

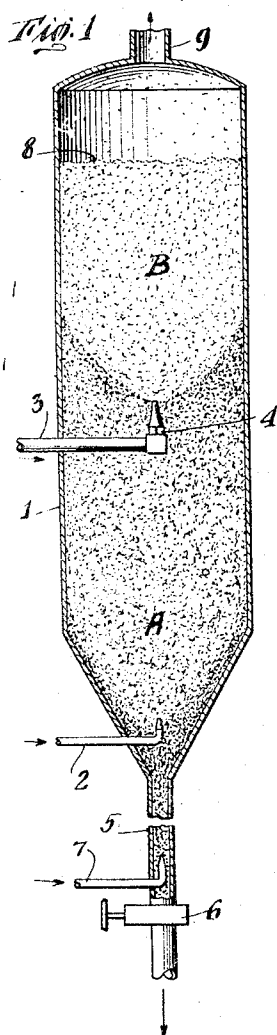
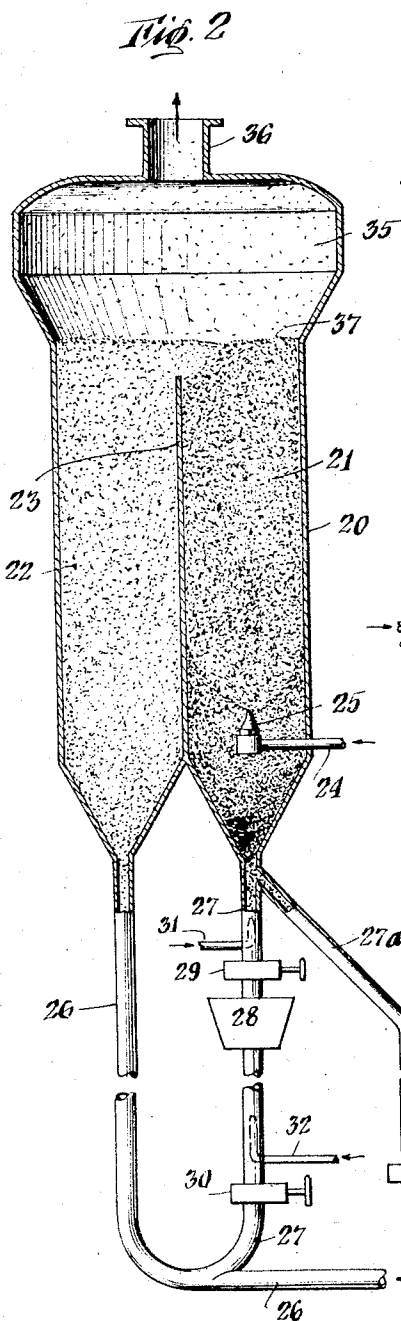
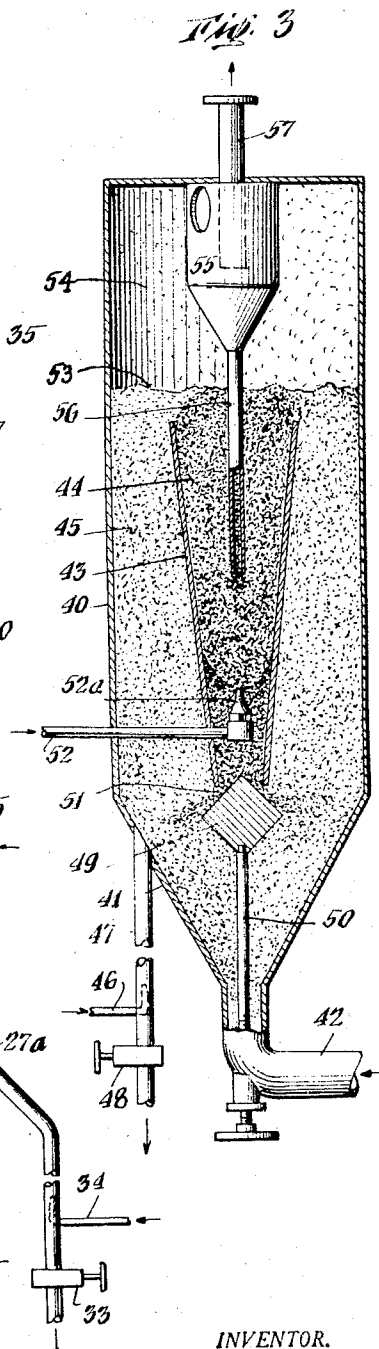
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CONVERSION PROCESS FOR HEAVY HYDROCARBONS

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CONVERSION PROCESS FOR HEAVY HYDROCARBONS

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The present invention relates to an improved process for the conversion of heavy hydrocarbons such as topped petroleum crude oils. More particularly, my invention relates to the treatment of heavy hydrocarbon oils in the presence of finely divided coke which is formed in the process and which is partially burned to supply a substantial portion of the heat required for treating the oil charge.

A principal object of this invention is to provide a simple and inexpensive method for the continuous conversion of heavy hydrocarbons to useful gaseous, liquid and solid products.

Another important object is to conduct the conversion of heavy hydrocarbons under conditions favoring the formation of more of the valuable products, i. e., liquids and gases, and less of solid coke.

A further object of the invention is the utilization of coke produced in the pyrolytic treatment of heavy hydrocarbons for the purposes of providing the heat of pyrolysis and of generating gas rich in carbon oxides.

Additional objects will become apparent hereinafter.

In a broad aspect, my invention pertains to a process which comprises subjecting a topped petroleum crude oil or like heavy hydrocarbon fraction to cracking temperatures, the heat required for this treatment being generated to a substantial extent by the combustion of some of the coke formed in the process. More specifically, the process of my invention comprises introducing a heavy hydrocarbon oil into a reactor containing a fluidized mass of hot, powdered coke to effect the conversion of the oil to gaseous and liquid hydrocarbons as well as additional coke, and simultaneously burning some of the coke with oxygen under fluidizing conditions in the same reactor to maintain the coke mass at an elevated temperature suitable for the conversion of the oil to products of greater value. The reactor employed in my process has two zones: a conversion or coking zone and a burning or heating zone, which are contiguous and yet so disposed with respect to each other that very little, if any, of the hydrocarbons produced in the conversion zone are consumed in the burning zone. Stated in another way, oxygen is introduced into the reactor at such a place that the oxygen is substantially completely consumed in the combustion of coke to carbon monoxide and dioxide before it reaches the zone in which the heavy hydrocarbon oil feed is pyrolyzed to products of lower molecular

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weight. Typical arrangements by which these results may be attained appear hereinafter.

My invention makes it possible to mix the products coming from the conversion and combustion zones so that the mixture may be processed as a unit for the recovery and separation of desired gaseous, liquid and solid products. In the process of my invention, the oxygen supplied to the combustion zone is of at least about 80% purity. For example, the oxygen may be a product of an air liquefaction and rectification process but the product should contain not more than about 20% by volume of nitrogen and other rare gases of the atmosphere. Preferably, I use oxygen of at least about 95% purity. While the oxygen may be derived from any known source, such as electrolytic processes, from the economic point of view, I generally select an oxygen fraction produced by the liquefaction and rectification of air along the lines of the processes disclosed in the patents to Frankl and Linde.

From the brief description of my invention hereinabove set forth, it can readily be seen that my process for the continuous conversion of heavy oils is distinguished by such features as: (1) curtailment of external heating and its attendant difficulties, e. g., coke deposition in heating coils, by generating within the coking vessel itself a substantial portion of the heat required for converting the oil; (2) utilization of the fluidization technique which obviates mechanical conveying equipment such as is usually employed in coking operations; and (3) simplification of equipment and operation while achieving high thermal efficiency. These and other features of the present invention will be set forth in greater detail in the further description of the invention which follows.

It has been common practice to subject a topped or reduced crude oil to thermal treatment to convert the oil to coke and volatile products by heating the oil, usually under pressure, and discharging the hot oil into a chamber or reactor wherein the desired conversion takes place. Conventionally, the heavy oil is passed through externally fired coils or the like in order to raise it to the desired conversion temperature. In contrast to such a method, I employ a fluidized process wherein a substantial portion or even all of the heat required for the operation is generated within the coking vessel itself and distributed very rapidly and thoroughly so that a uniformly high temperature is maintained throughout the conversion zone. At the same time, the gas resulting from the generation of

heat is a valuable raw material for chemical processes like the Fischer synthesis. Direct or internal heating as practiced in accordance with my invention makes the conversion operation highly flexible. Thus, a salient advantage of my process is that the hydrocarbon material charged into the reactor may or may not be preheated. If preheated, the heating may be limited so as to avoid any significant decomposition of the material and deposition of carbon while the material is passing through the heating pipe coil or like preheater; the remainder of the heat necessary for the conversion operation is then supplied within my reactor by internal combustion. Generally, heavy hydrocarbons can be heated to temperatures as high as 400° F. and even 600° F. without decomposition and carbon deposition within the preheater.

The technique of conducting reactions by the passage of reactant gases or vapors through a bed of powdered solid material at such velocities that the powder becomes suspended in the gas but exhibits what has been termed "hindered settling" so that a relatively small proportion of the powder is carried out of the reactor, has in recent years been developed rapidly and extensively for petroleum processing. This technique of fluidization, as it is more commonly called, which keeps a mass of powdered material in a fluid, agitated state resembling a boiling liquid and which permits the establishment of a pseudo-liquid level between the fluidized mass and the gas space above it, is the basis of the "fluid catalyst" process that has attained prominence in the manufacture of aviation gasoline. By incorporating the fluidization technique in the process of my invention, I have made the continuous conversion of heavy hydrocarbons simple and commercially attractive. Furthermore, fluidization both ensures the automatic flow of powdered coke between the conversion and burning zones of my unitary reactor and permits of much closer temperature control than has been heretofore possible with any known coking process.

The accompanying drawings are vertical sectional diagrams illustrating three forms of reactor suitable for carrying out the process of my invention and reference thereto will serve to clarify further the invention. These examples are not to be interpreted in a restrictive sense.

Figure 1 illustrates a reaction vessel 1 wherein the conversion and combustion zones are so disposed that the former is situated above the latter, in open communication with each other. Oxygen is admitted through pipe 2 at the bottom of reactor 1 into the lower or combustion zone A of reactor 1 wherein it promotes combustion of coke formed in the process.

The hydrocarbon charge, e. g., a topped crude oil, is introduced through pipe 3 into the upper or conversion zone B of reactor 1, preferably through an atomizing nozzle 4, so that it comes into contact with the heated mass of powdered coke. In the intimate contact resulting from the dispersion of oil in the fluidized mass of hot coke, the heavy hydrocarbons are pyrolyzed to more volatile products and additional coke. The volatile products flow upwardly through the coke mass and thus aid fluidization. Similarly in lower zone A, the oxygen entering by way of pipe 2 supports combustion of the coke, forming carbon monoxide and dioxide. The carbon oxides as well as any other constituents present in the oxygen used, e. g., nitrogen, flow up through reactor 1. The combustion zone A is made of suffi-

cient depth so that all of the oxygen is substantially completely consumed before it passes up through the upper conversion zone B. The gases from combustion zone A become uniformly mixed with the volatile hydrocarbons and hydrogen produced in conversion zone B. Being inert, the gases of combustion fill two useful roles, viz: (1) They act as a stripping medium to remove absorbed hydrocarbons from the coke as it moves from the conversion zone to the burning zone, and (2) they reduce the partial pressures of the hydrocarbons in the conversion zone, a favorable influence on the pyrolysis reaction.

In general, the oxygen is supplied to the system at such a rate that the gas flowing up through the combustion zone has an average linear velocity of about 0.1 to 3.0 feet per second, preferably about 0.5 to 1.5 feet per second, and generates sufficient heat to maintain a temperature of not less than about 800° F., preferably a temperature in the range of about 850° to 1000° F., within the conversion or coking zone. The flow of the oil sprayed or atomized into the upper conversion zone is correlated with that of the oxygen so that the desired temperatures and gas velocities are obtained in the reactor. For a reactor of uniform horizontal cross-section, it is obvious that the gas velocity in the upper zone will be somewhat higher than in the lower combustion zone because of the cumulative flow of combustion gases and hydrocarbon vapors through the upper zone. If desired, a uniform gas velocity through the whole reactor may be attained by making the reactor of larger horizontal cross-section in the upper zone than in the lower zone.

Coke formed in the process in excess of that required to supply the heat of reaction is withdrawn through standpipe 5 and slide valve 6. The product coke is in a finely divided state which greatly facilitates handling and enhances its utility for a variety of commercial applications. It is suitable for fuel purposes (as such or briquetted), for metallurgical reduction, for the manufacture of electrodes, etc. To keep the solids in standpipe 5 fluid and to prevent clogging, a gas such as steam or carbon dioxide (recycled from the operation or obtained from an outside source) is introduced through pipe 7 above slide valve 6.

While the reaction gases become disengaged from the bulk of the mass of fluidized coke at pseudo-liquid level 8, any coke which remains entrained leaves the reactor with the effluent gases by way of pipe 9. These gases usually pass through a separating device, such as a cyclone separator or electrical precipitator, wherein entrained coke is removed and thence either returned to reactor 1 or sent to coke product storage. The reaction gases free of solids flow from the separating device to a conventional system for the recovery and separation of liquid and gaseous products.

Another type of apparatus suitable for conducting the process of my invention is illustrated in Figure 2 which represents a vessel 20 in which two contiguous zones 21 and 22 are separated by a baffle 23. A reduced crude charging stock is introduced into pyrolysis zone 21 through pipe 24 and an atomizing nozzle 25. Oxygen is supplied through line 26 to zone 22 wherein it sustains combustion of the coke produced in the process. The heat of combustion is made to satisfy the thermal requirements for converting the reduced crude. Powdered coke produced during the re-

action moves through the tapered bottom of zone 21 into standpipe 27 which discharges coke into pipe 26 so that the coke becomes suspended in the gas stream flowing through pipe 26 and thus is carried into combustion zone 22. If desired, a grinder 28 may be inserted in line 27 to reduce oversize coke particles to sizes in the range determined by test as being desirable for good fluidization. Slide valves 29 and 30, or their equivalent, e. g., star feeders or rotary bucket-type valves, control the flow of coke particles to the grinder 28 and recycle line 26, respectively. Tubes 31 and 32 are used to introduce small streams of fluidizing gas, e. g., steam, to prevent clogging in standpipe 27 above slide valves 29 and 30, respectively. Branch pipe 27a provides an outlet for discharging coke that is formed in excess of the thermal requirements of the process. Slide valve 33 regulates the withdrawal of powdered coke, while a fluidizing gas entering through tube 34 serves to prevent packing of the coke in pipe 27a. When employing this type of apparatus, the two zones which are filled with fluidized coke are maintained at different fluid-static heads. As used in the present specification, the term, fluid-static head, denotes a pressure condition which is comparable to a hydrostatic head in a liquid system. The fluid-static head is readily altered by varying the velocity of the fluidizing gas. Accordingly, different gas velocities may be used in the two zones of my reactor to create a difference in fluid-static heads. Because of this difference in heads, a continuous cyclic flow of the comminuted solid is maintained automatically. In the present example, by maintaining a relatively low gas velocity in zone 21 the fluid mass therein is made to exert a greater fluid-static head than in zone 22. Under the difference in fluid-static heads, the coke particles flow down zone 21 through lines 27 and 26, up zone 22 and over baffle 23 in a cyclic fashion, thus conveying from combustion zone 22 to conversion zone 21 the heat required for the coking of the reduced crude charge. The reaction gases from zones 21 and 22 are commingled in upper space 35 and leave the system through pipe 36. While the reaction gases become disengaged from the bulk of fluidized coke at the pseudo-liquid level 37, coke particles entrained in the gases may be removed by filters or separators commonly employed for handling dust-laden gases. As shown, it is often advisable to make the upper space 35 of larger horizontal cross-section than that of the rest of the reactor so as to reduce gas velocities and thus promote settling of entrained particles. The liquid hydrocarbon products together with fixed gases, such as methane, ethane, carbon dioxide, carbon monoxide and hydrogen, flow to a recovery plant which effects separation into desired products.

Operating conditions employed with this type of apparatus are similar to those given for the system of Figure 1.

Still another form of reactor which is suitable for conducting the present process is illustrated in Figure 3. The reactor comprises an upright cylindrical vessel 40 with a tapered bottom 41 to which pipe 42 is attached for the admission of oxygen. Within vessel 40, a frusto-conical baffle 43 separates a central zone 44 containing a relatively dense but fluidized mass of powdered coke within which a heavy oil is undergoing conversion from an annular zone 45 containing in a relatively less dense condition a fluidized mass of coke which is being partially burned to supply heat

for the pyrolytic conversion of the heavy hydrocarbon charging stock.

Oxygen or steam is also introduced through tube 46 into stand-pipe 47 so as to prevent clogging. Excess coke formed in the process is removed from the system through pipe 47 and slide valve 48. A deflector 49 formed by two cones joined at their bases is positioned below the frusto-conical baffle 43 on a movable support 50 so as to provide an adjustable opening or channel 51 between the upper cone of deflector 49 and the lower edge of baffle 43. As controlled by opening 51, the fluidized coke naturally flows from higher density zone 44 into the surrounding lower density zone 45. The lower cone of deflector 49 shields zone 44 from the incoming stream by deflecting the oxygen away from the opening 51. The deflected gas rises through the annular zone 45, causing fluidization and burning of the powdered coke. At the same time, the heavy hydrocarbon material charged through pipe 52 and spray nozzle 52a undergoes coking and conversion to relatively lower boiling material in zone 44. The body of coke in both zones has a pseudo-liquid level 53 at which point the hydrocarbon products and hydrogen resulting from the coking operation as well as the gases resulting from the combustion of coke disengage themselves from the bulk of the coke mass. The gas streams from the combustion and coking zones merge in space 54 above the coke mass. Any coke remaining in the reaction gases is removed therefrom by cyclone separator 55 and returned to zone 44 by way of standpipe 56. The reaction gases pass through pipe 57 to conventional recovery and fractionation equipment.

The rate of circulation of coke through the system again largely depends upon the difference in fluid-static head maintained between zones 44 and 45. The fluid-static head is dependent upon the density (total weight of powder and gas present per unit volume of fluidized mass) and the height of the fluidized mass. The density of the fluid mass is in turn dependent upon such variables as solid density, size and shape, and gas density, velocity and viscosity. Gas densities and viscosities may be altered by changes in temperature and pressure. While any one or combination of the direct and indirect variables may be employed to create a difference in the fluid-static heads of the two reaction zones of my apparatus, for simplicity and ease of operation, I generally rely on gas velocity as the principal variable in the attainment of different fluid-static heads. Apparatus of the type shown in Figure 3 is included in the subject matter of my copending U. S. patent application, Serial No. 547,722, filed on August 2, 1944.

It is desirable that the difference in fluid-static head between the two zones of the reactor be not less than about 1 pound per square inch, and preferably not less than about 3 pounds per square inch. Such pressure differences promote satisfactory circulation of the fluidized coke through the two zones.

The foregoing description of the three illustrative embodiments of my invention pertains to the state of affairs prevailing when the reactors are "on-stream." If the reactors were being started up for the first time, powdered coke of petroleum or even coal origin, prepared by any conventional method, would be charged into the reactors through suitable openings (not shown) in the upper portions of the reactors or through

pipes employed for the introduction of the fluid stream. As an example of the latter method, powdered coke, all of which will pass through a 60-mesh screen, is suspended in a stream of air preheated to a temperature of about 600° F. and, thus suspended, is conveyed by the air through pipe 2 into reactor 1 of Figure 1. The preheated air will partially burn the coke particles which form a fluidized mass within the reactor 1. When sufficient coke has been carried into reactor 1 to bring the pseudo-liquid level 3 of the fluidized mass to the desired height, no further coke from an extraneous source is added. As soon as the temperature of the fluidized coke bed has risen to about 800° F. or higher, the injection of oil by way of line 3 and nozzle 4 may be commenced. At the same time, oxygen of not less than 80% by volume purity, preferably not less than 95%, is used instead of air in pipe 2 to continue the partial combustion of the coke and to supply heat to the endothermic cracking conversion of the oil discharged from nozzle 4. The continued "on-stream" operation of the process has already been described.

The technique of fluidization by which comminuted solids are so treated with gases or vapors that the mass of solids is mobile and seething like a boiling liquid is already well developed and understood. Those skilled in the art know that any desired state of fluidization is attainable by controlling and balancing the various factors, like particle size, solid density and gas velocity. Thus, various investigators of fluid systems have advised the use of powders having a particle size of about 100 to 400 mesh as well as powders considerably coarser and finer. I have found it desirable to work with comminuted coke which is of such particle size that substantially all of it will pass through a 60-mesh screen and about 20 to 60% will pass through a 200-mesh screen.

To recapitulate, my invention provides a process for converting heavy hydrocarbons to valuable gaseous liquid and solid products within a single fluidizing reactor, in which simultaneously the hydrocarbons are pyrolytically converted and product coke is at least partially burned to supply the heat of pyrolysis and to form a useful gas rich in carbon oxides. The mixture of conversion and combustion products is then separated into desired fractions. Thus, for instance, from the combined products there may be fractionated by conventional methods gas oil, gasoline, a fraction in which C₃ and C₄ hydrocarbons predominate, and a gaseous residue comprising C₁ and C₂ hydrocarbons, carbon monoxide and dioxide, and hydrogen. The last-mentioned fraction is particularly well suited for the generation of synthesis gas, e. g., by passage through a reformer. The synthesis gas, a mixture of carbon monoxide and hydrogen, is then utilized in a Fischer-type catalytic process to form motor fuel and other hydrocarbon products. Those skilled in the art will appreciate that the same gas fraction may be converted directly into gaseous feeds for catalytic processes like hydrogenation and methanol synthesis. If desired, carbon dioxide may be separated for the manufacture of Dry Ice.

Hereinabove it has been emphasized that by my invention coke formed in the process is partially burned to supply the heat of pyrolysis and at the same time yield gas which is industrially useful. In other words, all the materials charged to my fluidizing reactor come out in a utilizable form; there are no waste products, like flue gases, which are commonly associated with prior processes.

While it has been stated that the comminuted coke formed in my process is partially burned to provide the required heat of pyrolysis, it will be realized that product coke may be consumed in excess of the thermal requirements when it is desired to produce a larger proportion of carbon oxides. In such case, excess heat may be withdrawn from the system, e. g., by generating steam in tubes disposed in the combustion zone of my unitary reactor.

Furthermore, where a large production of carbon oxides is desired to supply a synthetic process of the Fischer type, steam as well as oxygen may be introduced into the hot combustion zone to increase the yield of hydrogen and carbon monoxide. This is accomplished by the well known water-gas reaction; temperatures above about 1600° F. are favorable to this reaction. In a Fischer-type process, carbon dioxide is an unreactive diluent of the synthesis gas comprising carbon monoxide and hydrogen, and therefore it is desirable to suppress its formation. When a temperature of about 1100° F. is maintained in the combustion zone, the gases of combustion are predominantly a mixture of carbon oxides, in the approximate proportions of 1 part by volume of carbon monoxide to 2 parts of carbon dioxide. By permitting a higher temperature in the combustion zone, say above about 1600° F., the ratio of carbon monoxide to carbon dioxide is increased. Particularly at these higher temperatures, another way of converting carbon dioxide to the more useful carbon monoxide is to recycle carbon dioxide to the combustion zone after it has been scrubbed out of the mixed gases either prior or subsequent to a utilization step such as a Fischer-type synthesis operation.

Under some circumstances, it is advisable to inject the heavy hydrocarbon oil into the pyrolysis zone with the aid of steam; the presence of steam in the conversion zone often tends to promote increased production of liquid hydrocarbons.

Heavy hydrocarbon materials which may be converted by my fluid process include the various residues of petroleum refining operations, natural asphalts, tar sands, coal tars and pitches. When a material has very high viscosity even at elevated temperatures, it may be preferable to pulverize it at low temperatures and to charge the discrete particles to the fluidizing reactor rather than to melt and atomize the viscous material.

While the fluidizing reactors shown in the accompanying drawing exemplify the generally preferred type in which the powdered solids are withdrawn from the relatively dense, fluidized mass below the pseudo-liquid level, my process may also be conducted in the up-flow type of reactor in which the solids leave the reactor by entrainment in the effluent gases. Up-flow fluidizing reactors have been widely depicted in the technical literature. Those skilled in the art appreciate that a prime consideration in the adoption of a fluidized system is the cost of the equipment. My unitary reactor design achieves marked economies not only because of its simplicity and compactness, but also because of the reduction in size. The use of oxygen makes it possible to reduce the horizontal cross-section of the combustion zone to about one-fourth of that which would be required for air.

The above description and examples have been set forth with the purpose of being illustrative. Variations of my invention, conforming to its

spirit, are to be considered within the scope of the claims.

What I claim is:

1. A unitary reactor for conducting two fluidizing reactions simultaneously, which comprises a vertically elongate vessel, internal baffling which forms contiguous zones in the lower portion of said vessel, a U-tube connected to the bottom of said contiguous zones, means for introducing a fluidizing medium into one of said zones in the vicinity of its bottom, means for introducing a fluidizing medium into the other of said zones through said U-tube, and an outlet at the top of said reactor for discharging the mixed fluiding media from said zones, said reactor being constructed and arranged for promoting flow of fluidized material from one zone into the other across said baffling as a result of different fluid-static heads maintained in said respective zones.

2. The apparatus of claim 1 wherein a grinding device is disposed in one leg of the U-tube to reduce oversize particles flowing through said U-tube.

3. A noncatalytic process for the simultaneous conversion of heavy hydrocarbons of the type of topped petroleum crude oil to more volatile hydrocarbons, hydrogen and carbon monoxide, which comprises injecting said heavy hydrocarbons having a temperature not exceeding about 600° F. in discrete form into one zone of a single fluidized mass of coke particles deriving from said heavy hydrocarbons, maintaining said zone at a temperature above 800° F. to effect cracking of said hydrocarbons, cracking said heavy hydrocarbons to more volatile hydrocarbons, hydrogen and coke in particle form solely by contact with said fluidized mass, introducing oxygen of at least 80% by volume purity into another zone of said fluidized mass, both said zones being in merging relationship to permit the free movement of the fluidized coke particles therebetween, reacting said oxygen with said fluidized mass in the last said zone maintained at a temperature above 1600° F. to generate carbon monoxide and heat, promoting and controlling circulation of said fluidized mass by maintaining unequal gas velocities through said merging zones to convey said heat from the last said zone to the first said zone and thus to maintain said temperature above 800° F. in the first said zone, and withdrawing from said fluidized mass a single gaseous effluent containing said volatile hydrocarbons, hydrogen and carbon monoxide.

4. A noncatalytic process for the simultaneous conversion of heavy hydrocarbons of the type of topped petroleum crude oil to more volatile hydrocarbons, hydrogen and carbon monoxide, which comprises injecting said heavy hydrocarbons having a temperature not exceeding about 600° F. in discrete form into one zone of a single fluidized mass of coke particles deriving from said heavy hydrocarbons, maintaining said zone at a temperature above 800° F. to effect cracking of said hydrocarbons, cracking said heavy hydrocarbons to more volatile hydrocarbons, hydrogen and coke in particle form solely by contact with

said fluidized mass, introducing oxygen of at least 95% by volume purity into another zone of said fluidized mass, both said zones being in merging relationship to permit the free movement of the fluidized coke particles therebetween, reacting said oxygen with said fluidized mass in the last said zone maintained at a temperature above 1600° F. to generate carbon monoxide and heat, promoting and controlling circulation of said fluidized mass by maintaining unequal gas velocities through said merging zones to convey said heat from the last said zone to the first said zone and thus to maintain said temperature above 800° F. in the first said zone, and withdrawing from said fluidized mass a single gaseous effluent containing said volatile hydrocarbons, hydrogen and carbon monoxide.

5. A noncatalytic process for the simultaneous conversion of heavy hydrocarbons of the type of topped petroleum crude oil to more volatile hydrocarbons, hydrogen and carbon monoxide, which comprises injecting said heavy hydrocarbons having a temperature not exceeding about 600° F. in discrete form into one zone of a single fluidized mass of coke particles deriving from said heavy hydrocarbons, maintaining said zone at a temperature above 800° F. to effect cracking of said hydrocarbons, cracking said heavy hydrocarbons to more volatile hydrocarbons, hydrogen and coke in particle form solely by contact with said fluidized mass, introducing a gaseous stream consisting of a major proportion of oxygen of at least 95% by volume purity and a minor proportion of steam into another zone of said fluidized mass, both said zones being in merging relationship to permit the free movement of the fluidized coke particles therebetween, reacting said gaseous stream with said fluidized mass in the last said zone maintained at a temperature above 1600° F. to generate carbon monoxide, additional hydrogen and heat, promoting and controlling circulation of said fluidized mass by maintaining unequal gas velocities through said merging zones to convey said heat from the last said zone to the first said zone and thus to maintain said temperature above 800° F. in the first said zone, and withdrawing from said fluidized mass a single gaseous effluent containing said volatile hydrocarbons, hydrogen and carbon monoxide.

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