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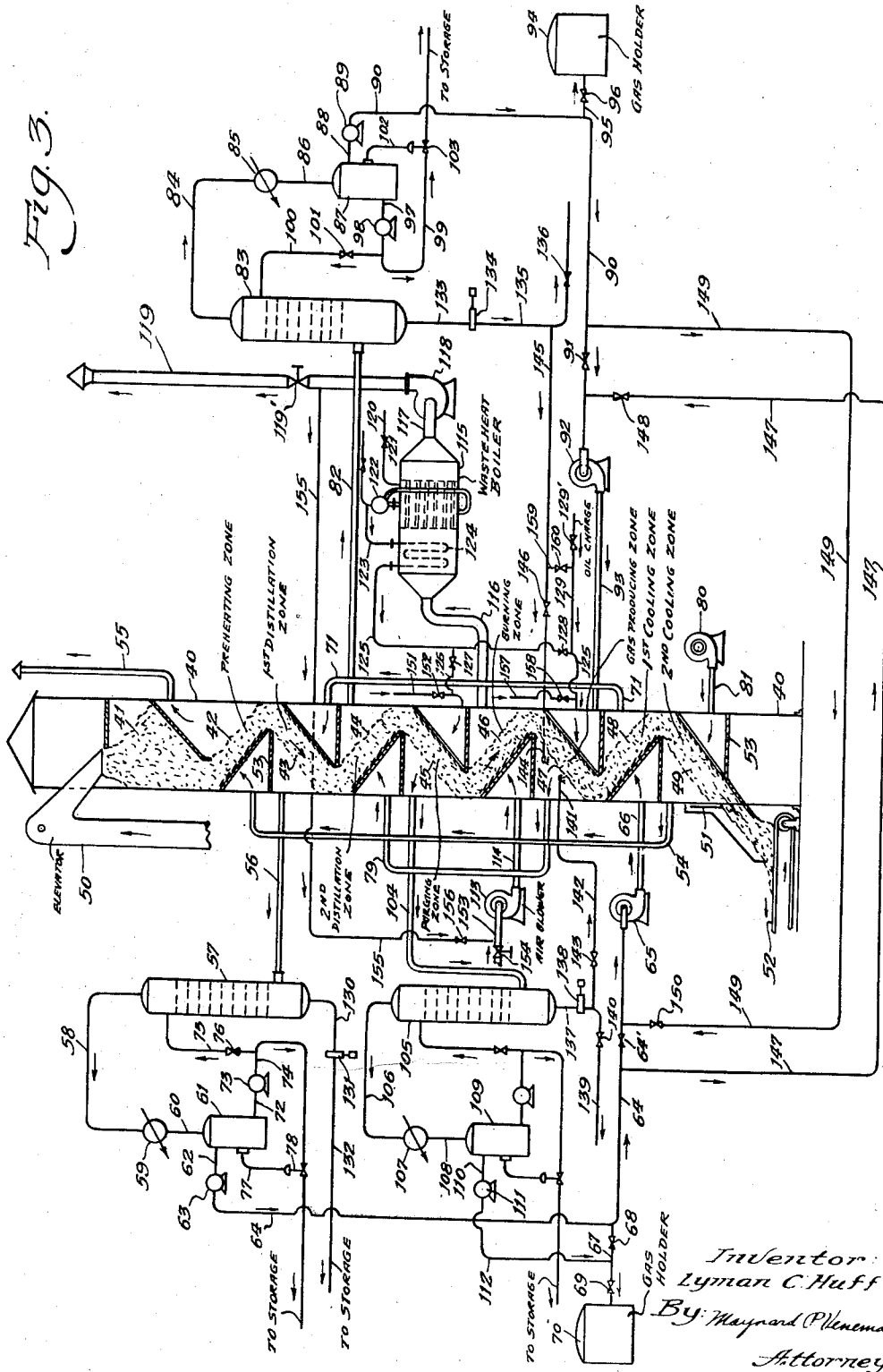
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DISTILLATION OF CARBONACEOUS SOLIDS

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DISTILLATION OF CARBONACEOUS SOLIDS

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This invention relates to an improved method and apparatus for distilling oil shales, tar sands, coal or other carbonaceous solids. More particularly the invention relates to an improved type of retort and means of direct contact between the bed of solids and heating mediums such that the apparatus will operate efficiently and in a flexible manner to pyrolytically convert the solids into hydrocarbon conversion products.

Broadly, my invention provides a unitary vertical retort having a series of contact zones arranged in vertical alignment whereby a moving bed of crushed hydrocarbonaceous solid material, such as oil shale, may continuously move by gravity from top to bottom through the various contact zones within the retort, with relatively all of the volatile materials being obtained from the solid material, and spent shale ash being removed from the lower end of the retort. In each of the zones, the bed of crushed oil shale, or other material, takes the form of a relatively shallow bed lying on a sloping grate at its angle of repose, such that the bed is of a uniform thickness in each of the contact zones throughout the retort from top to bottom. Partitioning members, grates, gas inlets and outlets are so arranged that the heating mediums and vapors will pass directly through each of the moving shallow beds in direct contact with the crushed solid material. One of the important advantages in the improved retort which makes possible the improved method of operation, is the provision for moving or oscillating the grates on which the shale bed reposes in each of the process zones such that the bed is agitated and prevented from coking and clinkering. Adjustment means is also provided for varying the slope of each grate and accommodating the moving grate to the angle of repose for the particular material within a given zone.

Oil shales are generally referred to as being of two different types, that is non-coking shales and coking shales. The latter type is difficult to process as it must be retorted in a manner to prevent clinkering of the entire mass as it is heated. However, a coking shale may be readily processed in the retort of this invention by reason of the continuously oscillating grates which keeps the material moving and agitated or broken up such that clinkering is substantially prevented.

It is an object of this invention to provide a method whereby large quantities of shale may be processed continuously in an economical manner.

Another object of the invention is to provide an apparatus and method operation which will accommodate itself to any type of crushed solid

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hydrocarbonaceous material, including oil shales of either the non-coking or coking types.

Still another object of the invention is to provide a retort having a plurality of contact zones or chambers in alignment, such that the solid material may proceed by gravity downwardly from one zone to the next, and is arranged so that distillation may be carried on first in a low temperature zone and then passed to zones of higher temperature for more complete distillation. The volatile products are thus removed from this improved system, without being subjected to highly destructive temperatures usually encountered in other methods and apparatus for processing oil shales.

An important advantage of this invention is that the shale bed is contacted directly while in a relatively uniform and shallow bed. It has been found that retorts employing indirect heating means are expensive to operate and that it is economically desirable to obtain direct contact between heating medium and the solids, such as provided by the apparatus and method of operation comprising this invention. A particular advantage has also been found in the arrangement of this new retort, whereby a plurality of contact zones are aligned one above the other and are substantially separated so that different contact operations are simultaneously and continuously carried out in each of the zones on a relatively uniform ribbon like moving bed of the hydrocarbonaceous material.

Other advantages will be apparent upon reference to the accompanying drawings and the following description thereof together with explanation of the processing operations which are employed in distilling the bed of hydrocarbonaceous solids.

Figure 1 of the drawing shows a sectional elevation view through one form of the improved distilling retort.

Figure 2 of the drawing shows an enlarged view through one of the movable and adjustable grates on which the crushed solids bed is supported.

Figure 3 of the drawing shows a diagrammatic view of a more elaborate form of the improved retort and accompanying apparatus and conduits employed in processing solid carbonaceous material to obtain valuable hydrocarbon conversion products therefrom.

Referring now to Figure 1 of the drawing, a vertical rectangular retort housing 1 is shown in cross-sectional elevation. The housing 1 is preferably made of steel or alloy construction

that is suitable to accommodate the temperature conditions prevailing throughout the height of the retort. A plurality of contact chambers are disposed vertically within the housing 1 such that a solid carbonaceous material passes successively from one to the other and from top to bottom. The various contact chambers or zones within the retort, from top to bottom may be identified as follows; a crushed shale hopper 2, a pre-heating zone 3, an initial distilling zone 4, a final distillation zone 5, a burning zone 6, a first cooling zone 7 and a final cooling zone 8. The crushed oil shale or other solid hydrocarbonaceous material is carried to the top of retort 1 and discharged into the hopper 2 by means of a conveyor or elevator 9. The shale then descends by gravity through each of the zones, being aided in its descent by oscillating grate 10. In the high temperature zones, such as the burning zone, a refractory lining 1' can be used to protect the interior of the housing 1.

Figure 2 of the drawing illustrates one form of the moving and adjustable supporting grate 10, which comprises primarily a steel frame and a series of tuyère plates 11. The tuyère plates 11 are mounted and arranged in an overlapping manner such that the descending bed of crushed solid material will not fall through the grate. However, the tuyères 11 are so constructed that gases and vapors will be equally distributed to the shale bed, through spaces 12, between each of the overlapping plates. The upper end of the frame of the grate 10 is pivoted on a bar 13, while at the lower end a rotating bar 14, having cams 15, will serve to move the end of the entire grate in a slight up and down movement. A slotted opening 16, in the side wall of the retort, having a cover plate and adjustment means not shown, makes it possible to vary the angle of the grate 10 to parallel the angle of repose of the shale bed in each of the contact chambers. It is evident, that by proper preliminary adjustment, the moving grate may be made to support the descending shale in a bed of relatively even thickness and thereby attain substantially uniform conversion throughout the entire length of the bed in each contact zone. The mechanical means and method of rotating the lower cam shaft 14 is not illustrated, as the means of rotation may be conventional and may be provided in any desired manner. It is however, within the scope of this invention to have varying sizes and types of cams and to thereby vary the frequency and the amount of movement or agitation of the bed in any one of the contact zones. Baffle members 17 are mounted in front of the various gas inlets, to each of the contact zones, and are found desirable to prevent direct channeling of the contact medium through a confined portion of the grate and the supported shale bed.

Referring again to Figure 1 of the drawing, the descending shale bed is contacted by hot air, within the pre-heating zone 3, with the heated air stream being admitted by way of conduit 18 and discharged through outlet conduit or stack 19. The shale is preferably preheated within this contact zone 3 to a temperature of the order of 500° F. However, relatively little volatile matter is removed at this temperature and the gases are normally allowed to be discharged to the atmosphere by way of the stack 19. A heated recycled stream of a portion of the uncondensed conversion product is employed, with or without an auxiliary supply of

high temperature super-heated steam, to effect a first distillation in zone 4. The recycled fluid medium enters by way of conduit 20, while steam is charged by way of line 21 having control valve 22, and the distillation products are carried by way of conduit 23 to suitable separating and fractionating equipment, not shown.

The moving bed of crushed material, following the first distillation in zone 4, is subjected to a higher temperature and final distillation within zone 5, the heating medium therein being hot combustion gases received directly from the burning zone 6. Air is blown to zone 6 by way of a blower 24 and conduit 25 such that relatively complete removal of all volatile or combustible material is effected therein. Combustion gases at a temperature of approximately 1500° F. are passed from the shale bed of burning zone 6 to the final distillation zone 5 and are discharged from the retort by way of outlet conduit 26. The combustion gases thus carry with them the distillation and conversion products from the pyrolytic contact within zone 5, and the combined conversion products are passed by way of conduit 26 to suitable separating and fractionating equipment, not shown. A first cooling operation for the hot spent shale is effected in zone 7 wherein a recycled stream of uncondensed conversion products from the separating step are charged by way of line 27 having control valve 28. In addition an auxiliary supply of oxygen containing gas, such as steam may be charged by way of line 29 having valve 30. The heated recycle gas stream leaves this first cooling zone 7 at a temperature of approximately 1300° F. and passes by way of conduit 20 to the initial distillation zone 4, to effect a first distillation, as previously noted in the description. In the final cooling zone 8, air from blower 31 and the inlet conduit 32 is passed through the spent shale bed prior to the discharge of the latter from the retort 1 through outlet gate 33. The air stream thus used to cool the spent shale, absorbs heat therefrom and is discharged from zone 8 at a temperature approximating 600° F. being carried by way of conduit 18 to the pre-heating zone 3, where as previously noted the heated air from line 18 is used to bring the incoming shale bed up to an initial temperature of 500°-550° F. The spent shale from the lower end of the retort 1 is passed on to a conveyor 34, in order that it may be continuously carried away from the retort and disposed of.

In a retort of the type disclosed and by means of the improved method of contact and operation which has been described, large quantities of oil shale may be continuously processed. For example, with a 10 ft. by 20 ft. rectangular retort, and a rate of movement through the retort being adjusted so that the shale bed is contacted for approximately one hour in each zone, then some 300 tons per day of shale may be subjected to complete conversion. In order to attain optimum operating conditions, the shale or carbonaceous material should be crushed to a size of approximately $\frac{1}{2}$ to $1\frac{1}{2}$ inches in size. Shale larger than $1\frac{1}{2}$ inches will require a longer period for the heat to penetrate to the center of the lumps or pieces and the capacity of the retort will have to be slowed down accordingly.

Referring now to Figure 3 of the drawing a more elaborate form of retort is shown in combination with accompanying process equipment employed to obtain both hydrocarbon distillates and producer gas from shale or other solid carbonaceous material. A plurality of contact zones

are spaced in vertical alignment within the retort 40 and from top to bottom may be identified as follows; a hopper 41, a pre-heating zone 42, a first distillation zone 43, a second distillation zone 44, a purging zone 45, a burning zone 46, a gas producing zone 47, a first cooling zone 48 and a second cooling zone 49.

Crushed material is lifted to the top of the retort 40 by means of a conveyor or elevator 50 and is then dumped into the hopper 41. The shale moves downwardly in a relatively uniform bed through each of the contact zones over moving grates 10, which may be constructed and operated as noted in connection with Figure 2 of the drawing. Shale ash is continuously removed from the lower end of the retort through gate 51 to a belt conveyor 52 which carries the spent material to a disposal zone, not shown. Each of the vertically aligned zones are separated one from another by partition plates 53 which may be constructed of steel or alloy suitable for the temperature conditions encountered.

Air is blown through the bed of spent shale in a second cooling zone 49 by means of air blower 80 and conduit 81 in order to effect a final cooling of the spent material prior to its discharge from the retort and to provide heated air, at approximately 600° F., which is channeled by way of conduit 54 to the upper pre-heating zone 42. The shale moving through zone 42 is thus heated to a temperature of approximately 500° F. At this temperature, little or no volatile matter is removed from the solids and the heating medium may be discharged through the outlet conduit or vent stack 55. The pre-heated shale bed then descends to the first distillation zone 43, wherein a hot fluid medium, comprising a recycled uncondensed fraction of the conversion products, is used to effect the removal of the more volatile products within this zone at a temperature of the order of 700° F. The conversion products from this first distillation zone 43 are passed by way of line 56 to a fractionator 57. The light fractions from fractionator 57 are passed by way of line 58 through a suitable condenser 59 and line 60 to a receiver tank 61. The uncondensed fractions from receiver 61 are withdrawn through line 62 by exhaustor pump 63 and passed to line 64 having a valve 64', being recycled to the retort by way of gas blower 65 and conduit 66. Any excess quantities of uncondensed gases may be passed by way of line 67, having control valve 68 and 69, to a gas holding vessel 70. The recycled gas passing to zone 48 of the retort effects a first cooling of the oxidized shale bed and thereby acquires a temperature of approximately 1000° F. The heated recycled stream is then passed from zone 48 by way of conduit 71 to the first distillation zone 43, as previously noted. The condensed hydrocarbon fraction collected within the receiver 61 may be passed to storage or to further treating equipment by way of line 72, pump 73 and line 74. A portion of this condensed fraction may be used for recycle to the fractionator 57, passing by way of line 75 having valve 76. The quantity of condensed fraction being removed from receiver 61 will be controlled by a liquid level controller 77 and automatic control valve 78 in line 74.

The shale bed, following the first distillation in contact zone 43, passes to a second distillation zone 44 wherein more complete distillation is effected by a hot stream of producer gas entering by way of conduit 79 from the lower gas producing zone 47. Conversion products from zone 75

44 are passed by way of line 82 to a fractionating column 83 for separation into light gaseous fractions and heavy liquid fractions. The light hydrocarbon fractions are passed by way of line 84 to a condenser 85 and therefrom by line 86 to a receiver 87. The uncondenser gas fractions from this conversion step are withdrawn from receiver 87 by way of line 88, exhaustor pump 89, and may be passed through line 90 having a valve 91 to gas storage or it may be recycled to zone 47 of the retort 40. The gas blower 92 receives the uncondensed recycled gas stream and discharges it by way of line 93 to the gas producing zone 47. The recycled gas stream together with an oxygen containing gas, such as steam and air, forms producer gas which will be at a temperature of approximately 1350° F. The producer gas stream is passed by way of conduit 79 to the second distillation zone 44 to effect a high temperature conversion therein as has been previously noted. Excess quantities of uncondensed gases from the second stage distillation and fractionation step may be withdrawn from the line 90 to a gas holder 94 by way of line 95 having valve 96. The condensed fractions from the receiver 87 may also be passed to storage or to further treating equipment by way of line 97, pump 98 and line 99, although a portion of the condensed fraction may be recycled to the fractionator 83 by way of line 100 having valve 101. The rate of flow to storage, through line 99, will be controlled by a liquid level controller 102 and automatic control valve 103.

The descending shale bed having substantially all of its volatile material removed in the second distillation zone 44 is then passed to the purging zone 45 wherein it is purged with a relatively inert gas, flue gas, or preferably superheated steam, to effect the further removal of any remaining volatile matter as well as drive off any gaseous materials that have formed during the distillation operation. The purging medium is charged by way of line 151 having valve 152 to the zone 45. The products from the purging zone are passed by way of line 104 to fractionator 105 to be suitably separated and fractionated. The light fractions from fractionator 105 are passed by way of line 106, condenser 107 and line 108 to a receiver tank 109. The uncondensed gases from receiver 109 are passed to the gas holding tank 70 by way of line 110, exhaustor pump 111, line 112 and line 67. In an alternate method of operation, a portion of the uncondensed gases from receiver 109 may be passed by way of line 112 and line 67 to line 64 such that the gases will be recycled to the retort by blower 65.

Following the purging operation in zone 45, the shale bed passes to the burning zone 46 wherein the bed is subjected to relatively complete oxidation and thereby increased in temperature to approximately 1550° F. Air is charged to zone 46 by way of air intake line 153 having control valve 154, air blower 113 and conduit 114 such that substantially complete oxidation of all carbonaceous and volatile matter is accomplished as the shale passes therethrough. To provide a control for the amount of oxygen and extent of burning in the zone 46, a flue gas stream is mixed with the air stream. This flue gas stream is recycled to the retort by way of line 155 having control valve 156. The resulting combustion or flue gases from zone 46 are withdrawn through a waste heat boiler 115, by way of conduit 116, and are discharged therefrom through conduit 117,

gas blower 118 and vent stack 119. The stack 119 has a valve 119' which may be adjusted, to permit a portion of the flue gas to pass to the burning zone 46, in admixture with air entering by way of conduit 114. Steam is generated within the waste heat boiler 115, feed water being charged by way of line 120 having valve 121. The steam generated in the waste heat boiler is collected in a steam drum 122 and withdrawn therefrom through line 123, passing through a steam pre-heating coil 124 within the inlet end of the waste heat boiler 115. Superheated steam from coil 124 passes to line 125 and then to both the purging zone 45 and the gas producing zone 47, the superheated steam being passed to the first mentioned zone by way of line 126 having valve 127 and to the latter zone by line 125 having valve 128. When the oxidation in zone 46 has been only partial, the partially burned or oxidized shale passes from zone 46 to the gas producing zone 47 at a sufficiently high temperature, approximately 1550° F., such that water-gas is produced by contact with superheated steam and air which is charged thereto by line 125. Air is received and passed by way of line 157, having control valve 158, to the line 125 and to the gas producing zone 48. Producer gas may thus be produced in large quantities having a high B. t. u. value. In an alternate operation, within the gas producing zone, oil may be introduced with the superheated steam to produce a much higher B. t. u. producer gas. Oil may be charged from an external source through line 129 having valve 129' to combine with the steam within line 125, or it may be received as a condensed fraction from fractionator 83, by way of connecting line 159 having valve 160.

It is also within the scope of the invention to recover selected normally liquid fractions formed in the fractionators 57, 83 and 105 as final products of the process. Line 130 having pump 131 may be employed to withdraw the liquid fraction from fractionator 57 and pass the recovered material by way of line 132 to a suitable storage tank. The liquid fraction from fractionator 83 may be withdrawn by way of line 133, pump 134 and line 135 having valve 136 to further treating equipment or to storage. Likewise, the liquid fraction from fractionator 105 may be withdrawn through line 137, pump 138, and line 139 having valve 140 to further treating or storage equipment.

As a means of further increasing the B. t. u. value of the producer gas in zone 47 a portion of the normally liquid fraction from fractionator 105 may be passed to a spray nozzle 141 above the bed of solids, within zone 47, by way of line 142 having control valve 143. In like manner, a portion of the liquid fraction from fractionator 83 may be passed to a spray nozzle 144 within zone 47 by way of line 145, having valve 146, and line 135 connecting with the lower end of the fractionator.

In order to provide greater flexibility in the operation of the unit a line 147 having valve 148 is provided so that a portion of the recycled uncondensed gas from the first distillation zone may be passed either alone or in combination with uncondensed gas from the second distillation zone, to the gas producing zone 47. Also, line 149 having valve 150 is provided to connect between line 90 and line 64 such that uncondensed gas from the second distillation zone may be passed, either alone or in combination with uncondensed gas from the first distillation zone,

to the first cooling zone 48 within the retort 40.

Pressure control and pressure indicating devices are not illustrated in the drawings, however, such devices are desirable and normally may be connected with each of the contact chambers, such that the proper operating pressures and gas flows may be maintained throughout the retort.

It may be noted from the above description that this process provides a convenient and economical method for continuously processing crushed oil shales or other types of carbonaceous solids in successively higher temperature conversion zones, without involving destructive distillation of the material. Also, an excess of heat will be available from the improved process and apparatus which can be used to generate steam or electrical energy.

While the term oil shale has been used in much of the above description, it should be understood that the use of this type of retort is not limited to any one material, but is adaptable for use with any crushed hydrocarbonaceous material.

I claim as my invention:

1. A method for producing valuable hydrocarbon liquids and gases from hydrocarbonaceous solids which comprises maintaining an agitated and descending bed of said solids in continuous travel through a series of contact zones within a retort, contacting said solids bed with a heated gaseous medium in a pre-heating zone, passing said bed to a first distillation zone and contacting with a second heated gaseous medium to effect distillation of vaporous and gaseous products therefrom, passing said bed to a second distillation zone and contacting therein with a third heated gaseous medium to effect a higher temperature distillation of vaporous products therefrom, passing said bed to a burning zone and contacting therein with a gas containing free oxygen to effect oxidation of combustible material therein, passing the hot oxidized solids bed to a hot gas producing zone and contacting said solids bed therein with said third gaseous medium prior to passing it with absorbed heat to said second distillation zone, passing said solids bed to a cooling zone and contacting said bed therein with said second mentioned gaseous medium prior to passing it with absorbed heat to said first distillation zone, passing said solids bed to a final cooling zone and contacting said bed therein with the first mentioned gaseous medium prior to passing it with absorbed heat to said pre-heating zone.

2. A method for producing valuable hydrocarbon liquids and gases from hydrocarbonaceous solids which comprises maintaining an agitated and descending bed of said solids in continuous travel through a series of separate superimposed contact zones, independently and adjustably agitating said bed in each of said zones, contacting said solids bed with a heated gaseous medium in a first distillation zone to effect partial distillation of vaporous products therefrom, subjecting said vaporous products to condensation and separation, contacting said solids bed with a second heated gaseous medium in a second distillation zone to effect a higher temperature distillation and removal of vaporous products therefrom, subjecting said vaporous and gaseous products from said second distillation zone to condensation and separation, passing said solids bed to a burning zone and contacting said bed

therein with a gas containing free oxygen to effect oxidation of combustibles therein, passing the hot solids bed from said burning zone to a cooling zone, returning regulated portions of uncondensed fractions from first said condensation and separation step to contact said solids bed in said cooling zone, passing said recycled uncondensed gas stream with absorbed heat to said second distillation zone as said second heated gaseous medium, returning regulated portions of uncondensed fractions from said second condensation and separation step to contact said solids bed in a second cooling zone and passing the last said recycled uncondensed gas stream with absorbed heat from said second cooling zone to said first distillation zone as the first mentioned heated gaseous medium.

3. A method for producing valuable hydrocarbon liquids and gases from hydrocarbonaceous solids which comprises maintaining an agitated and descending bed of said solids in continuous travel through a series of contact zones within a retort, contacting said solids bed in a pre-heating zone with a heated air stream, passing said solids bed from said pre-heating zone to a first distillation zone and contacting said bed therein with a hot gaseous medium to effect a partial distillation of vaporous products, passing said solids bed to a second distillation zone and contacting said bed with hot combustion gases to effect a more complete distillation, passing said solids bed from the second distillation zone to a burning zone and oxidizing said bed therein to effect the removal of residual combustibles, passing combustion gases from said burning zone directly to said second distillation zone, removing vaporous and gaseous products from each of said distillation zones and subjecting said products to condensation and separation, passing said solids bed from said burning zone to a first cooling zone, returning a regulated portion of uncondensed gases from said condensation and separation step to said first cooling zone and therein contacting said solids bed, passing the resulting gas stream from said first cooling zone with absorbed heat therein to first said distillation zone, passing said solids bed from the first cooling zone to a second cooling zone, contacting said solids bed in said second cooling zone with an air stream and passing said air with absorbed heat to said pre-heating zone.

4. A method for producing valuable hydrocarbon liquids and gases from hydrocarbonaceous solids which comprises maintaining an agitated and descending bed of said solids in continuous movement through a series of contact zones within a retort, contacting said solids bed in a pre-heating zone with a heated air stream, passing said solids bed to a first distillation zone and contacting therein with a hot gaseous medium to effect a partial distillation of vaporous products, passing said solids bed to a second distillation zone and therein contacting said bed with a second hot gaseous medium to effect a more complete distillation, passing said solids bed to a purging zone and therein contacting said bed with a third gaseous stream to effect removal of volatile and gaseous products, subjecting vaporous and gaseous products from said distilla-

tion zones and said purging zone to condensation and separation, passing said solids bed to a burning zone and therein oxidizing said bed to effect the removal of combustibles, passing the oxidized solids from said burning zone to a gas producing zone, returning a regulated portion of uncondensed gases from the second distillation zone to said gas producing zone and passing the resulting hot gases to said second distillation zone as said second gaseous medium, passing said solids from the gas producing zone to a first cooling zone and contacting said solids therein with a regulated portion of uncondensed gases separated from a first distillation zone and passing the resulting hot gases to said first distillation zone as said gaseous medium, passing solids from said first cooling zone to a second cooling zone and contacting said solids therein with an air stream prior to discharging said solids from the retort and passing the resulting hot air stream from said second cooling zone to the pre-heating zone.

5. A retort for recovering valuable products from crushed hydrocarbonaceous solids which comprises an elongated vertical housing, partitioning means dividing the housing into a series of at least six contact chambers aligned therein, an inlet for charging crushed solids to the upper end of said retort, an outlet for discharging ash from the lower end of said retort, a fluid inlet and a fluid outlet to each of said contact chambers, a conduit connecting the fluid outlet of the lowest contact chamber with the inlet to the upper contact chamber, a conduit connecting the next to the lowest contact chamber with the next to the upper contact chamber and a conduit connecting the third from lower contact chamber with the third from the upper contact chamber whereby heated fluid mediums may be passed from the lower chambers to the upper chambers.

6. The retort of claim 5 further characterized in that said chambers are provided with sloping grates which are adjustable as to their angle of slope.

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