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PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

Improvements in or relating to the Method of Producing Hydrogen.

We, PITTSBURGH CONSOLIDATION COAL COMPANY, a Corporation organised under the laws of the State of Pennsylvania, United States of America, of Koppers Building, Pittsburgh, Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the manufacture of hydrogen and, more particularly, to a method of and apparatus for making hydrogen from a gaseous hydrocarbon by reacting the latter with steam.

The primary object of the invention is to provide an improved method and apparatus for making hydrogen by the reaction of steam and a hydrocarbon gas in the presence of barium oxide.

Another object of the present invention is to react steam and a gaseous hydrocarbon in the presence of barium oxide under certain critical conditions of pressure and temperature so that a high yield of hydrogen is obtained with little heat other than that supplied by the reaction system being required to maintain the reaction.

A still further object of the invention is to provide an improved method for making hydrogen at elevated pressures and temperatures.

In accordance with the invention, a method and apparatus have been provided for producing hydrogen in high yields in relatively pure form under generally thermoneutral conditions, i.e., little or no heat other than that supplied in the reaction is required. The method comprises circulating steam and a hydrocarbon in gaseous phase in intimate contact with each other through a reaction zone containing sufficient barium oxide in granular form to absorb substantially all the carbon dioxide produced by the steam-hydrocarbon reaction, the temperature of the reaction zone being between 1700° and 2300°F., and the pressure being about one atmosphere absolute or greater. For each temperature within the range recited there is a corresponding minimum pressure above one atmosphere which must be maintained or exceeded. In the range 1700° to 2050°F., the minimum pressure is about one atmosphere and, in the range 2050° to 2300°F., the relationship between the temperature and minimum pressure is expressed by the following empirical equation:

$$(1)\ p = 1 + 4.9 \times 10^{-2}\ (t - 2050)$$

where p is pressure in atmospheres and t is temperature in °F.

The practice of the invention produces large yields of hydrogen gas of relatively high purity with very little heat being required to maintain the reaction. Optimum results are obtained if the temperature lies within the range 1750° to 2050°F. and the pressure is between 20 and 50 atmospheres. Also we prefer that there be at least 800 parts by weight of barium oxide present for each 100 parts by weight of carbon contained in the hydrocarbon gas passed through the bed of barium oxide. Furthermore, when the hydrocarbon gas consists essentially of methane, it is desirable and preferred to employ a steam-hydrocarbon ratio of from 2 to 1 to 4 to 1.

For a better understanding of the invention, its objects and advantages, reference should be had to the following description and to the attached drawings, in which:

Fig. 1 is a diagrammatic illustration of an apparatus for carrying out an embodiment of the invention; and

Fig. 2 is a diagrammatic illustration of a modification of the apparatus of Fig. 1 which is adapted to carry out another and preferred embodiment of the invention.

In the following description of a specific embodiment of the invention, by way of example only, the new process and apparatus are applied to the conversion of methane to hydrogen. It is to be understood, however, that the invention is generally applicable to any hydrocarbon gas which reacts with steam to produce hydrogen. Among such hydrocarbon gases are included the so-called permanent gases, such as methane, ethane, propane, or butane. Also included are hydrocarbon gases produced by the vaporization of liquid hydrocarbons at the temperatures of the reaction, as well as hydrocarbon gases resulting from the thermal decomposition or cracking of liquid or solid hydrocarbonaceous materials, such as coal tar pitch.

The apparatus shown in Fig. 1 and its operation will now be described. Numeral *10* designates a reaction vessel which is adapted to hold a bed of barium oxide supported upon a porous plate member 12 disposed near the bottom of the vessel. The bed of barium oxide is first brought to a temperature between 1700° and 2300°F. by any suitable means such as external heating of the vessel or by burning producer gas or pulverized coal in the vessel itself. As soon as this temperature is attained, steam and methane are introduced through valved conduits 14 and 16, respectively, through the bottom of the vessel *10*. The relative proportions of steam and methane preferably lie between 2 to 1 and 4 to 1 in order to obtain the optimum conversion of methane consistent with the efficient utilization of the steam. The amount of barium oxide contained in the bed is sufficient to absorb substantially all of the CO_2 produced as a result of the reaction between the steam and methane. Preferably, there are at least 800 parts by weight of the oxide for each 100 parts by weight of carbon contained in the methane passed through the bed. The particle size of the oxide and the velocity of the steam and methane are preferably regulated so that the bed of barium oxide is maintained as fixed or non-fluidized bed rather than as a fluidized bed. The latter, however, may be employed if desired. The pressure within the vessel is maintained above one atmosphere in accordance with the relationship expressed by equation 1 by means of a valve located in the product gas line 20.

The reaction between the steam and methane at a temperature between 1700° and 2300°F. under pressure as specified above produces a gas containing principally hydrogen with relatively small amounts of carbon dioxide, carbon monoxide and unreacted methane. Substantially all of the carbon dioxide produced is absorbed by the barium oxide *in situ* with the resulting generation of heat which serves to supply most, if not all, of the heat required for the endothermic reaction between the steam and the methane. Additional heat, although it is generally not required, may be supplied in any suitable manner as by external heating of the walls of the vessel (not shown) or by burning the necessary amount of carbonaceous material within the vessel itself, that is, by feeding in limited amounts of oxygen. The product gas is conveyed to any suitable storage place or to a subsequent processing stage through the conduit 20.

The regeneration of barium oxide from the barium carbonate produced in the above reaction may be accomplished by heating the bed to the decomposition temperature, preferably 2300° to 2350°F. by means of the heat supplied by the combustion of producer gas or pulverized coal. Preferably, however, the regeneration of the barium oxide is carried out in a fluidized bed rather than a fixed bed. In order to accomplish this, it is necessary to regulate the particle size of the oxide so that under certain velocities of inlet gas a non-fluidized bed may be maintained, that is, during the hydrogen generation cycle; and under certain other velocities of inlet gas, a fluidized bed may be maintained. The particle size range of the oxide to permit fluidized operation in the regeneration cycle and non-fluidized operation in the steam-hydrocarbon reaction cycle may be, for example, —20 to +200 mesh on the Tyler scale. The linear velocities of the inlet gases during the steam-hydrocarbon cycle may be of the order of 0.03 to 0.30 feet per second under the pressures required for this particular cycle. On the other hand, a fluidized oxide regeneration process which is carried out at substantially atmospheric pressure may be conducted at linear velocities of the order of 2.0 feet per second, i.e., 1.0 to 4.0 ft/sec.

The following table lists the percentages of each of the components of the dry gas product as produced under different sets of temperature and pressure under relatively long residence time conditions to permit the reaction to reach equilibrium. The table also includes, for purpose of comparison, the composition of gas produced under the same set of conditions where no barium oxide is employed. It should be borne in mind that where no barium oxide is employed all the heat must be supplied for the endothermic reaction of steam with methane, while in the cases where barium oxide is employed, very little or no heat need be supplied in the system to maintain the reaction. In the examples of the table, instead of pure barium oxide its equivalent $BaO.BaCO_3$ was employed. The latter is preferred in those instances where it is desired to operate at as low temperatures as possible since its regeneration temperature is lower than that of barium oxide.

Temperature °F.	*Pressure Atm. Absolute*	*H_2O/CH_4 Ratio*	*Gas Compositions Vol. %*				*Per cent of Conversion*	
			H_2	*CO*	*CO_2*	*CH_4*	*Steam*	*Methane*
Without $BaO.BaCO_3$								
1750	30	2	73.1	18.6	4.3	4	50.6	85.1
	30	3	75.1	16.6	6.4	1.9	39.3	92.1
1900	10	2	75.8	20.7	3.3	0.7	55.3	97.2
	30	2	74.7	20.3	3.4	1.5	54.0	94.0
With $BaO.BaCO_3$								
1750	30	2	98.1	0.3	—	1.7	92.8	93.5
	30	3	99.9	0.04	—	.03	66.5	99.9
1900	10	2	95.5	4.0	0.1	0.4	90.3	98.6
	30	2	97.2	1.4	0.03	1.4	91.9	94.6

Referring now to Fig. 2 of the drawings, the preferred embodiment of the invention is disclosed. The apparatus disclosed therein comprises three similar reaction vessels *30, 32* and *34*, respectively, in which a bed of barium oxide is adapted to be supported on porous plate 36, 38 and 40 respectively, in the lower portions of the several vessels at the temperatures and pressures previously specified. Each of the vessels is provided with corresponding inlet and outlet valved conduits for carrying gases.

A main steam line 42 connects with a hydrocarbon gas feed line 44 to form a conduit 46 for conveying a mixture of steam and hydrocarbon gas. A valve 48 in conduit 42 and a valve 50 in conduit 44 serve to control the flow of steam and hydrocarbon gas, respectively, into the common conduit 46. The latter opens into the top portion of vessel *30* under the control of a valve 52, while a branch conduit 54 and a branch conduit 56 connect conduit 46 with the corresponding top portions of vessels *32* and *34* with control valves 58 and and 60, respectively disposed in the branch conduits 54 and 56 and corresponding to control valve 52.

The three vessels *30, 32* and *34* are interconnected at the bottom of each vessel by means of gas conduits 62, 64 and 66, having valve 68, 70 and 72, respectively, disposed thereon. By means of these valved interconnecting conduits, gas may be conveyed from the bottom of one vessel to either of the other two vessels.

At the top of the three vessels *30, 32* and *34*, gas conduits 74, 76 and 78, respectively, are provided for conducting exit or product gases from the vessels to cyclone separators 80, 82 and 84, respectively. The latter serve to separate solids entrained in the discharged gases and return the solids to the beds in the vessels through dip legs 86, 88 and 90, respectively. The solid-free gases from the cyclone separators are carried by gas conduits 92, 94 and 96, respectively, to the juncture of the two main gaseous discharge systems. One system is adapted to carry off the hydrogen product when the vessel in question is functioning as a hydrogen generator as will be described later, while the other system is adapted to carry off flue gas when the vessel in question is operating to regenerate the barium oxide.

The hydrogen discharge system comprises a main gas conduit 98 leading from conduit 92 and communicating with conduits 94 and 96 by means of two branch conduits 100 and 102, respectively. Valves 104, 106 and 108 in conduits 98, 100 and 102 control the flow of hydrogen gas therethrough. The main line 98 is arranged in heat exchange relation with the main steam line 42 by means of a heat exchanger 110. A valve 112 in the hydrogen line 98 serves to regulate the pressure maintained in the vessels *30, 32* and *34* when they are functioning as hydrogen generators.

The flue gas discharge system comprises a main gas conduit 114 which connects directly with conduit 92 and with conduits 94 and 96 by means of branch conduit 116 and 118 respectively. Valves 120, 122 and 124 are provided in the conduits 114, 116 and 118, respectively, to control the flow of flue gas from the respective vessels when they are functioning as barium oxide regenerators. The main flue gas discharge conduit 114 is arranged to pass in heat exchange relation with the inlet gas-steam feed line 46.

Associated with the three vessels *30, 32* and *34* is a producer gas generation system for providing fuel to regenerate barium oxide in each of the vessels. This system comprises a coal lock hopper 126 which discharges into a motor-driven screw 128 through a connecting conduit 130. A conduit 132 connects the screw 128 to a steam line 134 which is a branch from the main steam conduit 42. The passage of steam through line 134 is controlled by a valve

136. The branch steam line 134 communicates with the bottom of a producer gas generator vessel 138 which is provided with a valve ash discharge conduit 140, an air inlet conduit 142, and a valved producer gas discharge line 144 at the top. The latter communicates with a cyclone separator 146 from which entrained solids are returned to vessel 138 through a dip leg 148. A main conduit 150 for carrying solid free producer gas leads from the cyclone 146 to the bottom of vessel *30* and communicates with the bottom of vessels *32* and *34* by means of branch conduits 152 and 154, respectively. Valves 156, 158 and 160 are disposed in conduits 150, 152 and 154, respectively, to control the flow of producer gas to the vessels acting as barium oxide regenerators. A branch air conduit 162 leading from the main air conduit 142 carries air to vessel *30* and to vessels *32* and *34* through branch conduits 164 and 166, respectively. Valves 168, 170 and 172 are provided in these air conduits 162, 164 and 166, respectively, to control the flow of air to the respective vessels. Also valves 174 and 176 are disposed in air conduits 142 and 162, respectively, to regulate the flow of air therethrough.

The apparatus shown in Fig. 2 and just described operates as follows. We have found that the yield of hydrogen is increased even above that obtained by operation of the system shown in Fig. 1 if the reaction between the steam and hydrocarbon gas is first conducted in the higher end of the range 1700° and 2300°F. and then completed at a temperature at least 100 degrees lower but still within the same range. This may be accomplished by establishing a temperature gradient of at least 100 degrees within the bed of barium oxide in one of the vessels *30, 32* or *34*.

The complete operation of the system takes place in three consecutive cycles. In a given cycle, each of the vessels *30, 32* and *34* performs a different function from that of the other two vessels; and in the three consecutive cycles, the same vessel performs a different function in each cycle. While one vessel is operating to regenerate barium oxide from barium carbonate formed in the preceding cycle, one of the other two vessels is utilized as a preheating zone to bring the fresh reactants up to temperature by means of a hot bed of barium oxide which was regenerated in the immediately preceding cycle. Since some reaction takes place between the steam and hydrocarbon gas in this preheating vessel, a temperature gradient is established between the inlet and outlet end of the bed and thus conditions the bed to act as the main hydrogen generator in the next ensuing cycle. The third vessel is one in which a temperature gradient obtains in the bed by virtue of its having operated at a preheating zone in the preceding cycle. This vessel functions as the main hydrogen generator and accordingly at the conclusion of the cycle contains largely barium carbonate which must then be regenerated in the next cycle.

Referring specifically to Fig. 2, let us assume the vessel *30* is operating as a barium oxide regenerator, i.e., the barium carbonate formed in the preceding cycle is being reconverted to barium oxide.

The operation of vessel *30* as a barium oxide regenerator is as follows. It should first be stated that, unless otherwise specifically provided, all numbered valves in the system are closed. Producer gas and air in the proper proportions are introduced into the vessel through conduits 150 and 162, respectively. Valves 156, 168 and 176 are open. The velocity of the gases so introduced is of the order of two feet per second. The particle size of the solids in the bed is between —20 and +200 mesh. Consequently, the bed of solids is maintained in a fluidized state upon the porous plate 36. The combustion of the producer gas is regulated to produce a temperature in the bed in the neighbourhood of 2300° to 2350°F. which is above the decomposition point of barium carbonate. The pressure in the vessel at the top of the bed is essentially atmospheric. The flue gases of combustion and the carbon dioxide evolved from the carbonate are carried to the cyclone separator 80 by conduit 74 and are there freed of entrained solids which are returned to the bed by dip leg 86. The solid free gases are then discharged through conduits 92 and 114, valve 120 being open. The operation of the vessel is discontinued when the bed of solids consists essentially of barium oxide at a temperature of about 2300° to 2350°F.

The producer gas utilized in the above operation is generated in vessel 138. Finely divided coal from hopper 126 is fed through conduit 130 to screw 128 which forces the coal through conduit 132 into steam line 134. The steam picks up the coal and conveys it to vessel 138. Air is simultaneously introduced into the vessel through conduit 142, valve 174 being open. The coil particle size and inlet gas velocities are so regulated that a fluidized bed is maintained in the vessel. Ash is withdrawn as necessary through conduit 140 while producer gas is conducted through conduit 144 to cyclone separator 146. Entrained solids are returned to the bed through dip leg 148 and producer gas is discharged to conduit 150.

The operation of the preheating vessel *32* is as follows. Steam and methane are fed from conduits 42 and 44 into conduit 46 in relative proportions, regulated by valves 48 and 50, of preferably from 2 to 1 to 4 to 1.

The resulting mixture of steam and methane is circulated in heat exchange relation with the hot flue gas from vessel *30* and thereafter conducted by conduit 46 to branch conduit 54 in which valve 58 is open. Within the vessel *32*, a bed of finely divided barium oxide having a particle size between —20 and +200 mesh is supported upon the porous plate 38 in a fixed or non-fluidized state. The temperature of the bed is substantially uniform throughout and lies between about 2300° and 2350°F. by virtue of having been subjected to the above regeneration treatment in the preceding cycle. The pressure in the vessel is governed by the pressure established in vessel *34* as will be explained later and lies above one atmosphere.

The mixture of steam and methane entering the vessel *32* from conduit 54 is forced to circulate downwardly through the hot bed of barium oxide since all valves in the top discharge lines are closed. As the gaseous mixture progresses downwardly through the bed and becomes heated to reaction temperature, some reaction takes place. The carbon dioxide produced reacts with some of the barium oxide with resultant evolution of heat. A temperature gradient is therefore established in the bed with the lower temperature being at the top where most of the heating of the incoming gas is effected and where initial endothermic reaction between steam and methane occurs. The lower end of the bed is maintained substantially at the original temperature by the heat evolved in the barium oxide-CO_2 reaction. The amount of the temperature gradient can be regulated by the velocity and temperature of the inlet gases, and by the depth of the bed. We prefer that the gradient be at least 100°F. The downflowing gases leave vessel *32* through conduit 64 in which valve 70 is open and are then conveyed by conduit 66 to the bottom of vessel *34*, valve 72 being open. These gases are now at a temperature in the neighborhood of 2350°F. and contain, to a minor extent, some reaction products by virtue of the preliminary reaction in the preheating vessel *32*.

The operation of vessel *34* as a hydrogen generator is as follows. A bed comprising largely finely divided barium oxide of a particle size between —20 and +200 mesh is supported on the porous plate 40. A temperature gradient of at least 100°F. exists between the bottom and top of the bed by virtue of its having functioned as a preheating zone corresponding to vessel *32* in the immediately preceding cycle. The temperature at the bottom of the bed is the higher and lies in the neighborhood of about 2350°F. The preheated reactant gases from vessel *32* enter the bottom of the vessel *34* from conduit 66 at a velocity of 0.03 to 0.30 feet per second, which is insufficient to effect fluidization of the solids in the bed. However, the porous plate 40 serves to disperse the gases uniformly thorughout the bed. Most of the reaction between the steam and methane takes place at the higher temperature existing in the lower portion of the bed and is completed at the lower temperature obtained in the upper portion. The temperatures in the bed are maintained substantially at the same level by the heat of reaction between the barium oxide and carbon dioxide. Should any additional heat be required, it may be supplied by transfer through the vessel walls or by combustion of a portion of the methane in the bed by the addition of small amounts of oxygen through conduit 166 (valves 176 and 172 being open). The product gases, comprising largely hydrogen, are discharged from the vessel through conduit 78 to a cyclone separator 84. The latter returns entrained solids to the bed through dip leg 90. The solid free product gas is then conveyed through conduits 96 and 102 (valve 108 being open) to the main hydrogen line 98. The latter carries the hot gas in heat exchange relation with the inlet steam line 42 to a suitable storage or subsequent purification stage. Valve 112 is regulated to maintain the pressure in vessel *34* and also in vessel *32* at the desired value which is determined by equation 1, the temperature employed for this purpose being the lowest existing in the hydrogen generator *34*.

The composition of the gas produced in vessel *34* depends upon the temperatures and pressures employed but in general a somewhat larger percentage of hydrogen is obtained than in the case where no temperature gradient is employed. For example, reference to the above table shows that at 1900°F. and 30 atmospheres pressure with a H_2O/CH_4 ratio of 2/1, a gas containing 97% hydrogen is obtained. Under the same conditions except that the upper portion of the bed in vessel *30* is maintained at 1900°F. and the lower portion at 2300°F., a gas containing 98.5% hydrogen is produced. Although this type of operation does not produce hydrogen of substantially greater purity than the isothermal type of operation it has the advantages of (1) superior heat economy since the latent heat of the regenerated BaO bed is utilized and (2) a higher throughput is attained without sacrificing hydrogen purity by taking advantage of the higher reaction rate at the higher temperatures prevailing over a portion of the bed.

In the second cycle of operation of the three vessels *30*, *32* and *34*, vessel *30* operates as a preheating zone and consequently functions in the same manner as vessel *32* in the first cycle just described. Vessel *32* becomes the hydrogen generator and is operated the same as vessel *34* in the above described cycle. Vessel *34* becomes the barium oxide regenerator upon reduction of pressure to atmospheric corresponding to vessel *30* in the first cycle. Similarly in the third and last cycle, vessel *30* becomes the hydrogen generator; vessel *32*, the barium oxide regenerator; and vessel *34*, the preheating zone. Since each vessel is provided with corresponding conduits, etc., the operation of each in its respective role is the same as that of the corresponding role just described.

What we claim is:—

1. The method of producing hydrogen from gaseous hydrocarbons which comprises circulating a hydrocarbon in gaseous phase and steam in concurrent intimate contact with each other through a reaction zone containing barium oxide in granular form, maintaining the reaction zone at a temperature between 1700° and 2300°F. and at a pressure of at least one atmosphere absolute, the amount of oxide being sufficient to absorb substantially all the carbon dioxide produced, the minimum pressure of the reaction zone being about one atmosphere for the range 1700° to 2050°F. and being related to the temperature for the range 2050° to 2300°F. by the empirical equation

$$p=1+4.9\times10^{-2}\,(t-2050)$$

where p is the reaction pressure in atmospheres and t is the temperature of the reaction zone in °F.

2. The method according to Claim 1, in which the amount of oxide is at least 800 parts by weight for each 100 parts by weight of carbon contained in the gaseous hydrocarbon.

3. The method according to Claim 2, in which the temperature of the reaction zone is maintained between 1750° and 2050°F. and the pressure between 20 and 50 atmospheres.

4 The method according to any of the preceding claims, in which the hydrocarbon gas is methane and the steam-methane ratio is from 2 to 1 to 4 to 1.

5. The method according to Claim 1, in which there is a temperature gradient of at least 100°F. between the inlet and the outlet of the reaction zone with the higher temperature being at the inlet end.

6. The method of producing hydrogen which comprises heating a bed of granular barium oxide in a reaction zone to a substantially uniform temperature in the upper end of the range 1700° to 2300°F., circulating steam and a hydrocarbon in gaseous phase downwardly through said bed until a temperature gradient of at least 100°F. is established between the top and bottom thereof, circulating the gases issuing from the bottom of said reaction zone upwardly through a bed of granular barium oxide in a second reaction zone, the temperature of the bed in said second zone being between 1700° and 2300°F., maintaining a pressure in both of said reaction zones of at least one atmosphere absolute, the amount of barium oxide in said zones being sufficient to absorb all the carbon dioxide produced by the reaction of steam and hydrocarbon gas circulating therethrough, recovering the gaseous product from the second zone, thereafter circulating steam and hydrocarbon gas upwardly through the first zone, and recovering the gaseous product.

7. The method according to any of the preceding claims, wherein the barium oxide is regenerated from the barium carbonate resulting from the method according to the preceding claims, the regeneration being effected by heating the barium carbonate to its decomposition temperature of preferably between 2300° and 2350°F., the heat being preferably supplied by the combustion of producer gas or of pulverized coal.

8. The method according to Claim 7, wherein the regeneration takes place under fluidized conditions.

9. The method according to Claim 8, wherein the particle size of the barium oxide is so regulated that under certain velocities of inlet gas a fluidized bed is maintained, while under certain other velocities a non-fluidized bed is maintained, the non-fluidized bed being maintained during the hydrogen generation cycle.

10. The method according to Claim 9, wherein to permit fluidized operation in the regeneration cycle and non-fluidized operation in the steam-hydrocarbon reaction cycle, the particle size of the barium oxide is between —20 and +200 mesh on the Tyler scale, the inlet gas velocity during the steam-hydrocarbon reaction cycle is between 0.03 and 0.30 ft. per second, and the inlet gas velocity during the regeneration cycle is between 1.0 and 4.0 ft. per second.

11. The method of producing hydrogen substantially as described with reference to the accompanying drawings.

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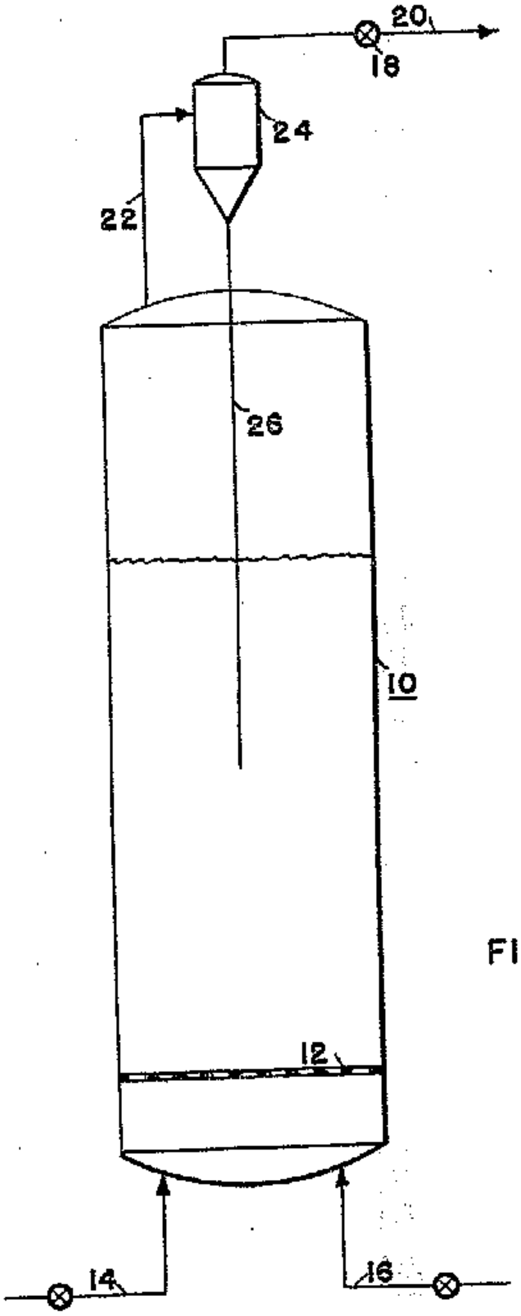

FIG. 1

673,332 COMPLETE SPECIFICATIO

2 SHEETS

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SHEETS 1 & 2

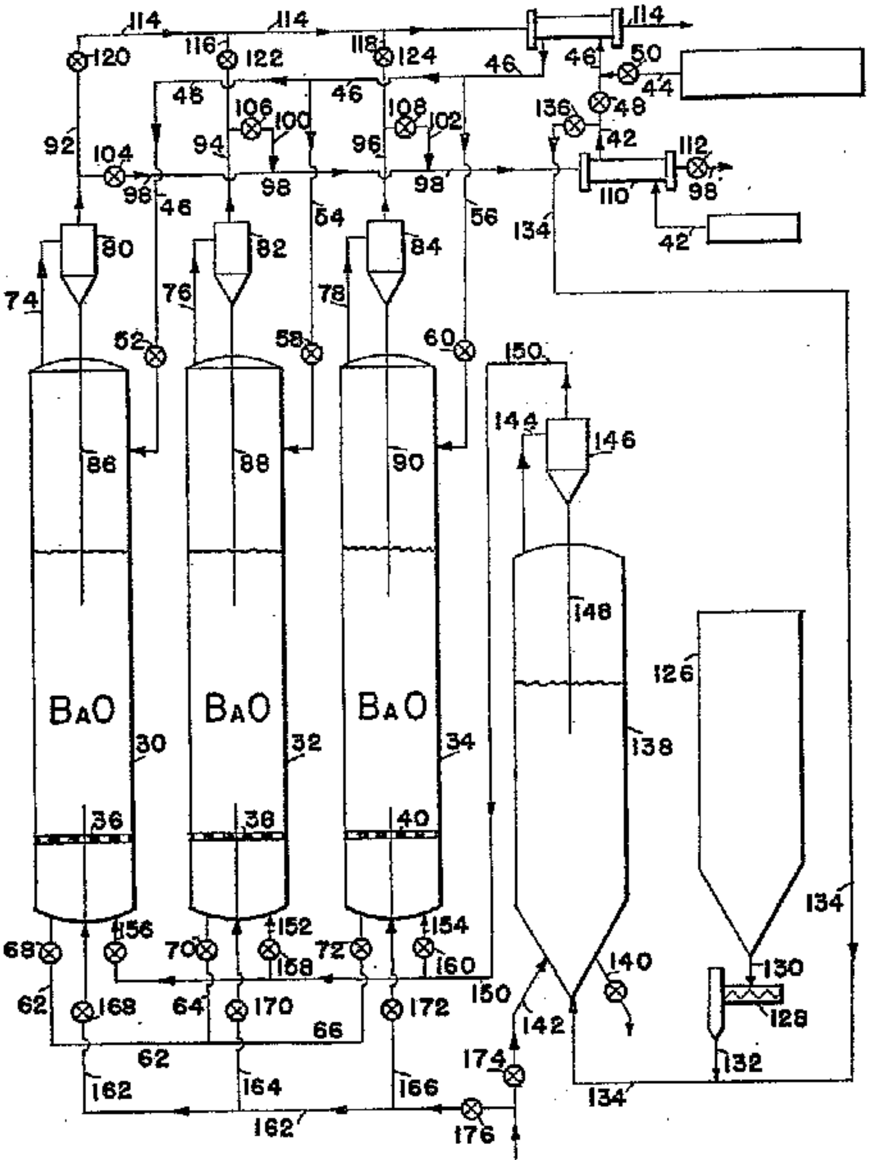

FIG. 2

673,332 COMPLETE SPECIFICATIO

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SHEETS 1 & 2

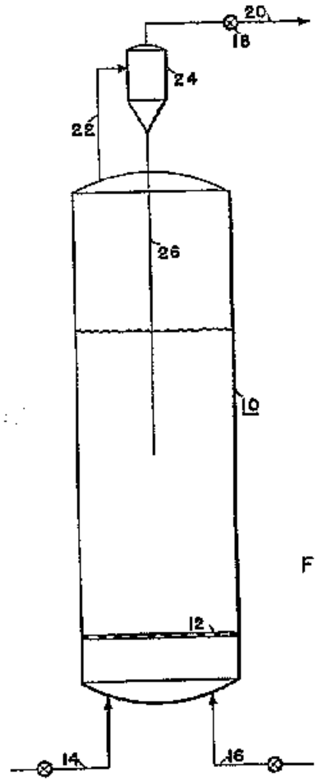

FIG. 1

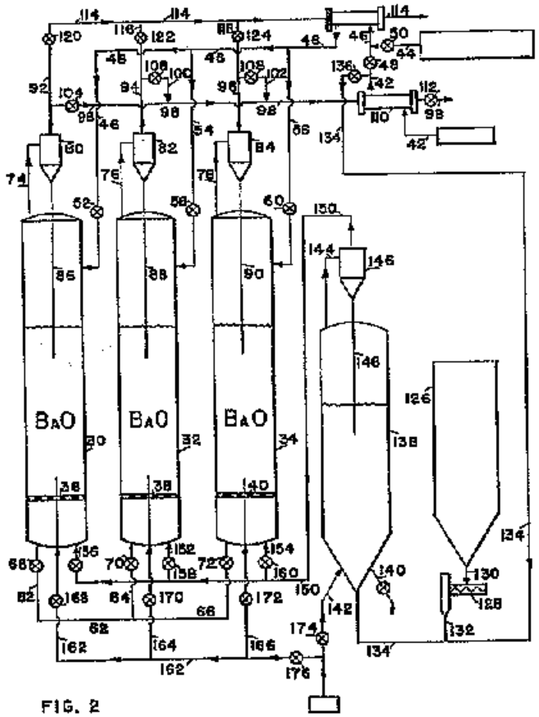

FIG. 2