fig. 2.

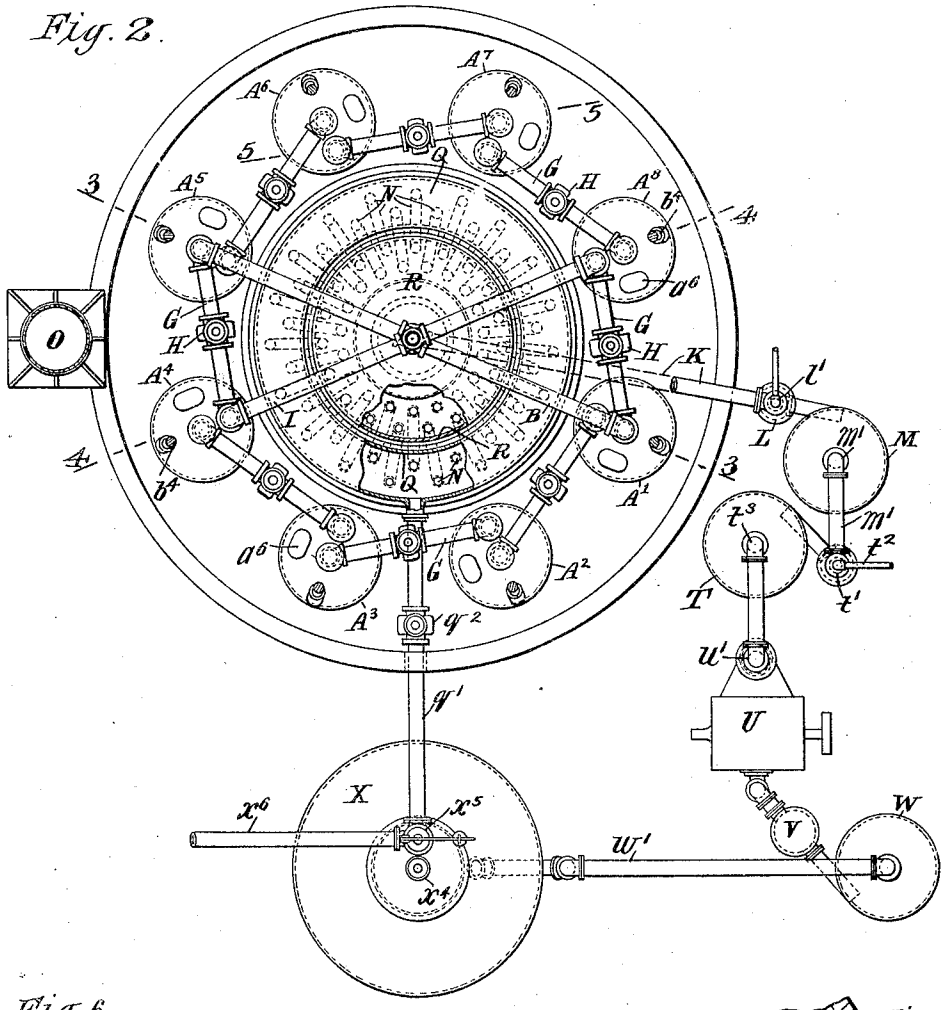


Fig. 6.

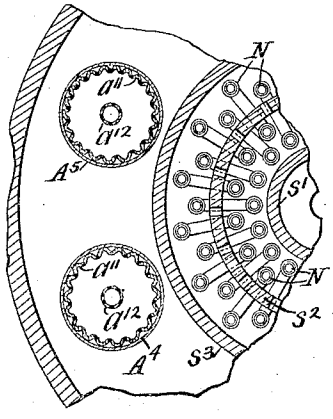
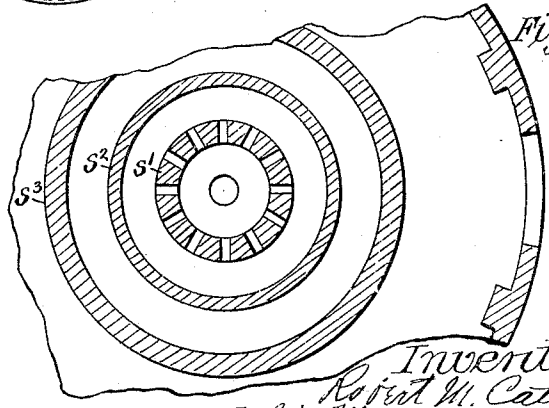


Fig. 7.



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Filed Aug. 4, 1920 3 Sheets-Sheet 3

Fig. 3.

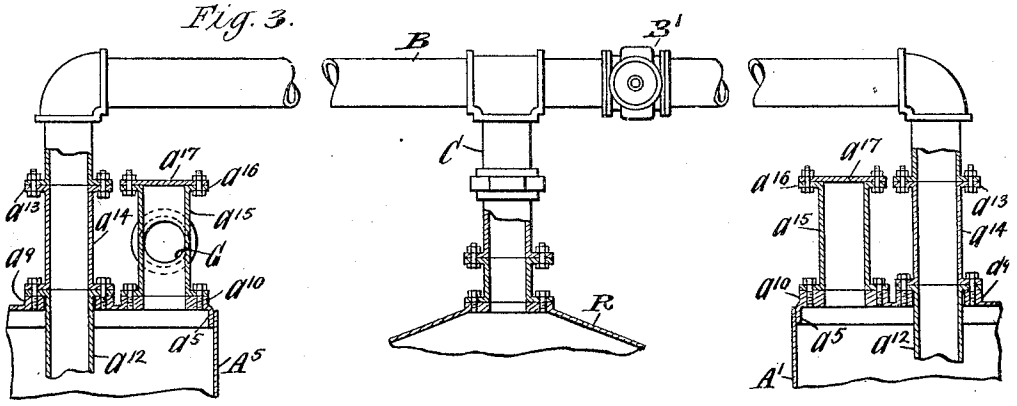


Fig. 4.

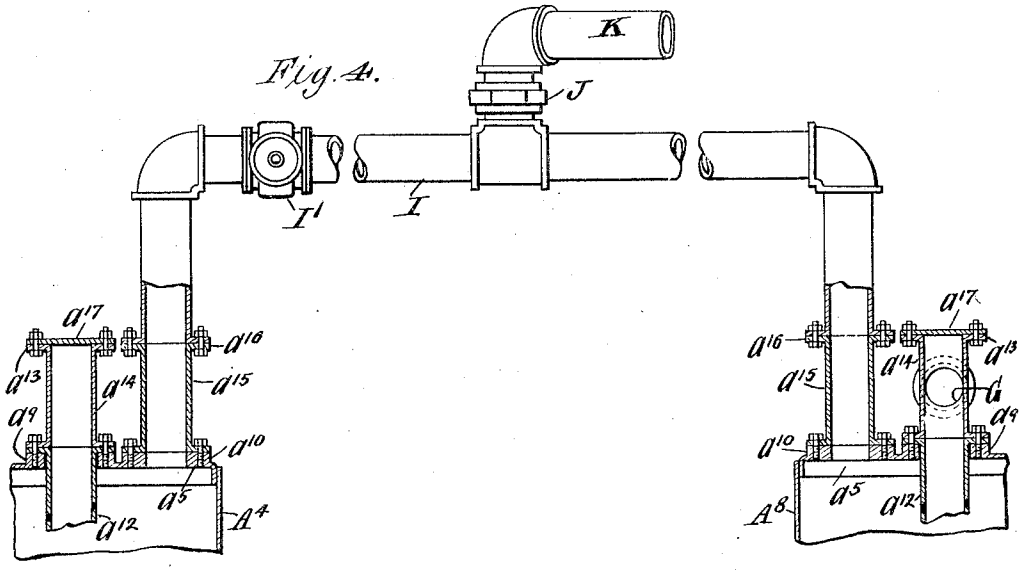


Fig. 5.

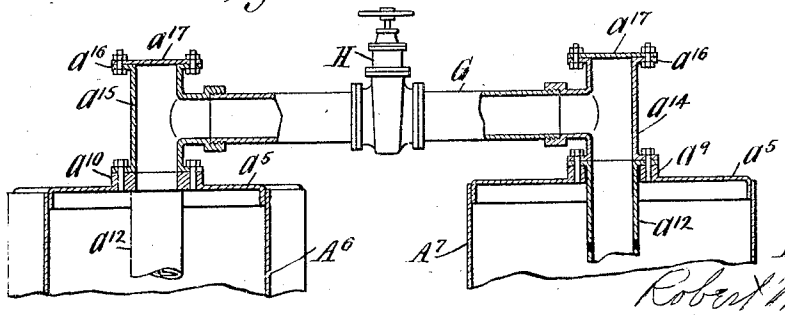
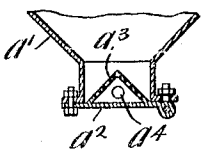


Fig. 8.



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Patented July 29, 1924. 1,503,093

UNITED STATES PATENT OFFICE.

ROBERT M. CATLIN, OF FRANKLIN, NEW JERSEY, ASSIGNOR TO CATLIN SHALE PRODUCTS COMPANY, OF WILMINGTON, DELAWARE, A CORPORATION OF DELAWARE.

SHALE-DISTILLATION APPARATUS.

Application filed August 4, 1920. Serial No. 401,130.

To all whom it may concern:

Be it known that I, ROBERT M. CATLIN, a citizen of the United States, residing at Franklin, county of Sussex, and State of New Jersey, have invented certain new and useful Improvements in Shale-Distillation Apparatus, fully described and represented in the following specification and the accompanying drawings, forming a part of the same.

The present invention relates to improvements in the art of shale distillation, and comprises an improved process as well as an improved apparatus.

The invention will first be described in connection with the accompanying drawings, and then more particularly pointed out in the claims.

In the drawings—

Fig. 1 is a sectional view of the assembled apparatus;

Fig. 2 is a plan view of the same;

Fig. 3 is a sectional view along the lines 3—3 of Fig. 2 showing the means for conducting the heated gases from the superheater to the shale retorts;

Fig. 4 is a sectional view along the line 4—4 of Fig. 2 showing the means for conducting the heated gases from the final stage shale retorts to the apparatus for clearing and cooling the gases used for heating;

Fig. 5 is a sectional view along the line 5—5 of Fig. 2 showing the means for connecting the outlet of one shale retort to the inlet of the adjacent shale retort;

Fig. 6 is a sectional view along the line 6—6 of Fig. 1 showing the heating coils of the gas superheater, and two of the shale retorts;

Fig. 7 is a sectional view along the line 7—7 of Fig. 1 showing the arrangement of the supporting and enclosing brick-work of the furnace, superheater, and retort chambers;

Fig. 8 is a sectional view of the lower end of one of the shale retorts; and

Fig. 9 is a sectional view of the charging manhole and gas valve mounted on the top of each shale retort.

Referring to the drawings, A^1 to A^8, Figs. 1 and 2, are receptacles which may be termed retorts. Each is arranged to receive shale in fragments. In the best embodiment of the invention each receptacle has a conical bottom a^1, as shown in Figs. 1 and 8, closed by a removable cap a^2 having an upwardly extending dome a^3 suitably perforated, so as to admit liquid products of distillation to the space beneath the dome, from whence they can escape through an escape pipe a^4. The cap a^2 may be held in place by a suitable means such, for example, as bolts and nuts, not shown.

Each receptacle is provided with a head a^5 provided with three openings, one serving as a charging opening and provided with a closing means. In the present case such closing means consists of the manhole part a^6, as shown in Fig. 9, carried by the receptacle head a^5. Removably attached to a^6 is the closure part a^7 which, in conjunction with the plate a^8, the springs a^{18}, guide rods a^{19} and top plate a^{20}, also provides the relief gas valve for the receptacle. In order to charge the receptacle the bolts a^{21} holding the closure part a^7 to the manhole part a^6 must be removed and the part a^7 lifted our.

A second opening, in the head a^5, which is centrally arranged, serves as an inlet for hot gases, and is provided with a flange a^9 (Figs. 3 to 5) to which is attached the inlet gas pipe hereinafter explained. A third opening, in the head a^5, serves as an outlet and is provided with a flange a^{10} to which an outlet pipe may be connected as hereinafter explained.

The receptacle is provided with an inner wall suitably perforated and arranged to provide a suitable space for the passage of gas after escaping from the shale charge through the perforations of the inner wall, such space being in communication with the outlet opening. In the particular embodiment shown in the drawing, Figs. 1 and 6, the inner wall is in the form of a vertically corrugated perforated cylinder a^{11}, the corrugations forming vertical ducts around the charge of shale, which ducts terminate below the top of the receptacle. The gas escapes from the various ducts into the open space at the top of the receptacle and thence passes through the discharge opening. The center of the receptacle is provided with a downwardly extending perforated distributing pipe for heated gas, as indicated at a^{12}. This pipe extends downward from the inlet opening nearly to the bottom of the receptacle and has its lower end closed.

It will be seen that when a receptacle is filled with shale fragments, and heated gas

is forced into the central perforated pipe, the gas will escape from the pipe a^{12} and flow in a generally radial direction to and through the perforated inner wall a^{11} of the receptacle. Thence the gas will pass upward and out of the receptacle. In this way the shale will be heated in a substantially uniform manner, and owing to the relatively short and direct path of the gas through the shale it will not be necessary to use such high gas pressures for driving the hot gases through the shale, as has been necessary heretofore where the gases have had to travel long distances through the shale.

The heated gas is at such a temperature that it will cause a distillation of the shale with consequent evolution of gas and liquid constituents of the shale. The gas, as well as some of the vapors evolved, will pass out of the receptacle, and the mixture of gas and any accompanying vapors is led into the inlet pipe of another receptacle, thence from such other receptacle to still others, so that the gas passes in series through a number of receptacles such that the gas issuing from the last receptacle will be at a temperature below that at which gas is evolved from the shale. In the last receptacle most of the vapors carried over from the preceding shale charges will condense. The gas is led away from the last receptacle for further treatment to remove any remaining vapors, is then heated up, and returned to the first receptacle of a series to go through the series again.

Since the first receptacle of a series receives the hottest gases, the distilling operation will be completed in this first receptacle before it is finished in the following ones of the series. As soon as this occurs the first receptacle is cut out of the gas circuit, the second one of the series is connected up with the others so that it will be the first to receive the heated gas, and the former first one is recharged with fresh shale and connected in the series so that it will be the last of the series.

The apparatus shown in the drawings, is so constructed and arranged that it may be operated as one series or as two series of receptacles. In the latter case each series may comprise four receptacles. The two series are assumed to be in operation simultaneously. The first receptacle of each series receives heated gas from a supply pipe B, which is so arranged that it may be swung about a vertical axis at its center, so that the flanges on its downward extending ends may be attached to the respective flanges a^{13}, of the flanged T connectors a^{14}, of the inlet openings of the respective first receptacles. The supply pipe B receives the heated gas at its center through a vertical pipe C, Fig. 3, which receives the gas from a heater, hereinafter described, this vertical pipe being provided with a ground union joint, so that the pipe B and the attached part of the pipe C may be shifted angularly on the ground union joint.

To sustain the weight of the swinging pipe B, it may be provided with hangers, one of which is shown at b^1, Fig. 1, which has a roller running on a circular overhead track shown at b^2, this track being carried by brackets b^3 secured to legs b^4 extending upward from the tops of the receptacles. These legs serve to support a bin F, which is arranged to hold the shale, the bin being provided with a swinging spout f^1, arranged to swing into position to discharge into the charging opening of each receptacle. A suitable cut-off is provided for this spout as indicated by f^2.

As previously stated each inlet opening of each receptacle is provided with a T connector a^{14}. Similarly each outlet opening of each receptacle is provided with a T connector a^{15}. The receptacles are connected together in series through the T connectors a^{14} and a^{15}, by means of the connecting pipes G, provided with valves H as indicated in Figs. 2 and 5. By this means the outlet of one receptacle is connected to the inlet of an adjacent receptacle, and so on through the series of receptacles, making a complete cycle.

The last receptacle of each series discharges the gas through a discharge pipe I, which is so arranged that it may be swung about a vertical axis at its center, so that the flanges on its downward-extending ends may be attached to the respective flanges a^{16}, of the flanged T connectors a^{15}, of the outlet openings of the respective last receptacles. The discharge pipe I which discharges the gas to a series of separating tanks, a condenser, and filter, hereinafter described, is provided with a ground union joint J, so that it may be shifted angularly on the ground union joint.

The weight of the swinging pipe I, may be sustained by the connecting pipe K which may be supported at its ends by any suitable means, not shown. The outer end of the pipe K is connected to a spray pipe L, connected to the first separator or trap M as indicated in Fig. 1.

Each T connector a^{14} and a^{15} is provided with a closing plate a^{17} with the exception of two of the T connectors a^{14} which are connected to the two ends of the gas supply pipe B, and two of the T connectors a^{15} which are connected to the two ends of the gas discharge pipe I.

Means are provided for heating the gas used for distilling the shale, such means comprising the furnace formed by the supporting and enclosing walls for the gas heating coils N, supporting and enclosing walls

for the receptacles or retorts, the connecting flue to the stack O, burner P, gas inlet manifold Q, and gas outlet manifold R.

The heater coils N may be U-shaped, arranged and supported as shown in Figs. 1 and 6, one leg of each coil connecting with the outlet manifold R, which in turn is connected to the supply pipe B, and the other leg of each coil connecting with the inlet manifold Q, which in turn is connected to the inlet pipe q^1. The manifolds Q and R are supported by the furnace walls s^1, s^2, and s^3. The lower end of the circular wall s^1 is provided with holes to allow the passage of gases from the burner chamber within the wall s^1 to the coil chamber between the walls s^1 and s^2. The hot gases pass up around the heater coils to the top of the chamber, passing through holes in the top of the wall s^2 and thence downward around the heater coil through the chamber between the walls s^2 and s^3, and finally flowing out to the flue and stack O. It will be seen that the burner gases flow in the same direction as the gases used for distilling the shale which flow in the heater coils N. The gas supplied to the burner P preferably is that which is produced in the distilling apparatus, but other gas may be used either throughout the process or only on starting it.

In order that the gas issuing from the last receptacles of the two series through the discharge pipe I, may be reheated and returned to the first receptacles for distillation of shale, it must be passed through the proper apparatus which will remove any vapors carried over from the last receptacles. In the present invention this apparatus comprises the pipe L connected to the pipe K, and having in its upper end the sprayer l'. The pipe L is connected to the separating tank or trap M in such a manner that the gases flowing into M will flow circularly about an outlet pipe m^1, which is closed at its lower end, and perforated along its length within the tank M. A drain outlet m^2 is provided in M to drain off the condensed vapors which accumulate. The pipe m^1 connects with the separating tank T, through the pipe t^1 provided at its upper end with the sprayer t^2. Tank T is connected to the filter X by the perforated pipe t^3, pipe u^1, blower U, condenser V, tank W, and pipe w^1.

The filter X which contains suitable filtering material x^1 such, for example, as shavings, is provided with a perforated false bottom x^2 so that the incoming gas from w^1 may be distributed through the material x^1. The bottom of the filter is provided with a suitable outlet closed by a valve device such as indicated at x^3 having its valve stem operated by wheel x^4. By opening this valve device the accumulated condensed vapors may be drained off at intervals, as desired.

The outlet pipe from the top of the filter is connected to the supply pipe q^1, which is connected to the heater inlet manifold and controlled by the valve q^2, said outlet pipe being provided with a safety or relief valve x^5 and blow-off pipe x^6 which leads to a gas holder, not shown.

The general operation of the apparatus is as follows:

The shale or other carbonaceous material is charged into the two series of receptacles A^1 to A^8 by means of the bin F and swinging spout f^1, through the manholes a^6 in the top of each receptacle, each manhole being tightly closed after charging by the valved caps a^7. With the discharge pipe I and supply pipe B connecting the receptacles as shown in Fig. 2, A_1 and A_5 will be the first receptacles, which will receive the hottest gas direct from the heater, and consequently the distilling operation in A_1 and A_5 will take place first in these receptacles. The gases escaping from these receptacles will pass to the succeeding receptacles, gradually heating up their contents, while the gases themselves are gradually cooled and any accompanying vapors tend to condense. Finally the gases escape from the last receptacles through the pipe I.

When operating with the arrangement shown in Fig. 2, all valves H in the connecting pipes G should be open except those between A_4 and A_5, and also A_1 and A_8. This is necessary to prevent the gas flowing from one series to another without having to pass through the pipes B and I.

By starting the blower U the gas will be circulated in the system through the heater coils, in which the gas will be heated to its maximum temperature, thence out of the coils to the outlet manifold R, through the supply pipe B to the receptacles A_1 and A_5, thence from A^1 through A^2, A^3 and A^4 to the discharge pipe I, and similarly from A^5, through A^6, A^7 and A^8 to the discharge pipe I. The gas is led from I, through K and L where it encounters a spray of water or oil and passes into M, thence escapes through m^1 to t^1 where it is again sprayed with water or oil and then enters T whence it passes through t^3, u^1, V, W, w^1 to and through the filter X, finally returning to the heater coils through pipe q^1 and inlet manifold Q, to be reheated and passed on to the receptacles A_1 and A_5 to be passed through the series again.

As soon as distillation ceases in receptacles A_1 and A_5 the flow of gas is stopped, and the valves between the receptacles A_1 and A_2 of the first series, and those between A_5 and A_6 of the second series, are closed. The supply pipe B is disconnected from the

flanges of the T connectors a^{14} of receptacles A_1 and A_5 and swung clockwise, in the present case, and connected to the T connectors a^{14} on receptacles A_2 and A_6 after the cover plates a^{17} have been removed from the latter T connectors and fastened to the former T connectors. The flow of the hot gas is again started, and while the distilling operation is going on, the two receptacles A_1 and A_5 which are now cut out of the series, are discharged and charged with fresh shale fragments. The flow of gas is stopped, the discharge pipe I is disconnected from the flanges of the T connectors a^{15} of receptacles A_4 and A_8 and swung clockwise, in the present case, and connected to the T connectors a^{15} on the newly charged receptacles A_1 and A_5 after the cover plates a^{17} have been removed from the latter T connectors and fastened to the former T connectors. The valves between the receptacles A_1 and A_6, and also those between A_4 and A_5 are opened, and the flow of gas again started.

The receptacles A_2 and A_6 are now the first of the series and receive the hottest gas, while A_5 and A_1 are the last receptacles of the respective series. When distillation ceases in receptacles A_2 and A_6 the above operations are duplicated, and so on for the other receptacles of the two series.

The products obtained by this fractional distillation method flow to the bottom of the several receptacles and are carried off by pipes a^4.

While in the operation of the apparatus as above described, the plant is assumed to operate as a double series of retorts, it is clear that by providing a valve B^1 in one arm of the gas supply pipe B so as to shut off the supply at one side, and by providing a valve I^1 in one arm of the gas outlet pipe I, the retorts may all be operated in one series, the valve H being suitably opened for that purpose.

By this means it is possible to operate with as many receptacles in series as may be required to obtain the best results. In the apparatus shown from four to eight such receptacles may be worked in series.

A further important feature of my invention consists in subjecting the shale to be distilled to a hot gas of such composition that it will contain a material proportion of sulphur dioxide (SO_2) which enters into reaction with the unsaturated hydrocarbons evolved during the distilling process and thereby renders the said unsaturated hydrocarbons soluble in water so that they may be washed out in the water scrubber. Where the shales being operated upon themselves contain sulphur in such quantity that they will generate sufficient sulphur dioxide for the purpose some of the gas coming from one retort may be used in the succeeding retorts to accomplish the desired purpose.

In general, however, in order to obtain a sufficient amount of sulphur dioxide, it may be necessary to use an outside supply of sulphur dioxide, which is admitted to the gases in advance of the heater. This sulphur dioxide may be generated in any usual way but, as the residual carbonaceous material discharged from the retorts usually contains a large amount of sulphur, I have found it very advantageous to transfer such carbonaceous residues to a gas producer, where the residues may be distilled and blown in the usual way of operating gas producers, the blowing being done with air or steam, but most advantageously with steam. The resultant gaseous products contain a large amount of sulphur dioxide and these are passed through the retorts, to assist in heating the shales. The sulphur dioxide not only renders any unsaturated hydrocarbons soluble in water, but also breaks down any sulphuretted hydrogen (H_2S) which might otherwise contaminate the volatile products.

I therefore may provide means for passing such gases from the gas producer into the retort system, passing it through the superheater whereby the added gas helps to carry the heat into the retorts. When steam also is added to the system, it also is made to pass the superheater. By this means the oils distilled off from the shale are partly or wholly refined as they are produced.

At Z^4, Fig. 1, I have shown a pipe coming from a generator of sulphur dioxide (not shown) which may be the gas producer hereinbefore referred to. The pipe Z^4 is controlled by a suitable valve, as indicated at Z^5.

When much sulphur exists in the shale much of it may be recovered as a by-product.

What I claim is:

1. In a shale distilling plant the combination, with a receptacle for the shale charge, said receptacle having a perforated inner lining spaced from the outer wall of the receptacle to form an annular space for the escape of gas, and having a shale-charging opening and discharging opening for solid products, of means for closing said openings, means for conducting away gas from the annular space, means for heating said gas, a perforated pipe arranged to discharge said heated gas along the interior of the shale charge so that the gas may travel radially through the charge to the perforated lining, and means for drawing off the liquid products of distillation.

2. In a distilling plant, the combination with a plurality of receptacles arranged to receive the material to be treated, said receptacles being arranged around a center, of a central furnace chamber, means for heating the same, gas-heating coils arranged in said furnace, a central outlet pipe ar-

ranged to discharge gas from all of said gas-heating coils, a movable gas supply pipe connected to said central outlet pipe and arranged to be connected with any one of said receptacles, valved connections between the receptacles whereby heated gas may be passed from each receptacle to the next adjacent one in series, a central outlet pipe, a movable outlet pipe connected to said central outlet pipe and arranged to be connected to any one of the receptacles to receive escaping gases therefrom, means connected to said central outlet pipe for removing condensable vapors from the escaping gases, and means for driving the purified gases back to the gas heating coils and through the receptacles.

3. In a distilling plant, the combination with a plurality of receptacles arranged to receive the material to be treated, said receptacles being arranged around a center, of a central furnace chamber, means for heating the same, gas-heating coils arranged in said furnace, a central outlet pipe arranged to discharge gas from all of said gas-heating coils, a movable gas supply pipe connected to said central outlet pipe and arranged to be connected with any one of said receptacles, valved connections between the receptacles whereby heated gas may be passed from each receptacle to the next adjacent one in series, a central outlet pipe, a movable outlet pipe connected to said central outlet pipe and arranged to be connected to any one of the receptacles to receive escaping gases therefrom, means connected to said central outlet pipe for removing condensable vapors from the escaping gases, said means comprising sprayers for spraying the gases, and means for driving the purified gases back to the gas heating coils and through the receptacles.

4. In a distilling plant, the combination with a plurality of receptacles arranged to receive the material to be treated, said receptacles being arranged around a center, of a central furnace chamber, means for heating the same, gas-heating coils arranged in said furnace, a central outlet pipe arranged to discharge gas from all of said gas-heating coils, a movable gas supply pipe connected to said central outlet pipe and arranged to be connected with any one of said receptacles, valved connections between the receptacles whereby heated gas may be passed from each receptacle to the next adjacent one in series, a central outlet pipe, a movable outlet pipe connected to said central outlet pipe and arranged to be connected to any one of the receptacles to receive escaping gases therefrom, means connected to said central outlet pipe for removing condensable vapors from the escaping gases, said means comprising a filter device through which the gases are passed, and means for driving the purified gases back to the gas heating coils and through the receptacles.

5. In a distilling plant, the combination with a plurality of receptacles arranged to receive the material to be treated, said receptacles being arranged around a center, of a central furnace chamber, means for heating the same, gas-heating coils arranged in said furnace, a central outlet pipe arranged to discharge gas from all of said gas-heating coils, a movable gas supply pipe connected to said central outlet pipe and arranged to be connected with any one of said receptacles, valved connections between the receptacles whereby heated gas may be passed from each receptacle to the next adjacent one in series, a central outlet pipe, a movable outlet pipe connected to said central outlet pipe and arranged to be connected to any one of the receptacles to receive escaping gases therefrom, means for spraying the gases, a filter through which the gases pass in an upward direction, connections for conducting the gases from the central outlet pipe to the spraying means and thence to the filter, and means for returning the purified gases to the gas heating coils.

6. In a distilling plant, the combination, with a plurality of receptacles for material to be distilled, each provided with a gas inlet and a gas outlet, of a T-connection for each inlet, a T-connection for each outlet, a plurality of gas-conduits each connecting the outlet T-connection of one receptacle to the inlet T-connection of an adjacent receptacle, a movable gas supply pipe arranged to engage any one of the inlet T-connections of the receptacles, a movable gas outlet pipe arranged to engage any one of the outlet T-connections of the receptacles, and means for closing the inlet and outlet T-connections not engaged by the respective movable supply and outlet pipes.

In testimony whereof, I have hereunto set my hand.

ROBERT M. CATLIN.