April 7, 1970 A. POLGAR ETAL 3,505,029

APPARATUS FOR REACTING A PLURALITY OF FLUIDS

Filed June 9, 1966 4 Sheets-Sheet 1

FIG. 1

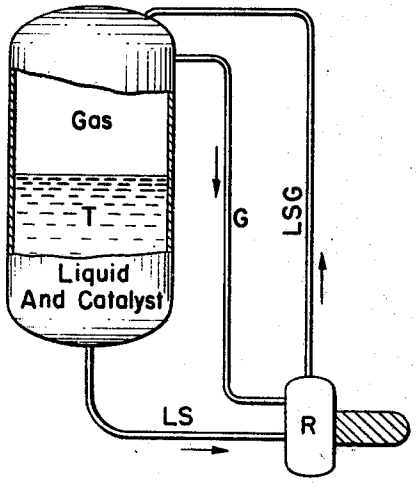


FIG. 2

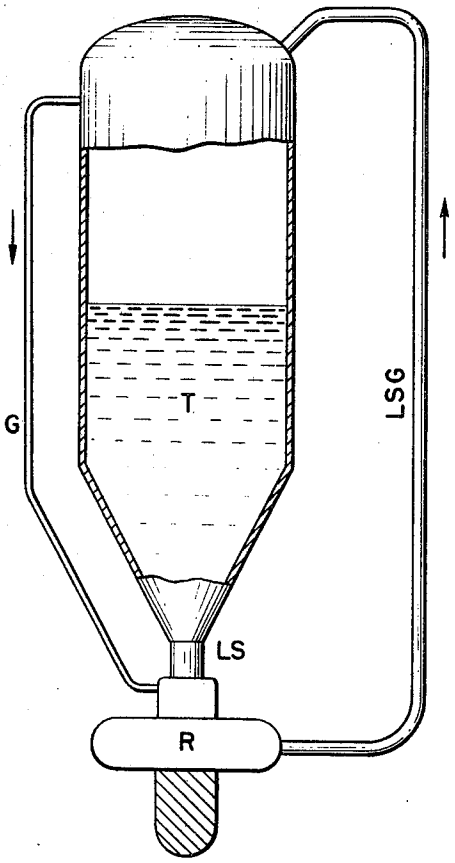
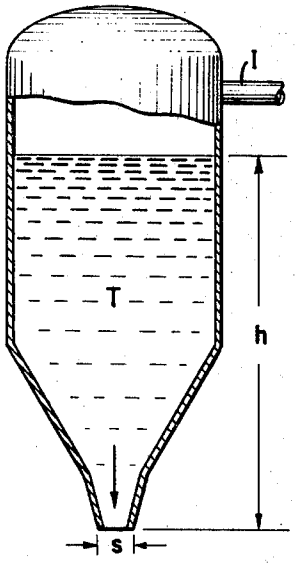


FIG. 3



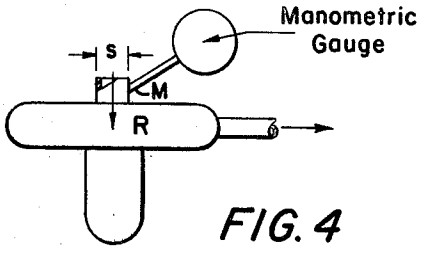


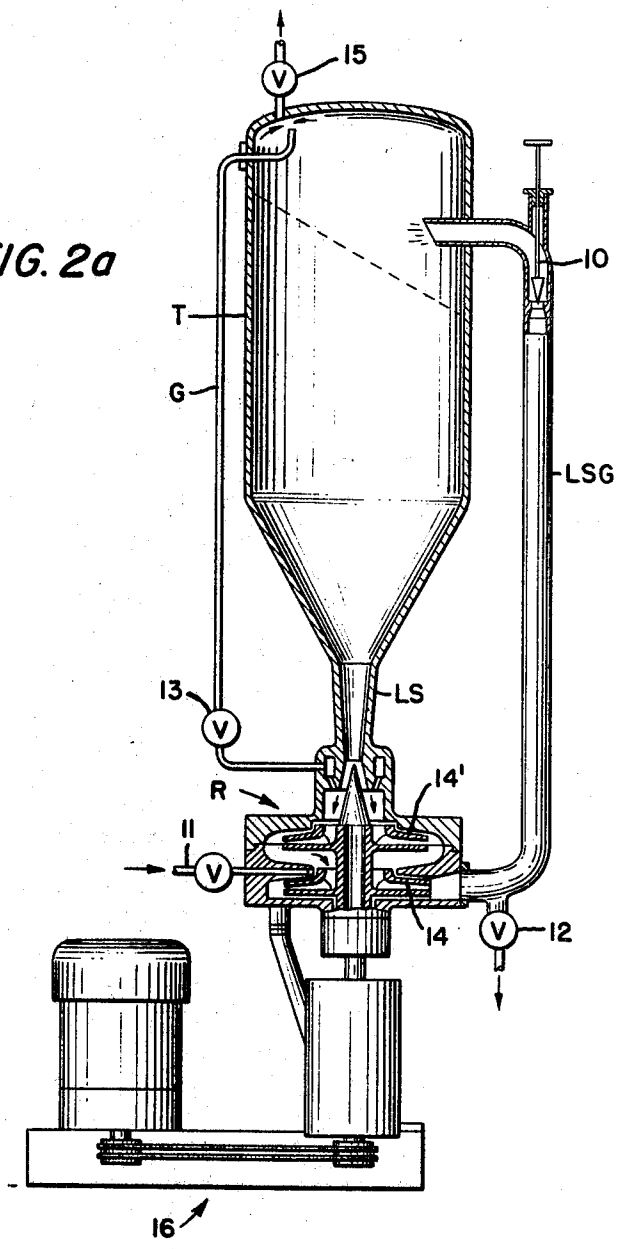
FIG. 4

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FIG. 2a



APPARATUS FOR REACTING A PLURALITY OF FLUIDS

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FIG. 5

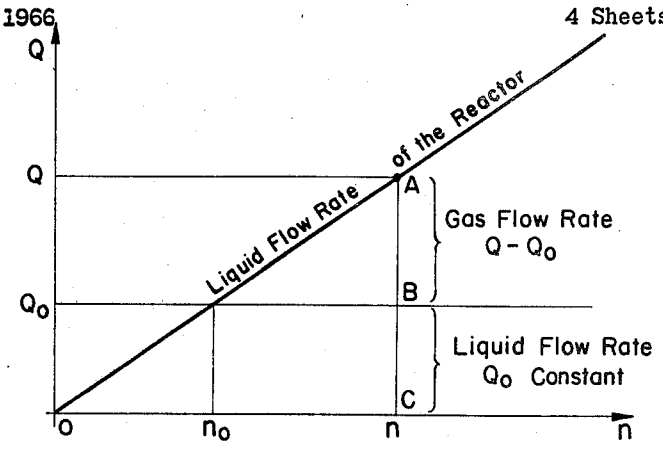


FIG. 6

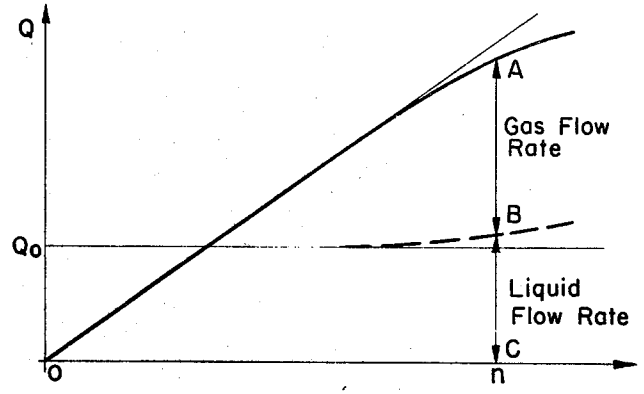
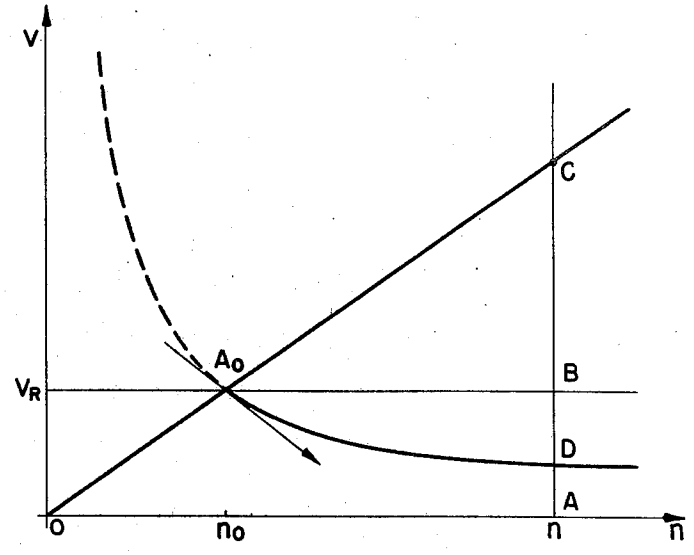


FIG. 7



APPARATUS FOR REACTING A PLURALITY OF FLUIDS

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FIG. 8

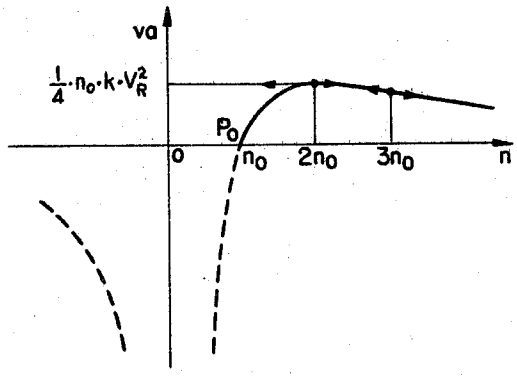
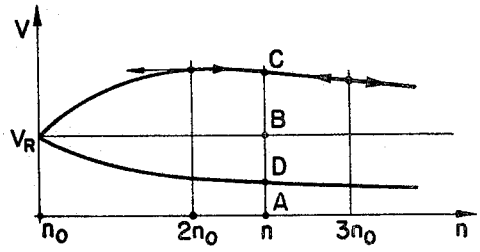


FIG. 9



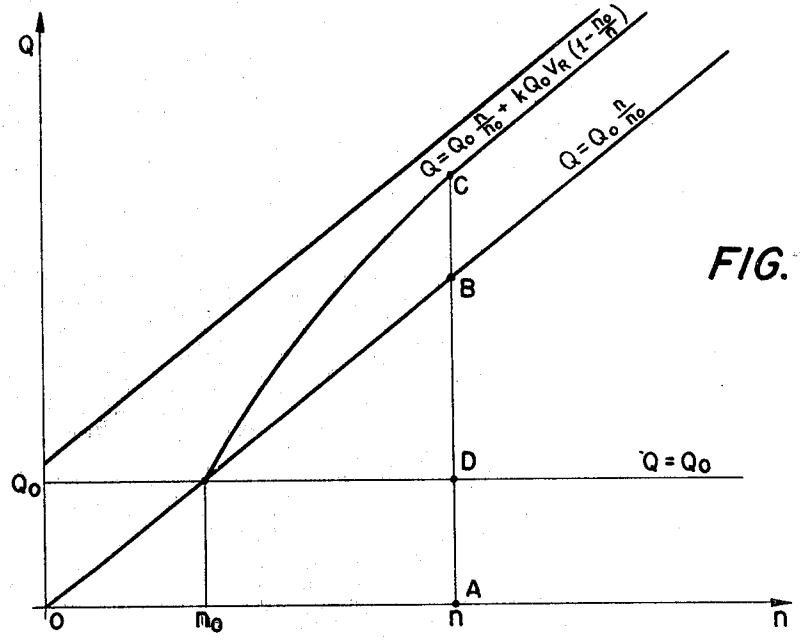


FIG. 10

FIG. 11

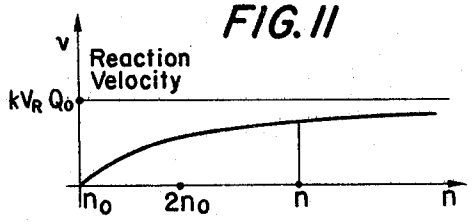
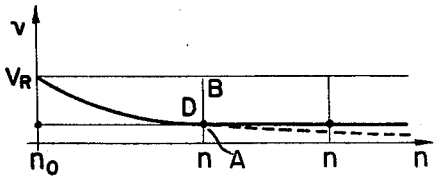


FIG. 12



United States Patent Office 3,505,029 Patented Apr. 7, 1970

3,505,029

APPARATUS FOR REACTING A PLURALITY OF FLUIDS

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Continuation-in-part of application Ser. No. 852,556, Nov. 12, 1959. This application June 9, 1966, Ser. No. 556,488

Claims priority, application France, Nov. 18, 1958, 779,383

Int. Cl. B01j 3/02

U.S. Cl. 23—285 11 Claims

ABSTRACT OF THE DISCLOSURE

Apparatus for interreacting two or more fluids comprises a reaction chamber and an impeller rotating in the reaction chamber to produce marked changes in the velocity, pressure and direction of fluid passing through the chamber. A passageway supplies a metered flow of one fluid from a container to the reaction chamber while a second fluid is drawn into the reaction chamber at a rate dependent on the speed of rotation of the impeller. Fluid is discharged from the reaction chamber back to the fluid container or other receptacle.

This is a continuation-in-part of our copending application Ser. No. 852,556, filed Nov. 12, 1959, now abandoned.

This invention relates to improved apparatus and methods for carrying out chemical reactions between gases and liquids, with or without dispersed solids.

Gas-liquid reactions are very important in chemistry. They may be very easy or very difficult to bring about. When the gas is soluble in the liquid and reacts rapidly, reaction proceeds fast. In many cases, when the gas is slightly soluble, reaction may be accelerated by a proper use of physical factors: pressure will increase the solubility of the gas; temperature will act on molecular velocity; above all, mixing is essential to insure good contact between the two phases, particularly when the solubility of the gas in the liquid is low.

Many of these reactions cannot be carried out in the absence of a catalyst. The catalyst is frequently a very finely divided powder, not soluble in the liquid; when such a catalyst is used, the system consists of three different phases, a solid phase, a liquid phase and a gas phase. A minimum requirement for carrying out a reaction in such a system is that the three phases be in mutual contact. Thus, aside from other considerations, a very difficult mixing problem is presented.

It is known that chemical reactions between gases and liquids, with or without dispersed solids, can be carried out in a system using a centrifugal pump. For this purpose, any type of centrifugal pump can be used.

For example, a known method of causing unsaturated compounds to react with hydrogen is to circulate the hydrogen and a dispersion containing the unsaturated compound at great speed in a closed circuit between at least one closed container and at least one centrifugal pump, so that there are several complete cycles per minute. In this case, a high capacity pump is used, which constitutes the reaction zone, assures recycling of the products, and draws in the reacting gases by suction.

Also, a known method of making cyanamide from calcium cyanamide by treating the calcium cyanamide with water and carbon dioxide is to gradually add the calcium cyanamide to the water and to circulate the water containing the calcium cyanamide by means of a centrifugal pump and to introduce the carbon dioxide into the system

downstream of the suction side of the centrifugal pump by means of a compressor and to beat the mixture of the water, calcium cyanamide and carbon dioxide with beater means located immediately downstream from the suction side of the centrifugal pump (U.S. Patent No. 1,444,255).

It is a principal object of this invention to provide new and improved apparatus and methods for facilitating the carrying out of gas-liquid reactions; the gas-liquid reaction may also involve a dispersed solid such as a catalyst. Other objects will become apparent from the detailed description and claims which follow.

According to the present invention, it has been found possible under conditions in which the gauge pressure would not normally permit, to effect chemical reactions between gases and liquids, which hitherto took place only under more rigorous conditions of pressure and mixing.

In the present invention, there is carried out very efficient reaction in a reactor defining a reaction chamber or zone which may be connected to a feed tank holding the reaction components. In the reaction chamber or zone of this invention there is provided remarkably thorough mixing. Furthermore, the reactants and catalyst, if any, are subjected together to a pressure increase while being intermixed and, at the same time, the reactant molecules are greatly accelerated. A result is remarkably efficient reaction. A further feature of the invention is that the reactor takes from the feed tank the proper ratio of liquid to gas. Thus, operation of the reactor in and of itself proportionates the liquid and gas. The liquid may have a solid dispersed in it and in that case the solid too will be proportionated, with the liquid. The resulting product can either be discharged or more generally be sent back to the feed tank one or more times to complete the reaction and then discharged.

The liquid to be reacted is fed to the reaction chamber through a conduit communicating between the reaction chamber and a source of the liquid, such as the reservoir referred to above. An impeller is rotatable in the reaction chamber to draw fluid into the chamber and to discharge it therefrom while producing marked changes in the velocity, pressure and direction of fluid passing through the reaction chamber.

A conduit is provided communicating between a supply of the gas to be reacted and the intake of the reaction chamber or zone, the pressure of the gas at the supply being higher than the pressure of said intake. Simultaneously with the feeding of the liquid to the reactor, the gas is allowed to be sucked into the reaction chamber or zone through the aforementioned conduit. It is found that the suction, when unthrottled, automatically proportionates the gas and the liquid. In the reaction chamber or zone, the above mentioned remarkable efficient mixing and a pressurizing and increase in velocity of the mixture take place and the driven means also accelerates the gas and liquid molecules, whereby the gas and liquid very efficiently react. Downstream of the impeller, the resultant reaction mixture may be discharged or part or all thereof be recycled to the reactor for one or more additional passes, should the same be necessary or desirable for more complete conversion.

The invention will now be described in detail, with reference to the drawings.

In the drawings:

FIG. 1 and FIG. 2 are each a diagrammatic elevation view, partly in section, of an apparatus of this invention;

FIG. 2*a* is an elevation view, partly in section, of a specific embodiment of the type of apparatus shown in simplified form in FIG. 2;

FIG. 3 shows, partly in section, the reservoir or tank of the apparatus of FIGS. 2 and 2*a*;

FIG. 4 is a schematic of the reactor portion of the apparatus of FIGS. 1, 2 and 2*a*, with a manometric gauge connected thereto;

FIGS. 5 to 12 are graphs for illustrating the theoretical and practical operation of the apparatus of the invention.

Referring to FIGURE 1 a pipe LS carries a liquid and a catalyst fed from a feed tank T by gravity and the proper quantity of a gas is sucked through pipe G from the upper part of the tank T and the two streams are mixed in a reactor R. The resulting product is sent back to the tank T through the pipe LGS and continuously replaced by fresh reactants from the tank T. The tank T may be provided with inlets communicating with sources of supply of additional quantities of the liquid and catalyst and of an additional quantity of the gas.

In order to achieve a high concentration of catalyst in the liquid and catalyst mixture fed to the reactor R from the feed tank T, the feeding may be by gravity. However, pump or pressure feeding may be substituted for gravity feeding or may be used to augment gravity feeding. The apparent density of the powdered catalyst is generally higher than the liquid density, and the catalyst has a tendency to settle. When the reactor R is placed under the feed tank (FIG. 2) the reactor R gets the maximum proportion of catalyst in the liquid and catalyst mixture because the catalyst is heavier than the liquid.

The operation of this invention may be explained by first considering the feed tank T which is fed through an inlet conduit I to maintain a constant level of liquid while its contents are flowing under gravity through a bottom outlet. The liquid level is at a constant height h above the outlet and the area of the bottom outlet is s. Liquid alone will be referred to since any dispersed solid present is carried along with the liquid and may be considered as part of the liquid. Thus, where the constant g is gravitational acceleration, the velocity (V_o) of liquid at the outlet is:

$$V_o=c\sqrt{2gh}$$

where c is a constant characteristic of the outlet. Similarly, the volume flow rate (Q_o) through the outlet is:

$$Q_o=cs\sqrt{2gh}$$

(Q_o may also be considered the flow required to maintain a constant level in the tank T).

In the apparatus of the invention the reactor R is provided with driven means for imparting an increase in pressure and velocity to and for mixing the liquid. The driven means comprises an impeller **14** (FIG. 2*a*), which generally includes vanes. For a value n_o of rotational speed of the impeller, flow rate Q out of the reactor will be equal to Q_o.

If the inlet of the reactor R is connected to the outlet of the feed tank T, all the liquid from the feed tank goes through the reactor. A manometer connected to a hole at point M in the wall of the reactor inlet will show n_o pressure. If the rotational speed n of the driven impeller is less than n_o and the volume flow rate capacity Q of the reactor, accordingly, is less than Q_o, the reactor cannot take the total flow from the feed tank. A pressure is developed at point M opposing gravitational flow. If the gauge would be removed, a stream of liquid would issue from the hole.

On the other hand, when the rotational speed n is greater than n_o and the volume flow rate capacity Q of the reactor, accordingly, is greater than Q_o, the reactor sucks liquid from the tank. A manometer connected to a hole at point M would indicate a negative pressure. Thus, if this hole and the upper part of the tank are connected with a pipe, gas is sucked through the pipe (G) from the upper part of the tank to the inside of the reactor (FIGS. 1, 2), when rotational speed n is greater than n_o.

At the reactor inlet, there is a negative pressure which can be cancelled by a supplementary admittance of liquid through the outlet of the feed tank or of gas through the pipe G. Owing to the low viscosity and internal friction of a gas compared with a liquid, it is found that essentially only gas will cancel the reduced pressure. Thus, gas will be admitted into the reactor at a flow rate $Q-Q_o$ which, added to liquid flow rate Q_o from the outlet of the tank, gives a total flow rate Q.

Thus, in a given apparatus of the present invention (as in FIG. 2*a*) an impeller rotational speed n_o exists below which no gas is sucked into the reactor and above which the volume flow rate of sucked gas is proportional to the impeller rotational speed and the volume flow rate of liquid through the reactor remains constant. This speed can be determined for a given tank and reactor. Thus,

$$Q_o=CS\sqrt{2gh}$$

where:

Q_o is the volume flow rate from the feed tank under gravity;
C is a constant, characteristic of the tank outlet (velocity coefficient);
S is the tank outlet area;
h is the height of liquid in the tank above the outlet;
g is the acceleration of gravity.

The curve representing volume flow rate values in relation to impeller rotational speed for the reactor is a straight line passing through the origin O (FIG. 5). One point A will determine it. Intersection of $Q=Q_o$ with that curve gives n_o. The curve gives the theoretical result. When gas compressibility and viscosity are considered, there is some modification. Though it is very small, gas viscosity exists and when large volume flow rates of gas are involved, the volume flow rates may be slightly lower in relation to the impeller rotational speed than when gas viscosity is not considered. To minimize this effect, a wide path should be provided for the gas, without narrow cross sections or abrupt changes of direction which would restrict the gas flow. The upper part of the tank is at a higher pressure than the reactor inlet. Because of this pressure differential the gas expands in the connecting pipe and its speed increases from the tank to the reactor. This has the same effect as a change in viscosity. The effect of gas viscosity and compressibility is shown in FIG. 6. The theoretical curves are shown in broken lines so that the deviation therefrom may be seen. When the volume flow rate of the gas is below the theoretical level, the volume flow rate of the liquid is above the theoretical level so that the total volume flow rate is the same.

Thus, the volume rate of gas sucked in increases essentially linearly with the impeller rotational speed. The inside volume of the reactor V_R is constant and when the reactor is considered at a given instant when filled with a given volume V_G of gas and V_L of liquid,

$$V_R=V_G+V_L$$

In FIGURE 5:

$$C_B=Q_o$$

$$B_A=Q-Q_o$$

If t is the time required for the reactor to suck in the volume V_L of the liquid, then

$$V_L=Q_o\Delta t$$

During the time Δt, the volume of gas sucked in is

$$V_G=(Q-Q_o)\Delta t$$

Accordingly, the gas-liquid ratio will be:

$$\frac{V_G}{V_L}=\frac{Q-Q_o}{Q_o}=\frac{B_A}{C_B}=\frac{n-n_o}{n_o}$$

Thus,

$$V_L = V_R \cdot \frac{n_o}{n}$$

where:

V_L is the volume of liquid in the reactor;
V_R is the total volume of the reactor;
n_o is the minimum impeller rotation speed which will cause suction on the gas;
n is the actual rotational speed of the revolving device.

This equation defines the liquid volume in the reactor in relation to the speed n, and a plot of the values is a hyperbola as in FIGURE 7. Of practical interest is that part of the curve when $n > n_o$. For a speed n in a reactor of capacity AB, the volume of gas is DB and the volume of liquid is AD (FIG. 7). The gas-liquid ratio has very small variations. This curve will help in the study of a reaction in the system. Up to this point, only the problem of using a gas and a liquid which do not react has been treated. What happens will now be explained when, owing to the reaction with the liquid, gas is consumed.

The reactor intimately mixes the gas with the liquid. Due to reaction, a part of the gas is absorbed into the liquid causing a reduction of pressure inside the reactor. This reduction of pressure is essentially transmitted only to the inlet of the reactor and thus will augment the gas suction by the driven means (e.g., impeller) in the reactor. This further decrease of pressure will be balanced by the suction of an increased volume flow rate of gas. When reaction occurs, the total volume of gas sucked in is higher than the volume which would be sucked in by the driven means alone, the difference being the volume of gas which reacted.

FIGURE 7 shows the gas-liquid ratio in the reactor for $n > n_o$. DB represents the volume of gas (V_G) sucked in by the action of the impeller when liquid volume V_L, represented by AD, is admitted. As a volume of gas V_a reacts, an increased gas volume $V_a + V_G$ will be sucked in.

Considering a catalytic reaction (i.e., a catalyst is assumed to be dispersed in the liquid) the volume of reacted gas V_a is proportional to the number of molecules of liquid able to react and so to the liquid volume V_L. That is,

$$V_a = k_1 V_L$$

where k_1 is a constant. V_a is, likewise, proportional to the number of gas molecules and so to the gas volume V_G. That is,

$$V_a = k_2 V_G$$

where k_2 is another constant. Accordingly,

$$V_a = k V_G V_L$$

where $k = k_1 k_2$. The value of the constant k depends on the nature of the reactants and the size and configuration of the reactor.

The reaction velocity v in a given reactor can be expressed by the volume of combined gas per unit time. (Δt is the time interval during which volume of gas V_a is reacted).

$$v = \frac{V_a}{\Delta t} = \frac{k V_G V_L}{V_L / Q_o} = k Q_o V_G$$

Since,

$$V_G = V_R - V_L = V_R \left(1 - \frac{n_o}{n}\right)$$

$$v = k Q_o V_R \left(1 - \frac{n_o}{n}\right)$$

It is found that reaction velocity v varies with impeller speed n according to a ratio such as

$$\frac{n - n_o}{n}$$

so k is independent of impeller speed n. Accordingly,

$$V_a = k V_G V_L = k V_R^2 n_o \left(\frac{n - n_o}{n^2}\right)$$

This equation gives, for each value of impeller speed n, the volume V_a of reacted gas. It is represented by a third degree curve (FIG. 8) of which the part of practical interest is the interval $n - n_o$. V_a reaches a maximum value when $n = 2n_o$. When

$$V_L = V_G - \frac{V_R}{2}$$

V_a is at its maximum value.

On the FIG. 8 curve, there is a point of inflection at $n = 3n_o$ beyond which the volume of reacted gas V_a slowly decreases as n increases. Transposing this curve onto FIG. 7 we get a graph showing operating range of the reactor as n varies (FIG. 9). Point P_o in FIG. 8 corresponds to point A_o in FIG. 7. In FIG. 9, for a given value of n, during the time interval Δt: AD is the liquid volume through the reactor; DB is the gas volume going out of the reactor; DC is the gas volume going into the reactor; BC is the volume of gas which is reacted. FIG. 10 is the same graph as FIG. 9 but for volume flow rates. In FIG. 10: AD is the liquid volume flow rate through the reactor; DB is the gas volume flow rate going out of the reactor; DC is the gas volume flow rate going into the reactor; BC is the volume rate of reaction of gas.

In describing reaction of the gas, for illustrative purposes it has been indicated that a catalyst is present. If the reaction is conducted without a catalyst, while the general principles hithertofore described will still apply the reaction velocity will decrease rather than remain constant as when a catalyst is used. When a catalyst is used, since $V_a = k_1 V_L$, where k is a constant and V_L, the volume of liquid entering the reactor in the time interval Δt, is constant too, accordingly, the reaction velocity v remains constant. This is not true for reactions without a catalyst. When no catalyst is used, as the reaction proceeds in the given liquid volume V_L the number of molecules able to react is continuously decreasing, and cannot be proportional to the given volume V_L. The reaction velocity is continually decreasing. However, when catalyst is used, i.e., in the case of catalytic reactions, and, typically, a large excess of liquid beyond the stoichiometric requirements is used, the catalyst makes available for reaction with unreacted molecules of the gas a constant quantity of unreacted molecules of the liquid. Then, until, near the very end of the reaction, when even the catalyst cannot make available enough unreacted molecules of liquid for reaction with unreacted molecules of gas to maintain the reaction velocity, $V_a = k_1 V_L$. Thus, when catalyst is used, it can be concluded that there are two parts to the reaction: a first part with a constant reaction velocity; the latter part where reaction velocity gradually slows down. However, practical results fit the main constant velocity part.

As explained above, reaction velocity v in a given reactor at any time can be expressed by the following equation:

$$v = k V_R Q_o \left(1 - \frac{n_o}{n}\right)$$

Accordingly, v can be graphically illustrated as a function of impeller speed n (FIGURE 11). It can be seen from that curve that the reaction velocity, increasing from n_o, tends to an asymptotic value $k V_R Q_o$ as n increases. Thus the reaction velocity v remains essentially constant. For instance, when the impeller speed n increases from $4n_o$ to $8n_o$, the FIG. 11 curve would show that the reaction velocity v is increased by only 14%. This is based on the hypothesis that the volume of gas sucked in by the action of the impeller is a linear function of rotational speed n; it is found that actual results do

not differ significantly from the results predicted from this hypothesis. In fact, for high values of rotational speed *n*, the gas-liquid ratio is little different from the theoretical values in FIG. 7, which must be corrected as shown in FIG. 12. The solid line in FIG. 12 is a curve of actual results and the broken line is the theoretical curve as in FIG. 7. The curve of actual results tends to a horizontal straight line and the volume of gas sucked in by the action of the impeller tends to a constant value. When reaction occurs, the consumption of gas by reaction causes additional gas to be sucked in and the total volume rate of gas sucked into the reactor is greatly increased without any change of rotational speed *n*.

Referring further to FIG. 2*a*, the construction of a particular apparatus embodiment according to the invention is shown. The liquid and gas to be reacted are introduced into a tank T. For this purpose, the tank T is equipped, for example at its top, with closable inlets, e.g., valved conduits (not shown). Driving means **16** (a conventional motor and transmission means) is started and thereby impeller stages **14** and **14'** are rotated. Valved gas inlet conduit **11** is opened. As shown, the discharge end of conduit **11** is located at the suction side of impeller stage **14**.

The two impeller stages **14** and **14'** of reactor R are similar to the two stages of a two-stage centrifugal pump. It is seen that the discharge end of a conduit **11** is located at the suction side of the second stage. Similarly, a reactor having only one impeller stage or more than two impeller stages may be used, provided the discharge end of condit **11** be disposed at the suction side of an impeller in the reactor. In the same manner, a plurality of such conduit discharge ends may be so disposed, at the suction side of one or more impeller stages in the reactor.

Conduit **11** communicates with a supply of the gas to be reacted (not shown). The rotation of the impeller draws liquid from tank T through the reactor R. A suction is created in conduit **11** and the gas is drawn through the conduit **11** into the reactor where it is mixed with the liquid and where reaction takes place.

The reaction product and any unreacted liquid and gas may be discharged through a valved conduit **12** or may be partly or completely recirculated to the tank through the conduit LSG. The conduit LSG is provided with a needle valve **10**. Needle valve **10** would be set to an open position for recirculation. Needle valve **10** may be set at intermediate positions of opening to control the magnitude of recirculation. The purpose of recirculation is to complete reaction of liquid and gas which is not completed in one pass through the reactor.

A conduit G provides an additional supply of gas to the reactor. The conduit G communicates between the gas space in the upper portion of the tank and the suction side of the reactor. Gas is sucked into the reactor from the gas space, through the conduit G. The conduit G is provided with a valve **13**, by means of which gas flows through the conduit G may be regulated or completely cut off. The conduits **11** and G may both be used, as shown, or only one or the other may be used; thus, the invention includes apparatus with only one of these conduits.

A valved conduit **15** is provided as an auxiliary means which may be used to reduce pressure in the tank T. Thus, if the pressure should become too high, gas may be bled to the atmosphere from the tank through the valved conduit **15**. The apparatus may be provided with pressure, temperature, flow rate measuring means and the like (not shown). Heating and/or cooling means (not shown), such as coils for circulating heating or cooling fluid, may be provided in the tank or other parts of the apparatus. The auxiliary valved conduit **15**, in addition to being a means for decreasing pressure, may be used to vent the tank at the end of the reaction.

The apparatus of the present invention may be used to conduct a wide variety of reactions involving a liquid reactant and a gaseous reactant and, if desired or necessary, a catalyst. The invention involves the physical principles discussed above, regardless of the chemical identity of the reactants. Thus, according to the present invention, there may be reacted, for example, ethylenically unsaturated organic compound in liquid form with hydrogen gas, whereby the hydrogen gas reacts with the organic compound at its points of unsaturation to decrease or eliminate the unsaturation. The expression "in liquid form" is intended to indicate that the material so designated is a liquid in and of itself or is dissolved, dispersed or emulsified in a liquid solvent or carrier. The ethylenically unsaturated organic compound may, for example, be an ethylenically unsaturated fat such as stearin and the like, unsaturated fatty oil such as fish oil, castor oil and the like, an ethylenically unsaturated fatty alcohol such as oleyl alcohol and the like, an ethylenically unsaturated aliphatic hydrocarbon such as squalene and the like, or an ethylenically unsaturated dicarboxylic acid such as maleic acid and the like. Also, according to the invention, a phenol, such as aminophenol, in excess strong alkali may be reacted with carbon dioxide to form a salicylic acid. Furthermore, according to the invention, an ethylenically unsaturated compound may be reacted with ammonia to add the ammonia across the double bonds of the ethylenically unsaturated compound to form primary amino groups. Also, according to the invention, an aldehyde in liquid form may be reacted sequentially with ammonia and then hydrogen to form an amine. And, according to the invention, a solution of potassium carbonate may be reacted with carbon dioxide to form potassium bicarbonate. Also according to the invention, the liquid may be a solution of a sugar, such as dextrose, and the gas may be hydrogen, whereby the sugar is reduced. Still further according to the invention halogenations of liquids reagents may be carried out; and ethylene oxide reactions (e.g., etherifications, esterifications, reactions with amines and, in general, reactions with any organic compound having a labile hydrogen atom) with liquid reagents, the ethylene oxide being in gaseous form, may be carried out. These are general examples, and the invention will now be further described by reference to the following specific examples.

EXAMPLE 1.—HYDROGENATION OF CASTOR OIL

A mixture of 3 liters castor oil and 10 g. active Raney nickel is placed in an apparatus as illustrated in FIG. 2*a*, except provided with heating-cooling coils and wherein a single impeller stage rather than two is contained. Tank T has a capacty of 5 liters and the impeller is rotated at 2800 r.p.m. by a 1.5 H.P. motor. The apparatus is started up under a hydrogen pressure of 3 kg./sq. cm. and heated slowly to 100° C. Absorption of hydorgen becomes very noticeable and increases rapidly with the temperature. Because of the heat liberated by the reaction, external heating can be reduced and then stopped. When the temperature reaches 160° C., cooling with water is begun in order to keep the temperature below 180° C. The cooling is stopped when the temperature ceases to rise. Absorption of gas falls off rapidly and stops after 50 minutes. The machine is allowed to run while the system is cooled to 100° C. After venting, the catalyst is filtered off. The hydrogenated oil obtained melts at 80° C.; its iodine index being 0.7.

EXAMPLE 2.—STEARIN

A mixture of 450 kg. triple-pressed stearin (iodine index, 2.7) and 2 kg. active Raney nickel is placed in an apparatus as in Example 1, except tank T has a capacity of 700 liters and the impeller is rotated at 1400 r.p.m. by a 20 H.P. motor. The apparatus is started up under a hydrogen pressure of 3 kg./sq. cm. The mixture is heated to 140° C. and kept at this temperature for 40 minutes. After cooling to 80° C., venting, and filtering the nickel, a stearin

(iodine index, 0.012) is obtained containing only very small amounts of olefin.

EXAMPLE 3.—FATTY ALCOHOL

450 kg. of lauryl alcohol, containing a small amount of unsaturated fatty alcohols, is placed in an apparatus as in Example 2. (This mixture was obtained by high-pressure catalytic hydrogenation of coconut oil. Its iodine index is 12.) 2 kg. active nickel is added. The hydrogen pressure is 3 kg./sq. cm. The reaction is carried out at 120° C. for 35 minutes. The hydrogenated alcohol obtained after cooling and filtering off the nickel has an iodine index of 0.1 and may be sulfonated without appreciable discoloration.

EXAMPLE 4.—VITAMIN OIL

3 liters of fish oil of very disagreeable odor, enriched with vitamin A (500,000 units), is mixed with 10 g. of active Raney nickel and placed in an apparatus as in Example 1. The mixture is treated with hydrogen under a pressure of 4 kg./sq. cm. at a temperature accurately regulated at 70° C. for 45 minutes. Since the low reaction temperature prevents decomposition of the vitamins, the vitamin A content of the product is unchanged. The product has only a very slight odor and its iodine index has fallen from 170 to 116.

EXAMPLE 5.—SQUALENE

A mixture of 3 liters squalene (iodine index 372) and 15 g. active Raney nickel is placed in an apparatus as in Example 1 and heated under a hydrogen pressure of 2 kg./sq. cm. At 60° C. the absorption of the gas is considerable and the evolution of heat is so great that the mixture must be cooled rapidly to prevent the reaction from getting out of hand. After 40 minutes, the cooling is gradually reduced and the temperature is allowed to rise slowly. The reaction is finished at 150° C. The total reaction time is 1 hour 45 minutes. The iodine index of the final product is 0.2. Its molecular weight, determined cryometrically, is 420. It is practically pure squalene. It should be noted that, in order to avoid the formation of polymers which would render the product unusable, it is essential to carry out the reaction for the most part at a low temperature.

EXAMPLE 6.—AMINOPHENOL—CO_2

45 kg. aminophenol dissolved in excess caustic potash solution is placed in an apparatus as illustrated in FIG. 2*a*, except provided with heating-cooling coils. Tank T has a capacity of 200 liters and the impeller is rotated at 1400 r.p.m. by an 8 H.P. motor. Conduit 11 is connected with CO_2 cylinders. The apparatus is started up. The gas is absorbed very quickly and the temperature rises. When absorption ceases, the pressure is allowed to rise to 7 kg./sq. cm. The temperature is raised to 100° C. and kept at this point for 2 hours. After cooling, venting, and acidifying to pH 3, 43 kg. of crude p-aminosalicylic acid is obtained.

EXAMPLE 7.—ASPARTIC ACID

A solution of 1 kg. maleic acid in 3 liters water is placed in an apparatus as in Example 1. Ammonia gas is introduced while the impeller is rotated until a pressure of 10 kg./sq. cm. is reached. The temperature is kept at 150° C. for 1 hour by heating. The reaction mixture is cooled and vented, the excess ammonia is removed by distillation under reduced pressure, and the residue is acidified to pH 2.8. 800 g. crude aspartic acid is thus obtained, which can be recovered in very pure form by a single crystallization. The yield (70%) was further improved by using all the stages of a four stage impeller.

EXAMPLE 8.—SUCCINIC ACID

Two small reactors are used in series. The first reactor is a two-stage unit having a capacity of 2.7 liters and driven by a 3 H.P. motor at 1400 r.p.m. and the second reactor is a four-stage unit having a capacity of 1.5 liters and driven by a 5 H.P. motor at 2800 r.p.m. Each is connected to a hydrogen gas source and takes bydrogen gas at the suction side of the first stage. The use of this series arrangement obviates the use of recirculation means since the reaction is completed in one pass through the two reactors. The tank has a capacity of 50 liters. In the tank is placed 30 liters of a dispersion containing 8 kg. of maleic acid and 50 g. of 5% palladium on alumina. The catalyst is kept in suspension by means of an agitator in the tank. The reactors are operated at a throughput of 30 liters/hour and are cooled by coils containing circulating water. A cooler is placed at the outlet of the system. The reaction product, after filtering out the catalyst, yields 230 g. per liter of very pure succinic acid. On evaporation of the mother liquor, a 99.2% yield of succinic acid is obtained.

EXAMPLE 9.—POTASSIUM BICARBONATE

An apparatus as in Example 1 is used. A solution of 1.5 kg. caustic potash in 3 liters water is placed in the tank and carbon dioxide is led into the reactor. Very rapid absorption takes place with considerable evolution of heat. The reactor exit valve is opened at intervals and the pH of the reaction product is checked. In this way, it is possible to arrive at a yield of 10 liters/hour of a concentrated solution of potassium bicarbonate which crystallizes on cooling and may be recycled after addition of potassium hydroxide. The pH of the product leaving the reaction vessel indicates when the reaction is complete.

EXAMPLE 10.—HEPTYLAMINE

A mixture of 912 g. enanthal, 2 liters ethanol and 10 g. active Raney nickel is placed in an apparatus as in Example 1. The apparatus is started up and ammonia gas is passed in until the pressure reaches 0.3 kg. Hydrogen is then introduced and hydrogenation is carried out under a pressure of 5–10 kg. per sq. cm. at a temperature below 70° C. After 1 hour 40 minutes, absorption of hydrogen ceases. The gas is vented, the catalyst filtered out, the alcohol distilled off, and the heptylamine fractionated. Crude enanthal gives a 75% yield of pure heptylamine, the remainder of the product consisting of other lighter (18%) and heavier (7%) amines.

EXAMPLE 11.—SORBITOL

The apparatus used in this test is similar to that described in Example 8 except that the second reactor, instead of exhausting to the open air is connected to the closed 50-liter feed reservoir for recirculation. The reaction mixture is obtained by dissolving 10 kg. dextrose in 30 l. water. The catalyst consists of 1000 g. active Raney nickel. The initial hydrogen pressure is adjusted to 8 kg./sq. cm. The temperature is 100° C. The amount of hydrogen added is so regulated as to maintain a constant pressure. After 30 minutes, analysis of a sample shows 1.84% of unconverted reducing sugars. The proportion falls to 0.20% after 45 minutes and is not observable after 1 hour. The acidity and color show no appreciable change. After concentration of the filtered liquid, sorbitol is obtained in almost quantitative yield.

EXAMPLE 12.—2-METHYL-PENTANAL

A mixture of 2 kg. 2,2-dimethyl-pentanal, 500 cc. water, 15 g. caustic soda, and 10 g. active Raney nickel is placed in an apparatus as in Example 1. The hydrogen pressure is adjusted to 3 kg./sq. cm. and the apparatus is started up. The reaction mixture is strongly cooled to keep the temperature at 30° C. This is essential in order to protect the aldehyde group which begins to react with hydrogen under the same conditions at about 50° C. After 1 hour 10 minutes, absorption ceases. The catalyst is separated and the 2-methyl-pentanal obtained is decanted and vacuum-distilled. The yield is 98% of theoretical.

The invention is not to be construed as limited to the particular forms disclosed herein, since these are to be regarded as illustrative rather than restrictive.

What we claim and desire to secure by Letters Patent is:

1. A reactor for reacting together first and second fluids comprising a container for said first fluid, means defining a reaction chamber having an intake and discharge and a passageway connecting said container with the intake of said reaction chamber for flow of said first fluid from said container to said reaction chamber under a pressure head, an impeller rotatable in said reaction chamber to draw fluid into said reaction chamber and to discharge it therefrom while producing marked changes in velocity, pressure and direction of fluid passing through said chamber, means in said passageway metering the flow of fluid from said container to said reaction chamber under said pressure head to a volume flow rate Q_o, an inlet for admitting said second fluid between said metering means and said impeller on the intake side of said impeller and means for driving said impeller at a speed to produce flow of fluid through said reaction chamber at a volume flow rate Q which is substantially greater than said flow rate Q_o, said second fluid being thereby drawn into said reaction chamber with said first fluid and intimately contacted therewith by the velocity, pressure and direction changes effected by the rotation of said impeller.

2. A reactor according to claim 1, in which said first fluid is a liquid and said second fluid is a gas, and in which both said liquid and said gas are contained in said container with an interface between them, said passageway connecting with a lower part of said container below said interface to conduct said liquid to said reaction chamber and a second passageway connecting an upper part of said container above said interface to said inlet to conduct said gas to said reaction chamber.

3. A reactor according to claim 2, in which a third passageway connects the discharge of said reaction chamber to said container.

4. A reactor according to claim 3, in which valve means in said third passageway controls the flow of fluid from said reaction chamber to said container.

5. A reactor according to claim 2, in which said container is higher than said reaction chamber, whereby said pressure head is provided at least in part by gravity.

6. A reactor according to claim 1, in which said passageway extends downwardly from said container and in which said reaction chamber is below said container with the axis of said impeller vertical in line with said passageway.

7. A reactor according to claim 1, in which said inlet for said second fluid opens directly into the intake of said reaction chamber.

8. A reactor for reacting together a plurality of fluids comprising a fluid container, means defining first and second reaction chambers each having an intake and discharge, the discharge of said first reaction chamber communicating with the intake of said second reaction chamber, a first fluid passageway connecting said container with the intake of said first reaction chamber, an impeller rotatable in each of said reaction chambers to draw fluid into the respective reaction chamber and to discharge it therefrom while producing marked changes in the velocity, pressure and direction of fluid passing through said chamber, a second fluid passageway communicating with the intake of said first reaction chamber for introducing a second fluid into said first reaction chamber on the intake side of said impeller in said first chamber and a third fluid passageway communicating with the intake of said second reaction chamber for introducing fluid into said second reaction chamber on the intake side of said impeller in said second chamber, said fluids being intimately contacted with one another in said reaction chambers by the velocity, pressure and direction changes effected by the rotation of said impellers.

9. A reactor according to claim 8, in which said impeller in said first and second reaction chambers are on a common shaft.

10. A reactor according to claim 8, in which a fourth fluid passageway connects the discharge of said second reaction chamber with said container.

11. A reactor according to claim 10, in which said fluids comprise a liquid and a gas both in said container with an interface therebetween, said first passageway communicating with said container below said interface and one of said second and third passageways communicating with said container above said interface.

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