

Sept. 23, 1958

H. KÖLBEL ET AL. 2,853,369
APPARATUS FOR CARRYING OUT GASEOUS CATALYTIC
REACTIONS IN LIQUID MEDIUM

Filed Jan. 12, 1955

7 Sheets-Sheet 1

Fig. 1

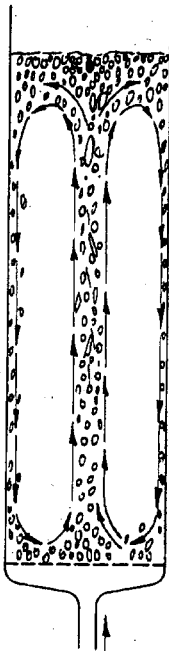


Fig. 2

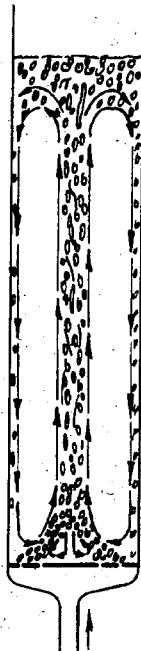
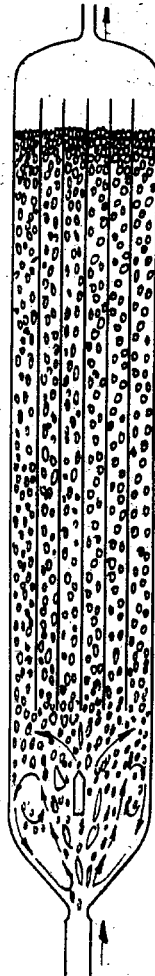


Fig. 3



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7 Sheets-Sheet 2

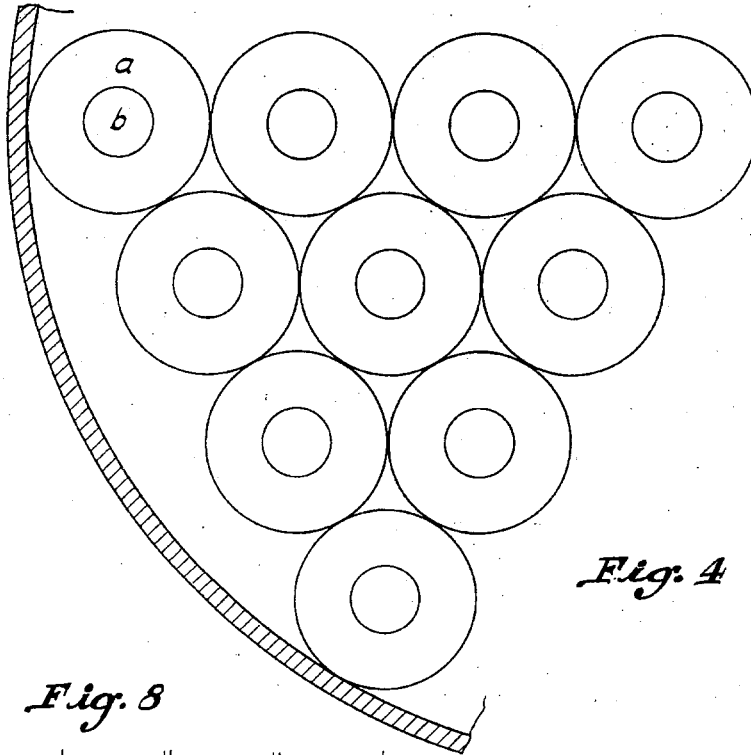


Fig. 4

Fig. 8

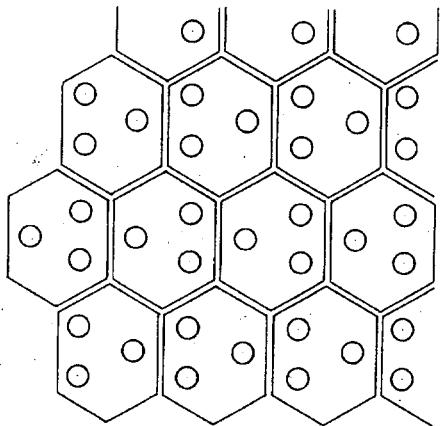
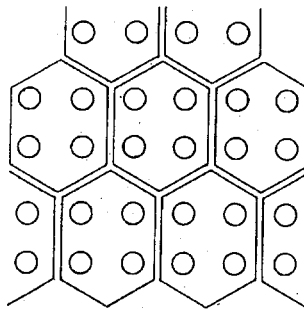


Fig. 9



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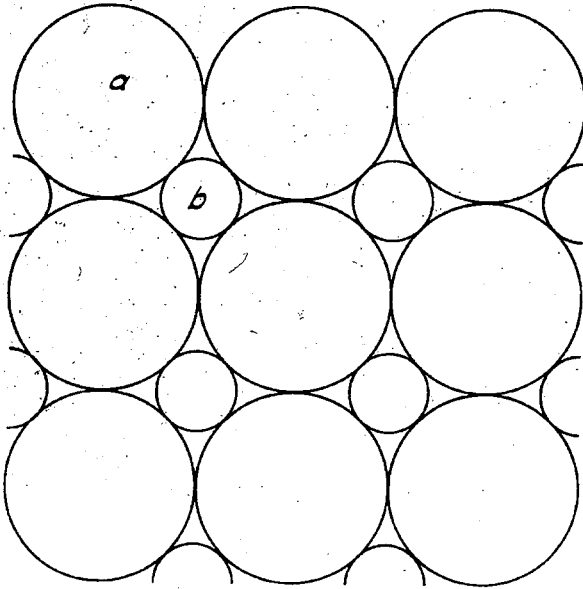


Fig. 5

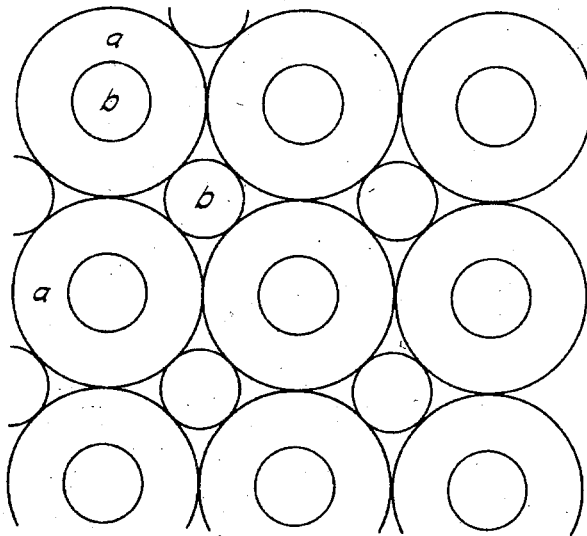


Fig. 6

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Fig. 7a

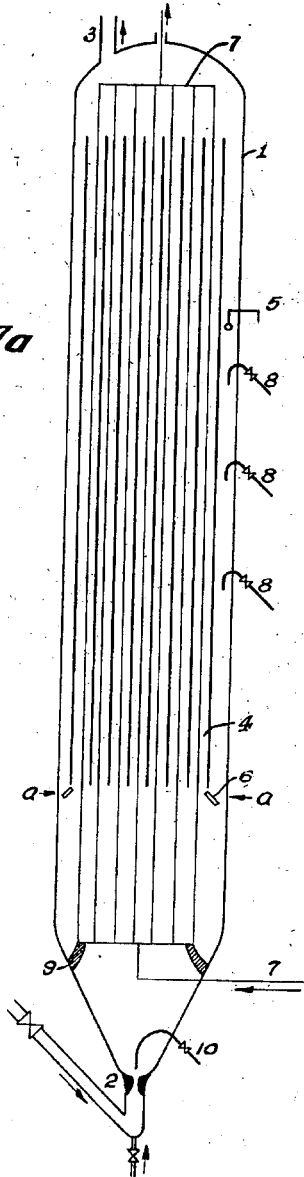
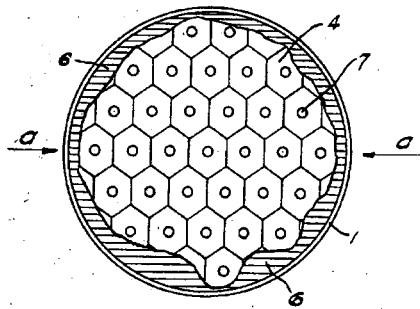


Fig. 7b



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Fig. 10 a

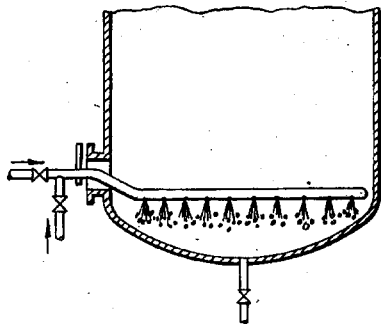


Fig. 10 c

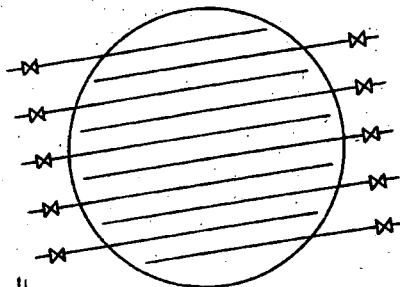


Fig. 10 b

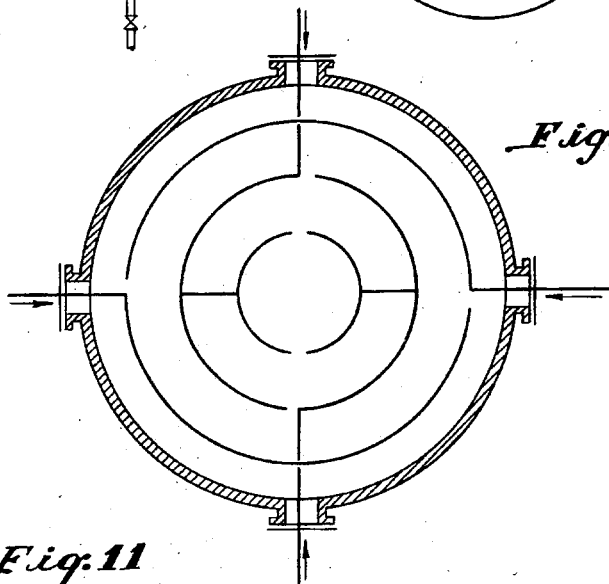
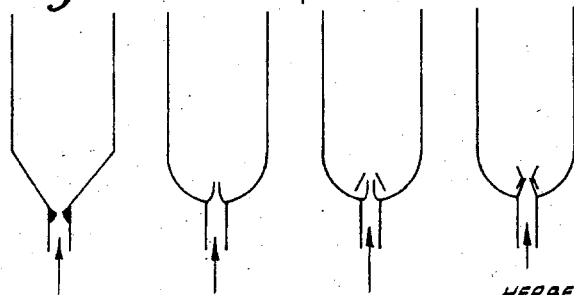


Fig. 11



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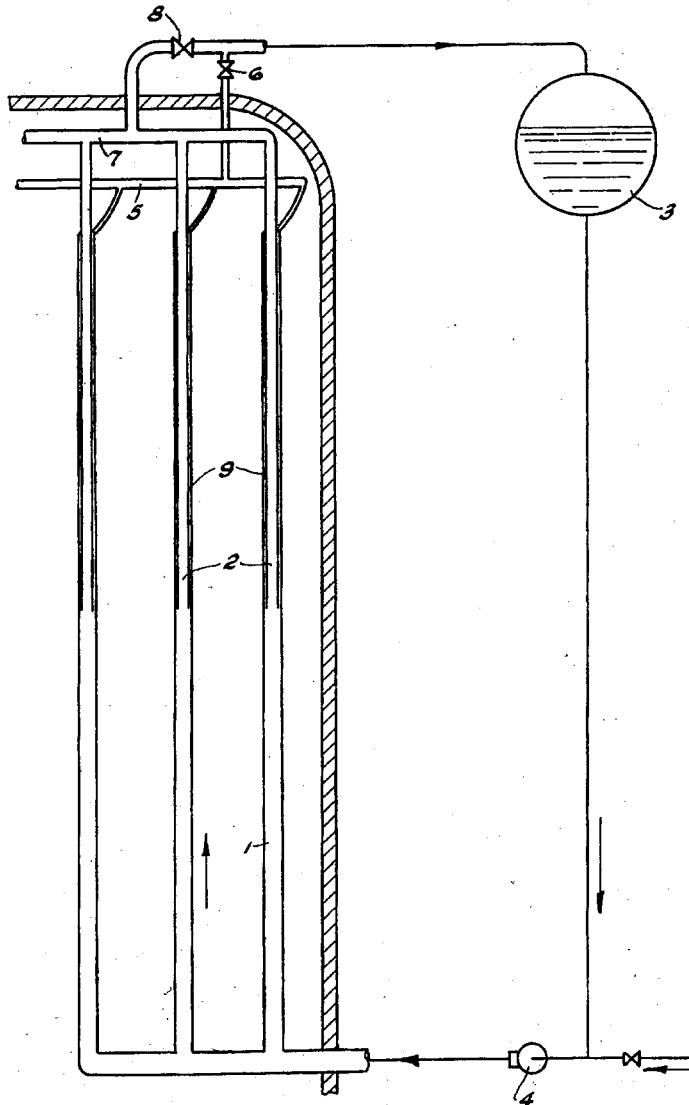
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Fig. 12



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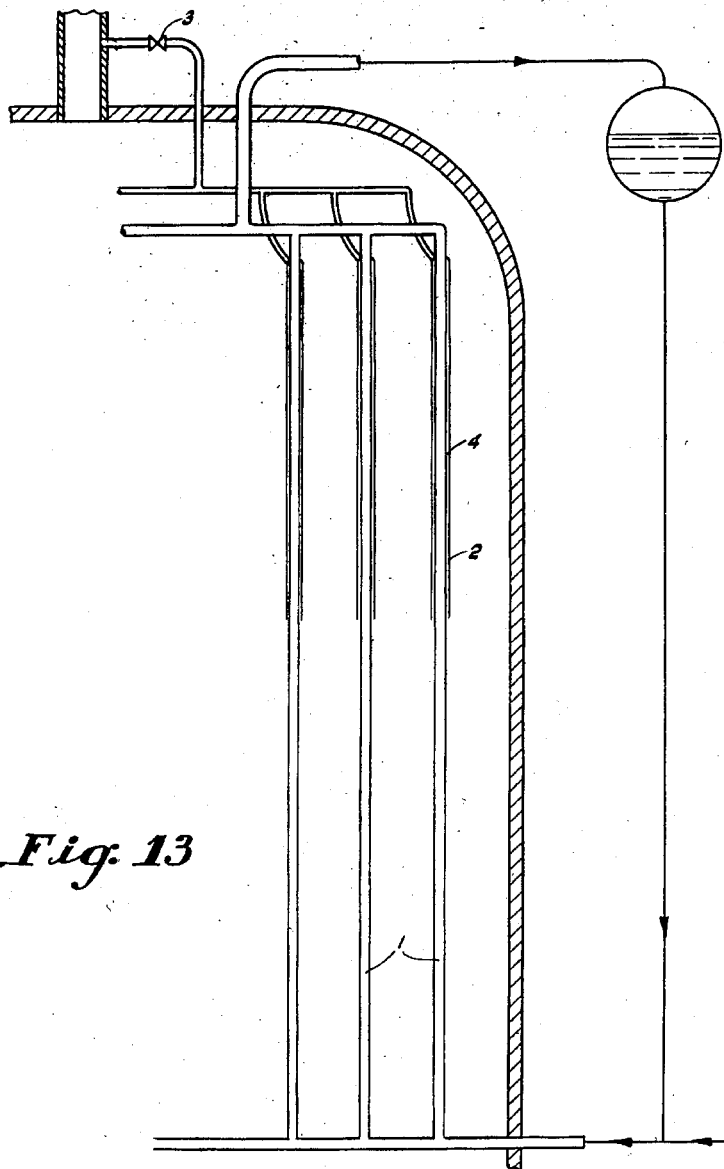


Fig. 13

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APPARATUS FOR CARRYING OUT GASEOUS CATALYTIC REACTIONS IN LIQUID MEDIUM

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Application January 12, 1955, Serial No. 481,468

16 Claims. (Cl. 23—288)

The present invention relates to an apparatus for carrying out gas reactions in the presence of liquid or solid catalysts dissolved in a liquid medium or held in suspension in finely divided form, especially for hydrogenation of carbon monoxide under increased gas pressure to form hydrocarbons or organic compounds containing oxygen or nitrogen.

The invention relates particularly to a reaction apparatus in which the liquid medium is held stationary during the gas reaction, that is, there is no liquid recirculation either inside or outside the reaction space.

The amount of gas conversion taking place in gas reactions in the presence of catalysts in a liquid medium, wherein the gaseous phase does not completely disappear on passage through the liquid medium, but is maintained as an independent phase by incomplete solubility of the gaseous reaction and end products throughout the entire length of the liquid phase, is, as is known, proportional to the magnitude of the contact surface between gas and liquid. In such a distribution system of gas in liquid in which the liquid forms a continuum, the magnitude of contact surface is dependent on the size of gas bubbles.

For certain processes, including Fischer-Tropsch synthesis, it has been shown as sufficient to operate with any size gas bubble, which is regulated almost independently of the type and method of introducing the gas into the liquid medium, on the basis of the surface tension and viscosity of the liquid under operating conditions. A special type of gas distribution is unnecessary. However, to attain a complete gas conversion, a definite residence time equalized as much as possible with the individual gas bubbles is necessary.

These requirements are fulfilled when the synthesis gas at high pressure and high temperature is forced from the bottom, without using a special device for gas dispersion, in a narrow and relatively high column of liquid in such amount that the volume charge amounts to between about 5 and 100 liters of gas measured at the operating pressure and temperature per liter liquid medium. There is thus formed, a certain distance above the gas inlet, a state of distribution of gas in liquid whereby gas bubbles of practically uniform size are distributed throughout the column across its entire horizontal cross section and move toward the top at practically uniform velocity. The total volume of the liquid-gas suspension system is directly proportional to the gas throughput whereas the total contact time of the gas bubbles is practically independent of the gas throughput. At increasing gas throughput the distances between the gas bubbles are diminished without the gas bubbles combining. The nature of this distribution system which we have indicated as a liquid-gas suspension system is not yet completely clarified. It is, however, known, that in this system the liquid is in a state of finest turbulence wherein a vortex appears only about of the order of magnitude of gas bubbles; that the liquid remains generally stationary, that is, only travels slightly towards the top with the gas bubbles; and that as a result of the friction between gas bubbles and liquid a contin-

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uous, rapid surface renewal takes place. Hereby, in combination with the fine turbulence the material exchange between gas phase, liquid phase, and catalyst surface is favored in such manner that the hydrogenation of carbon monoxide is no longer restricted to the liquid medium but proceeds at equally high velocity as in the known processes with liquid-like, whirled, dry catalyst, the so-called fluidized catalyst process (fluid bed). In the liquid phase process with stationary column of liquid up to 15 liters (760 mm. Hg, 0° C.) CO+H₂ per g. catalyst per hour can be converted. This conversion is even attained on passing the gas once through the liquid medium at temperatures below 300°.

Since a large amount of catalyst can be charged according to this process, it makes possible, in combination with a high maximum content of catalyst in suspension which can amount to 500 g. catalyst basic metal per liter unexpanded suspension without the suspension losing the properties of a slightly moving liquid, higher space-time yields of reaction product than according to other known liquid phase processes.

In Fischer-Tropsch synthesis with suspended iron catalyst, the grain size of which is between 0.005 and 1 mm., there was produced, at a yield of 170 g. per normal cubic meter CO+H₂ used and one gas throughput at about 275° C., 25 atmospheric gas pressure and an hourly throughput up to 1250 liters CO+H₂ (measured at 0° C. and 760 mm. Hg pressure) per liter reaction space in 24 hours, about 5 kg. synthesis product having more than 3 C atoms. The synthesis according to known processes is feasible without trouble in a reaction space of up to 20 cm. diameter. With increasing horizontal diameter of the reaction space the amount of gas conversion decreases and it is always more difficult to maintain a constant gas conversion. The larger the horizontal diameter of the reaction space, the more the liquid substance leans toward changing from stationary state to a state of vertical rotation. The liquid flows downwardly along the surface of the wall and flows along the bottom to the middle of the reaction space, whereby it is drawn out by the gas bubbles leaving at the middle of the bottom. The compressed central gas stream flows along with the liquid toward the top whereby the firmly compressed gas bubbles combine to form large elongated gas bubbles. Only at the upper reversal point of the liquid, in the vicinity of the surface of the column of liquid, the gas spreads out horizontally across the transverse section and the large gas particles are partially decomposed.

Figure 1 schematically illustrates the flow passage of gas and liquid. It is to be noted that the hydrodynamic pressure on the bottom of the gas distributor can be greater near the wall than in the center, whereby the gas passage is completely suppressed by the outer nozzles so that all the gas enters the reaction space through the central nozzles. Moreover, the flow velocity of the liquid currents increases with the absolute height of the liquid column. It is also useless to overcome this undesirable aspect only by peripheral discharge of the gas; as shown in Figure 2, a similar state is formed with the sole difference that in addition to the high liquid eddies a low eddy is formed under that formed by the gas stream.

In such a distribution system the contact time of the individual gas particles is extremely uneven as a result of the uneven size of the gas bubbles since part of the gas bubbles are involved in the recirculation of the liquid. Whereas on the one side, part of the gas does not generally come in contact with the catalyst, another part, together with the reaction products contained therein, does come in contact with the catalyst repeatedly and longer than necessary. Moreover, the final gas carried along in vertical recirculation dilutes the fresh gas.

The result of these liquid eddies or currents thus leads not only to a decreased gas conversion but also to the occurrence of undesired secondary reactions. Therefore, in the Fischer-Tropsch synthesis, the catalyst rapidly circulating with the liquid is considerably damaged by the rapid variations in the surrounding conditions and its serviceability is decreased in that it ages more quickly than in syntheses in stationary liquid columns where the individual particles of catalyst are maintained proportionately long in a definite zone of constant gas composition.

Various suggestions are known for improvement of the gas distribution in liquids in larger reaction spaces. Included herewith are the use of rotating means, so-called turbomixers, or introduction of the gas with a Segner's wheel. The fixing of the direction of the flow of gas bubbles entering at the bottom through porous plates or individual nozzles by guide means, such as pipes or sheets has also been suggested. These guide means are placed only on the bottom or near it and are shorter than the height of the liquid.

The installations have at times no individual liquid surfaces and operate like mammoth pumps, which is the reason that in such equipped reaction apparatus, although the circulation of liquid is regulated it is by all means too intense.

The disadvantageous results of circulation of liquid on the gas conversion, the product properties and the efficiency of the catalyst are not removed by these suggested processes. For gas reactions in which the final gas is chemically identical to the starting gas, such as in fat hardening these installations may be sufficient. For gas reactions in which the gas composition is changed, the reaction products must be delivered with the final gas, and the gas conversion should be complete as possible on passing the gas through once, all known suggestions are insufficient. The essential problems especially for Fischer-Tropsch synthesis—maintaining the liquid medium and the suspended catalyst stationary and nevertheless permitting the gas bubbles in uniform size and distribution to pass vertically through the liquid medium at equalized velocity—are not realized by any of the known processes.

It has now been found that, in gas reactions in the presence of liquid catalysts, or solid catalyst dissolved in a liquid medium or maintained in suspension therein in finely divided form, in reaction apparatus having a large horizontal diameter the above-described disadvantages can be overcome by subdividing the reaction space, consisting of a vertical cylinder having a horizontal diameter of more than 30 cm. and up to 3 m. or more, and more than 1.5 m. in height, which height above the gas inlet is at least as great as the diameter of the oven. Across most of the transverse section the column is subdivided into similar, vertical shafts, which are open at the top and bottom and have liquid-tight casing surfaces and a diameter of at least 5 cm., said shafts terminating in the free gas space above the highest level of the liquid expanded by the gas bubbles during operation.

Figure 3 illustrates the state of gas and liquid distribution obtained hereby. In the sump underneath the shafts the gas distribution is large and uneven. Liquid currents are formed here but to only slight vertical extent. The lower edge of the group of shafts acts upon the motion of the liquid and the gas in the sump as if it were the liquid surface, causing the liquid to roll with its upper reverse points at considerable distances from the lower shaft edges, and accordingly spread the stream of compressed gas bubbles toward the periphery. Hereby the shafts farther removed from the axis receive practically the same amount of gas as the central zone. In contrast to the sump there are formed in the shafts extremely stationary liquid columns whose expansion depends on the amount of gas.

The gas supply to the shafts is so extensively equalized by the fact that each shaft has its own surface in con-

trast with the upper common gas space, that the reaction apparatus according to the present invention exhibits the same operating results as a reaction apparatus which corresponds in its size only to an individual shaft of less than 20 cm. diameter and is provided, moreover, with an individual gas supply. In addition, the hydrostatic pressure of the liquid columns of the shafts is practically even for all shafts, independent of small or temporary differences in gas throughput.

The surprising effect of the division of the reaction apparatus into shafts according to the invention can be summarized as follows:

Equalizing effect of the shafts on the charging of the individual shafts with gas, practically independently of operation in the sump;

Formation of a stable liquid-gas suspension system in each shaft with practically stationary liquid column and with uniform sizes of gas bubbles and their rate of rise;

Automatic supply of the individual shafts with suspended catalyst is proportional to the particular amount of entering gas. If the introduction of gas to one shaft is prevented, for example, by a guide plate, the liquid in that shaft is practically free of suspended catalyst;

Complete gas reaction in the shafts identical with laboratory results under the same operating conditions and with the same output. Independence of the reaction process on the type of gas introduction or distribution at the bottom of the sump underneath the shafts.

A selection of special modifications of the reaction process of the invention is described as follows:

The diameter of the shafts should be at least 5 cm. and can amount to 30 cm. or more. It conforms to the height and to the cross section of the pipes installed in the shafts for heat exchange.

The height of the shafts conforms to the contact time of the gas bubbles with the catalyst suspension, which is suitably determined by small-scale experiments.

The ratio of the height to the diameter of the shafts can be between 10 and 200, preferably between 20 and 100.

Commercial manufacture of the reaction apparatus of the invention is greatly facilitated since each individual shaft comprises an individual casing. These casings must be liquid-tight and can be as thin as manufacture permits. Sheets up to 0.5 mm. wall thickness can be used, especially if corrosion can be eliminated.

Shafts of circular cross section, that is, standard thin-walled pipes can be used which are arranged in mutual dense packing (triangle fastening) or in square packing. Figures 4, 5 and 6 show the various arrangements of circular shafts, in which the shafts are identified by "a" and the heat-exchange pipes by "b."

An especially high utilization of the reaction space is made possible by ordinary, hexagonal hollow prisms since there are no dead spaces between the shafts.

To prevent the penetration of gas bubbles in the dead spaces between the circular shafts or in the segment spaces between the bundle of shafts and the inside wall of the reactor a guide member is mounted a slight distance directly underneath the lower edge of the perimeter of the bundle of shafts between this bundle of shafts and the inside wall of the oven, which guide member extends inwardly of the oven towards this bundle of shafts, and sufficient space being left free so that the liquid in the dead and segment spaces communicates with the liquid in the sump.

Figure 7a illustrates a modification of the reaction apparatus in longitudinal section.

The cylindrical reactor 1 conically narrowed at the lower end has at the bottom a central gas inlet 2 which is provided with nozzles, check valve, and a line for cleaning the nozzles with liquid. At 3 is placed the outlet for the product gas. The shafts are identified by 4. The regulator 5 for the contents of the reactor is placed in one of the segment spaces between the bundle of shafts

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and the inner wall of the reactor and regulates filling and discharge lines according to whether the contents of the reactor increases or diminishes during the reaction. Guide plates for preventing gas inlet in the segment spaces are identified by 6. A pipe system 7 for heat exchange is located on the supports 9 and its upper delivery pipe is conducted vertically by means of a stuffing box through the reactor wall. If necessary, catalyst-free liquid medium can be withdrawn from the reactor through pipes 8 to recover, for example, high molecular weight reaction products. The liquid exchange between shafts, sump, and segment spaces is generally sufficient for this purpose despite the practically stationary state in the shafts. The appropriate arrangement of a pipe for emptying and filling the reactor with catalyst suspension is identified by 10.

The pipe system for heat exchange should be conducted towards the bottom as deep as possible in the sump, whereby the discharge in the horizontal collecting pipe is advantageously placed about 100 cm. and more underneath the lower edge of the bundle of shafts.

The heat exchange pipes are conducted vertically through the reactor. All of them can be conducted through the inside of the shafts, as shown in Figures 4, 7, 8 and 9, or can be conducted in part, as in Figure 6, or entirely, as in Figure 5, through the intermediate spaces between the shafts.

When the pipes are conducted through the inside of the shafts, the distance between each pipe and between the pipe and the inside wall of the shaft should not be less than 3 cm.

The gas distribution at the bottom of the sump can also be effected across the entire cross-section, whereby in cases where solid, suspended catalysts are used the gas outlet openings can advantageously be directed downwardly towards the bottom. Furthermore, it is advantageous to wet the gas inlet nozzles from the gas side by means of a liquid medium. Figure 10 shows a modification thereof in a longitudinal section in the side view with gas and wash oil lines as well as downwardly directed gas outlet nozzles. Figures 10b and 10c show in top view two different types of gas inlet and distribution means across the bottom.

The introduction of gas in definite zones across the bottom as illustrated in Figure 10 makes possible a regulation of the amount of gas to each of these zones. In reaction apparatus of the present invention this regulation is necessary only in special instances, for example, after a standstill to whirl up catalyst sediment or when operating with such slight amount of gas that the cross-section charge is much below about 10-15 liters (at operating pressure and temperature) per square centimeter free reactor cross-sectional area per hour.

Possible modifications for central gas supply at the narrow lower part of the oven are illustrated in Figure 11.

In carrying out gas reactions in reaction apparatus of the present invention cross section charges in the range of about 3-200 operating liters of gas (operating pressure and temperature per cm.² free reactor cross sectional area per hour) have been determined as suitable for liquid-gas suspension systems.

Of greatest significance for the liquid-gas suspension system is the mobility of the liquid in operation, and the viscosity of the liquid medium containing the suspended catalyst should preferably be lower than about 3° E. (Engler), corresponding to about 21 centistokes. In the Fischer-Tropsch synthesis the reaction proceeds especially rapidly at suspension viscosities below about 1.4° E. or 5.2 centistokes.

For heating the reaction apparatus and for supplying heat of reaction in the case of endothermic reactions the heat of condensation of steam can be used in known manner, just as in the same pipe system for indirect heat exchange water can be evaporated for removing the heat of reaction.

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As a result of the intensely fine turbulence of the liquid in the liquid-gas suspension system in the shafts the transfer of heat to the cooling surface is hastened in such manner that even when using hydrocarbons as liquid medium that the heat transfer in vertical, smooth, steel pipes through which cooling water flows amounts to at least 300 kcal. and up to 600 kcal. per m.² cooling surface per 0° per hour.

In certain gas reactions, for example, Fischer-Tropsch synthesis, it can be an advantage to permit the temperature to rise from bottom to top as the gas gradually becomes poorer in active constituents. This can also be attained in the case of exothermic gas reactions either by decreasing the cooling surface or by providing a steam or gas buffer between the pipe (through which cooling medium flows) and the reaction medium.

Decreasing the cooling surface is effected according to known suggestions by decreasing the diameter of the cooling pipes. Even so the number of cooling pipes, calculated on the horizontal cross section, can be reduced in vertical direction when more than one cooling pipe is installed per shaft. Depending on the number of cooling pipes installed at the bottom in each shaft, two or more cooling pipes can be connected at one or several high zones so that only one cooling pipe leaves at the upper end of the shaft.

Furthermore, it is advantageous to provide a vapor buffer such as a steam buffer of variable height in the upper part of the cooling pipe, as shown in Figure 12. Dip pipes 2 are inserted in the cooling pipes 1. The cooling liquid such as water circulating through steam drum 3 and pump 4 can be flowed either entirely through collecting line 5 located outside the bundle of shafts or, depending on the amount of temperature rise in the reaction space, more or less completely through collecting line 7 located outside the bundle of shafts, which is regulated by valves 6 and 8 respectively. There is thus maintained an insulating layer of vapor such as steam in the intermediate annular insulating space 9 of regulatable length.

Figure 13 shows a modification for regulatable decrease of the transfer of heat in the direction of the passage of gas by pipes 2 open at the bottom and slipped over the cooling pipes, said pipes 2 being regulatably connected by a valve 3 with the gas space above the shafts. The length of the gas buffer formed in annular space 4 by extracting gas from the liquid medium is determined by more or less by opening valve 3. On account of clearness the shafts are not shown in Figures 12 and 13.

In reaction apparatus of the invention in place of a heat exchange system with evaporated cooling medium there can also be used such a system with unevaporated cooling medium. The cooling medium is thereby recirculated from bottom to top through the cooling system across an outside heat exchanger in which, for example, the heat of reaction is utilized for steam. Regulation of the vertical temperature course in the shafts is effected by regulating the circulating velocity of the cooling mediums.

A further advantage of the mode of operation of the reaction apparatus of the present invention is that in contrast to reaction apparatus with circulating catalyst suspension there is no deposition of catalyst at any place on the walls, apparently caused by the fine, intense turbulence of the liquid medium in the stationary distribution system.

The invention claimed is:

1. Apparatus for reacting a gas in the presence of a catalyst in a liquid medium that is held substantially stationary during the reaction as the gas in the form of bubbles is flowed upwardly through the liquid medium, comprising a reactor having a gas inlet and a gas outlet, a cylindrical vertically-positioned reaction oven whose diameter is greater than 30 centimeters and whose height is greater than 1.5 meters and which is positioned a height

above the gas inlet of at least as great as the diameter of the oven, liquid-tight casing surfaces subdividing said oven across most of its horizontal cross-section into a bundle of similar continuous and uninterrupted shafts whose minimum horizontal distance is greater than 5 centimeters, said shafts being open at the bottom and top and terminating at their tops at a spaced distance below the top of the reactor to leave a free gas space above the top level of the shafts for said liquid medium, a guide member mounted underneath the lower edge of the perimeter of said bundle and between said bundle and the wall of said reactor and extending inwardly of said oven toward said bundle for preventing large amounts of gas from penetrating the space between said bundle of shafts and the wall of said reactor.

2. Apparatus for reacting a gas in the presence of a catalyst in a liquid medium that is held substantially stationary as the gas in the form of bubbles is flowed upwardly through the liquid medium, comprising a vertical cylindrical reaction oven, liquid-tight casing surfaces subdividing said oven across most of its horizontal cross-section into a bundle of similar continuous and uninterrupted open shafts which terminate at their tops in a free gas space above the top level of the shafts, a first vertically elongated pipe mounted in at least one of said shafts and adapted to contain a liquid media in heat exchange relationship with the reaction mixture within said shaft, first valve means for controlling the rate of flow of said liquid media in said first pipe, a second pipe slightly larger than said first pipe and concentric therewith and extending from the upper end of said first pipe downwardly in said shaft to a level above the lower half of said first pipe whereby an annular space is formed between said pipes, a collecting manifold for collecting fluid from said annular space, second valve means for controlling the flow of said fluid from said collecting manifold to said space whereby the flow through said first pipe and said annular space is controllable by said valves so that the heat due to the reaction of said gas in said shafts vaporizes the fluid in said annular space to provide a layer of vapor of a regulated length in said space for insulating said first pipe from said heat of reaction mixture within said shaft to effect a gradual reduction in heat transfer from the reaction of the gas to said liquid media.

3. An apparatus of claim 1 wherein a guide member is mounted a slight distance underneath the lower edge of the perimeter of a bundle of shafts and between said bundle of shafts and the inside wall of said oven, said guide member extending inwardly of said oven toward said bundle of shafts to prevent the penetration of large amounts of gas in the space between said bundle of shafts and said inside wall of the reaction oven.

4. An apparatus of claim 2 wherein a guide member is mounted a slight distance underneath the lower edge of the perimeter of a bundle of shafts and between said bundle of shafts and the inside wall of said oven, said guide member extending inwardly of said oven toward said bundle of shafts to prevent the penetration of large amounts of gas in the space between said bundle of shafts and said inside wall of the reaction oven.

5. Reaction apparatus of claim 1 in which the height of the shaft is 20 to 100 times as great as the average diameter of the shaft.

6. Reaction apparatus of claim 1 in which each individual shaft is formed by an individual casing surface independent of that of the adjacent shafts.

7. Reaction apparatus of claim 1 in which the continuous and uninterrupted shafts are formed by circular pipes open at the bottom and top.

8. Reaction apparatus of claim 1 in which the continuous and uninterrupted shafts are formed by ordinary hexagonal hollow prisms open at the bottom and top.

9. Reaction apparatus of claim 1 in which the lower ends of the shafts are in the same horizontal plane.

10. Reaction apparatus of claim 1 in which regulator means for the liquid contents are placed in the space between the bundle of shafts and the inside wall of the oven wherein there is no passage of gas, said regulator means regulating liquid content filling and discharge lines for the reaction oven according to whether the quantity of liquid contents in said reaction oven increases or diminishes during the reaction.

11. Reaction apparatus of claim 1 having at least one pipe for withdrawing from the space between the bundle of shafts and the inside wall of the reaction oven high molecular weight reaction product, which is liquid under the reaction conditions and substantially free of suspended catalyst and which is not carried out from the reaction space with the product gas.

12. Reaction apparatus of claim 11 in which vertical pipes for indirectly supplying and removing heat are arranged coaxially to the shafts and these heat exchange pipes discharge into collecting pipes located outside the bundle of shafts for heat exchange media.

13. Reaction apparatus of claim 12 wherein there is a vertical distance of at least 100 cm. from the lower edge of the bundle of shafts to the lower collecting pipe for the heat transfer medium.

14. Reaction apparatus of claim 12 in which the vertical heat exchange pipes are conducted through the inside of the shafts and the distance between each of these pipes and between the inside wall of the shaft is more than 3 cm.

15. Reaction apparatus of claim 13 in which at least part of the heat exchange pipes are arranged outside the shafts.

16. Reaction apparatus of claim 13 in which the reaction space is narrowed underneath the bundle of shafts to an individual, axially arranged gas inlet opening.

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