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(54) METHOD FOR CONDUCTING CHEMICAL REACTIONS IN FLUID MEDIA

(54) METHODE D'EFFECTUER DES REACTIONS CHIMIQUES DANS DES VEHICULES LIQUIDES

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ABSTRACT:

CLAIMS: Show all claims

*** Note: Data on abstracts and claims is shown in the official language in which it was submitted.

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This invention relates to a new and improved method for conducting chemical reactions and other treatments in homogeneous fluid reaction media. A particular aspect of the invention relates to a method for conducting homogeneous fluid phase reactions which have appreciable endothermic or exothermic heats of reaction. Another particular aspect relates to a method for conducting wall catalyzed fluid phase reactions. More specific aspects of the invention relate to methods for conducting homogeneous gas phase and homogeneous liquid phase reactions.

In carrying out homogeneous fluid phase reactions it is usually desired, as in heterogeneous phase reactions to maintain the reactant or reactants under the reaction conditions for a definite, uniform period of time and to maintain the reaction conditions of temperature and pressure substantially uniform and constant throughout the reaction zone. In carrying out most homogeneous fluid phase reactions which do not take place at a very rapid rate, it is therefore a common practice to pass the homogeneous reaction fluid through a large reaction zone. While such reaction zones are suitable for many reactions having small heat effects, they are relatively inefficient when the heat of the reaction is appreciable.

The object of the invention is to provide a new and improved method for conducting various homogeneous fluid phase reactions. A more particular object of the invention is to provide a method for conducting a homogeneous fluid phase reactions whereby the temperature at which the reaction takes place may be more accurately and uniformly maintained. A further more particular object of the invention is to provide a method for effecting homogeneous fluid phase reactions in which the residence time of the homogeneous fluid reactant phase may be made suitably long without resort to an abnormally large or complicated reaction chamber and with little or no pressure drop and no plugging or channelling in the reaction chamber.

These and other objects which will be apparent from the following description are realized according to the process of the invention by passing the fluid reactant(s) as a single phase under the desired conditions of temperature and pressure and at a space velocity below that giving turbulent flow through an elongated reaction zone substantially defined by two spaced substantially smooth surfaces, which surfaces are maintained in relative motion to one another parallel to an element of one of said surfaces. As an example to illustrate the above, the reaction is carried out by passing the reactant(s) through a reaction zone which is the annular zone between two relatively smooth concentric cylinders, the outer cylinder being stationary and the inner cylinder being rotated at a constant angular velocity about its axis.

When a fluid is passed through such a reaction zone with the walls thereof stationary, the fluid flows therethrough in laminar, linear or streamlined flow. It is found, however, that if the substantially smooth walls substantially defining such a reaction zone are maintained in relative motion to one another in a direction of an element of the moying surface, the flow characteristics are markedly altered; more or less regular eddy currents are set up which cause the fluid to traverse the reaction zone in a very tortuous but more or less orderly path with frequent and more or less regular contact with the confining walls. Thus, by conducting homogeneous fluid phase reactions under such conditions, the residence time of each increment of reaction fluid is more or less uniform and there is exceptionally large contact with the confining walls of the reaction zone. Since the reaction zone is devoid of solid material, pressure drop, plugging and channelling are substantially eliminated. The system is particularly advantageous for effecting homogeneous fluid phase reactions which are catalyzed by the walls of the reaction chamber, since by this method the walls may be easily maintained at exactly the desired temperature and at the same time orderly and uniform contact of the fluid reactant(s) with the walls may be obtained.

The surfaces mentioned in the previous paragraph are relatively smooth surfaces, by which is meant surfaces which are devoid of paddles, vanes and/or other such protuberances which cause random mixing. Preferred surfaces are relatively smooth to the touch as an ordinary or polished metal surface or a cloth surface. The surface is preferably substantially continuous, but it may be perforate. If perforate, however, the actual area of surface should predominate over the area of the perforations as is the case, for example, in finely woven metal or fiber glass screens. The surfaces may be of metal such as stainless steel, monel metal, brass, nickel, aluminum, cadmium or may be of other materials such as glass, rubber, wood, plastic, porcelain, cloth, enamel, carbon or a coating of catalytic material.

The shape of the surface may vary. In general, the contour of the surface is preferably smooth or streamlined, particularly in the direction of relative motion. Examples of suitable surfaces are the surfaces of cylinders, cones, double cones, spheres, and the like, produced by the rotation of straight or curved lines about suitable axes which are parallel or at an acute angle thereto. In the application of such surfaces of revolution, the reaction zone is the annular zone between two such surfaces. The axes of the two surfaces of revolution are generally concentric and parallel, but they may be displaced and/or inclined to each other at an acute angle.

The respective surfaces do not touch and are spaced apart at a suitable distance to provide a cross-section sufficient to provide the desired throughput capacity with the desired space velocity corresponding normally to non-turbulent flow of the fluid reactant(s), i.e. the flow at the space velocity in question would be non-turbulent if the surfaces were stationary. Thus, for example, in a typical reactor for effecting reaction in a homogeneous liquid medium utilizing the annular space between two cylinders, the inner cylinder may be 20 feet long by 3 feet diameter, and the outer cylinder may be 28 feet long by 4 feet diameter, thus providing an annular space 6 inches wide. For effecting a reaction in a homogeneous gas or vapor phase, the inner cylinder may be 26 feet long by 3 feet, 6 inches diameter, thus providing an annular space 3 inches wide.

The described surfaces are maintained in relative motion to one another in a direction of an element thereof. Thus, one surface, usually the outermost, may be stationary and the second surface moved with respect thereto at a desired distance therefrom by rotation about its axis, or both surfaces may be rotated in the same or in opposite directions such that there is a difference in rotational absolute angular velocity between points on the two spaced surfaces. In most cases it is preferred that the inner of the two surfaces rotate at a greater peripheral speed than the outer of the surfaces and the simplest and the preferred method of accomplishing this is to maintain the outer surface stationary and rotate the inner surface.

In the case of surfaces of revolution, the path of the fluid reactant through the reaction zone between the surfaces (considering the surfaces stationary) is parallel to the axis of the surfaces. However, the actual path of the reactant(s) when the surfaces are in relative motion is spiral shaped due to the effect of the rotating surfaces.

As pointed out, the fluid reaction mixture is passed through the reaction zone at a desired space velocity which would afford a non-turbulent flow if the surfaces were stationary, i.e. in the case of liquids at a flow rate corresponding to a Reynolds Number for flow through a conduit below about 2500. By suitable relative motion of the surfaces, as described, a characteristic condition of flow is produced having orderly or regular eddy currents, which type of flow is neither laminar flow nor turbulent flow. This flow condition produces a uniform or orderly distribution and mixing of the homogeneous reactant phase which is particularly desirable for effecting reactions of the type in question. The amount of relative motion (i.e. the relative linear velocity of points on two opposing spaced surfaces substantially defining the reaction zone) necessary to produce this desired described condition of flow (eddy flow) depends upon the distance between the surfaces, the radius of curvature of one of the surfaces, the density and viscosity of the fluid reaction mixture and the feed rate. In the exceptional cases where the feed rate, with surfaces stationary, is in the narrow region of indeterminate flow between laminar flow and turbulent flow (a narrow critical region which, in the case of liquid, corresponds to Reynolds Numbers between about 2000 and 2500) the relative motion of the surfaces required to produce the eddy flow may be quite small; on the other hand, in the special case where the liquid flow rate approaches zero, the minimum relative velocity of the surfaces is defined approximately by the following formula:

$$V = \frac{100\mu}{\rho L}$$

wherein V is the relative velocity of points on the two spaced surfaces in ft./sec., μ is the viscosity of the liquid in the reaction zone expressed in lb./ft./sec. units; ρ is the density of the liquid in the reaction zone in lb./cubic foot; and L is a scale factor which varies with the apparatus. For instance, L is the distance between the surfaces, in feet, for the case of liquid reactants between parallel surfaces less than one foot apart or for the case of coaxial cylinders when the inner cylinder is rotated and the ratio of the radii of outer to inner surfaces is two and the annular distance between the surfaces is less than six inches. The viscosity of the fluid reactant depends upon the temperature and the composition. As the velocity of relative motion of the surfaces is increased beyond that required to establish the desired eddy flow, the degree or extent of contact of the fluid reaction mixture with the reactor walls is increased until, finally, at very high velocities a condition of turbulent flow is produced. Such turbulent flow is not desired since it results in much less efficient contact. Thus, under conditions of turbulent flow, some portions of the reacting fluids obtain excellent or excessive contact or resistance whereas others receive insufficient contact or residence. Ordinary mixing, as by blowers, fans, etc., results in a condition similar to turbulent flow. The relative linear velocity of the reactant(s) is therefore maintained below that giving turbulent flow. It should be pointed out, however, that in the type of reaction systems described where the surfaces are preferably smooth, as polished metal, quite high linear velocities may be used without destroying the desired orderly eddy flow and this limitation is therefore more of a theoretical nature than a practical one.

The desired condition of eddy flow is produced by the surfaces slipping through the fluid reaction mixture. Factors tending to produce ordinary turbulent mixing or stirring tend to disrupt the desired eddy flow. It is for this reason that smooth surfaces substantially free of sharp irregularities are preferred. Also, for this reason the surfaces are preferably moved evenly in a more or less constant spaced relationship with little vibration. In the absence of disturbing factors the desired eddy-type of flow may be produced with very slow relative motion of the surfaces. However, the contact of the fluid reactant with the confining walls increases as the eddy currents increase in intensity and for this reason the relative movement of the surfaces is preferably adjusted to give strong well-marked eddy currents. Strong well-marked eddy currents are recognizable on sight due to the regular or orderly patterns in the flow produced thereby. In some cases these currents produce very distinctive patterns such as "double doughnuts" or torii stacked axially one on top of the other around the rotating surface, torii spiralled around the rotating surface or a "herringbone" pattern. A condition of eddy flow however does not necessarily result in the formation of such unique patterns.

By adjustment of the relative velocity of the surfaces the degree or efficiency of contact of the homogeneous fluid reactant(s) with the reactor walls and the residence time may be adjusted and controlled within very fine limits to get optimum results. This adjustment may be accomplished by actually changing the rate of movement of one or more of the surfaces or it may be accomplished by adjustment of the shape of the surfaces, i.e. by varying the cross section of the annular zone along its length. This latter method allows the relative velocity of the surfaces to be varied throughout the length of the annular reaction zone to compensate for differences in viscosity or gas volume caused by the reaction.

A typical example of a reactor of the type contemplated is illustrated semi-diagrammatically in the attached drawing. Referring to the drawing, the reactor comprises an elongated cylindrical shell 1 surrounded on the outside by a jacket 2. Shell 1 has suitable top and bottom closures 3 and 4. A cylinder 5 is mounted centrally within cylinder 1 by means of a lower bearing 6, and upper bearing and stuffing box 7 so as to be rotatable about its vertical axis. Cylinder 5 has a length appreciably greater than its diameter and its diameter is such that the annular space between cylinders 5 and 1 is sufficient to pass the required volume of fluid reactant(s) at the desired space velocity without turbulent flow. The upper trunion 10 of cylinder 5 is a hollow tube communicating with the interior of the cylinder. The cylinder 5 is made to rotate within cylinder 1 by means of a variable speed motor 8 driving gear 9 which is attached to trunion 10. Within trunion 10 is a smaller open tube 11 which extends down well within the cylinder. Tubes 10 and 11 are broken and connected through a swivel joint 12.

In operation, a temperature controlling medium, for example hot water, oil, "Dowtherm" or the like, is introduced into the annular space between the cylinder 1, and jacket 2 by means of an inlet 13. This temperature controlling medium is then passed into cylinder 5 via tube 14 and is finally withdrawn via tube 11. The homogeneous fluid reactant(s) is (are) introduced into the annular reaction zone between cylinders 1 and 5 by means of inlet 15 at such a rate that with cylinder 5 stationary the flow is essentially streamline flow. Cylinder 5, however, is caused to rotate at such a rate that the streamline flow is changed to an eddy type of flow. The eddy currents may be observed in trial if desired by charging puffs of smoke, dust or lampblack with the fluid reactant(s). The reaction mixture is withdrawn from the reaction zone via outlet 16.

The process of the invention is applicable for carrying out homogeneous gas phase reactions such as vapor phase chlorinations, hydrobrominations, nitrations and oxidations. It may be also applied, however, for carrying out homogeneous liquid phase reactions such as liquid phase esterifications, saponifications, aminations, isomerizations and alkylations. These and various other types of reactions may be carried out at low, ordinary or raised temperatures and pressures.

The process of the invention is applicable in the absence of a catalyst in which case the homogeneous reaction mixture is introduced into the reaction zone and caused to react by being subjected to reaction conditions of temperature and/or pressure (as for instance in polymerization reactions) or in other cases simply by virtue of the reactants being in contact (as for instance in gas phase chlorinations where vapors of the material to be chlorinated and chlorine are introduced simultaneously and it is desired to effect a partial reaction with a minimum of side reactions). Also the process is applicable for many catalyzed reactions. In such cases the catalytic agent is a homogeneous catalyst except in the case of wall catalyst reactions, in which case the walls of the reaction zone may catalyze the reaction either by virtue of being a solid surface or by virtue of being composed of or coated with a catalytic material.

Thus, in the case of homogeneous catalysts the catalyst is a gas or vapor when operating in the vapor phase and is in solution in the reactant(s) when operating in the liquid phase. Examples of catalytic materials which may be employed in either the gaseous phase or liquid phase in many cases are hydrogen chloride, hydrogen bromide, hydrogen iodide, hydrogen fluoride, bromine, iodine, aluminum chloride, aluminum bromide, boron fluoride, and materials giving free radicals such as tetra ethyl lead.

The process of the invention is not restricted to any particular type or kind of homogeneous phase reaction, but, as pointed out above, it is particularly advantageous for certain types of reactions, namely, 1) reactions that have large heats of reaction (endothermic or exothermic); 2) reactions which require particular careful temperature control; and 3) reactions which may be catalyzed by the walls of the reaction zone. Examples of the first category include dehydrogenation, hydrogenation, polymerization, oxidation, cracking, chlorination, and nitration. Particular examples of the second category are the synthesis of alcohols, aldehydes, ketones and hydrocarbons by the hydrogenation of carbon oxides (the Fischer-Tropsch type syntheses). Examples of the third category include chlorination, bromination, oxidation, dehydrochlorination and hydrogenation.

In such cases where the walls of the reaction zone catalyze the reaction, the walls (for example the outside of cylinder 3 and/or the inside of cylinder 1) may comprise a suitable catalytic material which may be a metal, an alloy, a metal oxide, a metal sulfide or in fact any suitable solid catalytic material.

The process of the invention is particularly advantageous for effecting synthesis of hydrocarbons and/or oxygenated products from hydrogen and oxides of carbon. These various syntheses (referred to herein generally as Fischer-Tropsch type synthesis) are homogeneous in that the reactants and the products are both in vapor state under the synthesis conditions. In order to effect clean-cut synthesis, the contact time, and particularly the temperature, should be carefully controlled. This has been an exceedingly difficult task, however, even when employing the so-called fluidized catalyst technique due to the very large exothermic heat of reaction and the large contraction of gas volume in passing through the reactor. By employing the above-described reaction system with the catalyst comprising the walls of the reaction zone, exceedingly close temperature control may be easily maintained. Also, the contact of the reactant gas with the catalytic surface is large and uniform. The contraction of the gas volume as the reaction progresses may be provided for by employing a reactor of decreasing cross section in the direction of flow, as for example by employing a revolving cone within a larger outer fixed cone and passing the synthesis gas through the annular zone from the base towards the apex. In cases where there is an increase in the gas volume, for instance, the reaction

$$2\ CH_4 + O_2 \longrightarrow 2\ CO + 4H_2$$

the flow can be reversed, i.e. from the apex towards the base. The distance between the surfaces of the cones may also be larger near the base than near the apex to allow for the greater relative motion of the surfaces near the base.

Another particular application of the process and apparatus of the invention is in carrying out light-catalyzed reactions and light-treatments such, for example, as light-catalyzed gas reactions including photobromination, the irradiation of milk, etc. In such cases the temperature control medium may be provided solely in the inner body (cylinder, etc.) and the outer shell may be made of glass or other material transparent to the light of the desired wave length. In this system all portions of the gas or liquid receive substantially equal amounts of irradiation.

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THE INVENTION CLAIMED IS:

1. The method of conducting the hydrogenation of oxides of carbon in a homogeneous vapor phase which comprises passing a gaseous mixture of hydrogen and oxide of carbon under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces of Fischer-Tropsch synthesis catalyst, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, maintaining a condition of eddy flow in the homogeneous vapor mixture passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces and maintaining a substantially uniform temperature throughout said reaction zone by cooling through said catalytic surface.

2. The method of conducting photo-catalyzed reactions in a homogeneous fluid phase which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two relatively smooth surfaces, at least one of which is transparent to light of a catalyzing wave length, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, and maintaining a condition of eddy flow in said reactant fluid passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces while irradiating the reactant fluid in said reaction zone with light of a catalyzing wave length through said transparent surface.

3. The method of conducting catalyzed homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth catalytic surfaces, said reaction zone being free of solids and of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, and maintaining a condition of eddy flow in said fluid phase reactant passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces thereby to obtain large and uniform contact of said fluid reactant with said catalytic surfaces.

4. The method of conducting homogeneous vapor phase reactions which comprises passing the homogeneous reactant vapor under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, and inducing eddy currents in said vapor phase reactant passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces.

5. The method of conducting homogeneous liquid phase reactions which comprises passing the homogeneous reactant liquid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, and inducing eddy currents in said liquid phase reactant passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces.

6. The method for conducting homogeneous fluid phase exothermic reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow and maintaining a condition of eddy flow in said fluid phase reactant passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces while removing the exothermic heat of reaction through the wall(s) of the reaction zone, thereby to maintain a substantially uniform temperature throughout said reaction zone.

7. The method for conducting homogeneous fluid phase endothermic reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, and maintaining a condition of eddy flow in said fluid phase reactant passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces while supplying the endothermic heat of reaction through the wall(s) of the reaction zone, thereby to maintain a substantially uniform temperature throughout said reaction zone.

8. The method of conducting homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through an annular reaction zone between the inner surface of an outer cone and the outer surface of an inner cone having a common axis at the desired space velocity under conditions of non-turbulent flow, and maintaining a condition of eddy flow in said annular reaction zone by rotation of said inner cone about its axis.

9. The method according to claim 8 in which the homogeneous reactant fluid is introduced into said annular reaction zone at the base end of said cone.

10. The method for conducting homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through an annular reaction zone between two relatively smooth surfaced cylinders, said annular reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow, and maintaining a condition of eddy flow in said annular reaction zone by rotation of one of said cylinders about its axis at a relatively constant angular velocity.

11. The method according to claim 10 in which the outer cylinder is stationary and the inner cylinder is rotated.

12. The method of conducting homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces of revolution, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow and inducing eddy currents in said fluid reactant passing through said reaction zone by the rotation of at least one of said surfaces of revolution about its axis.

13. The method of conducting homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces of revolution, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow and maintaining said boundary surfaces in relative motion to one another by rotation of at least one of said surfaces of revolution about its axis.

14. The method of conducting homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow and inducing eddy currents in said fluid phase reactant passing through said reaction zone by maintaining said surfaces in relative motion to one another in a direction parallel to an element of one of said surfaces.

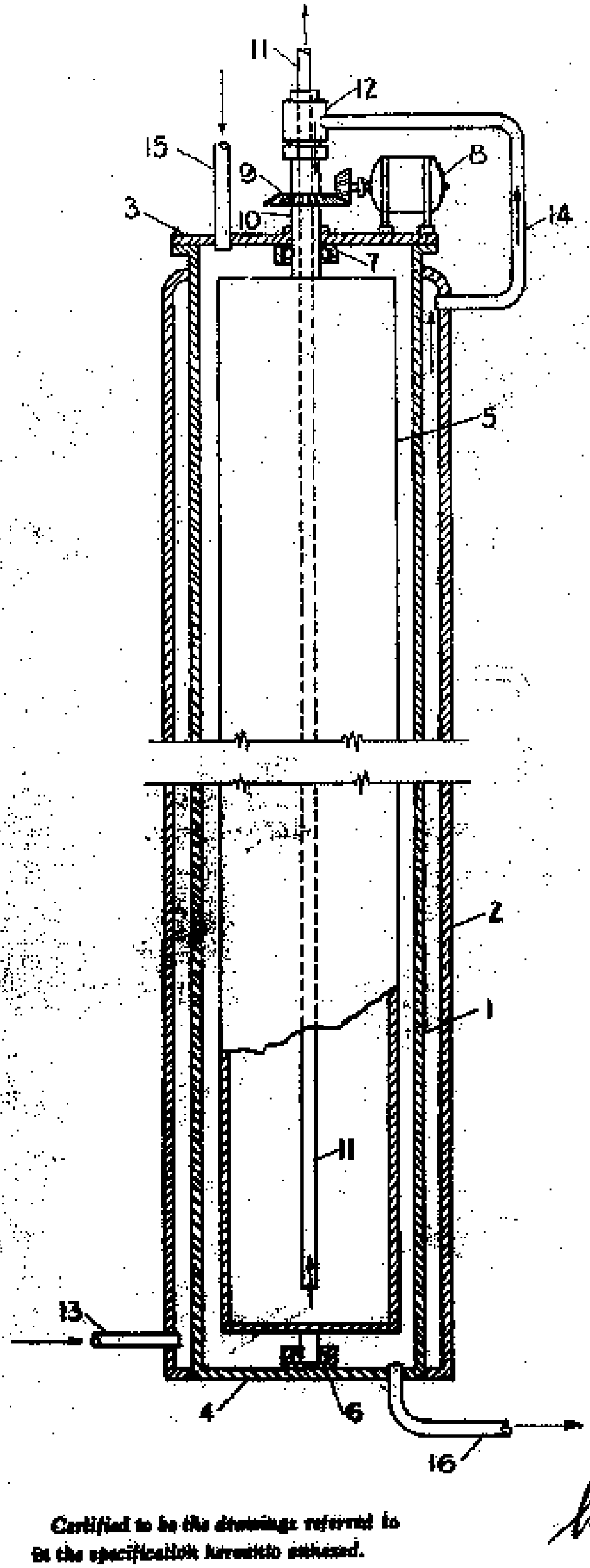
15. The method of conducting homogeneous fluid phase reactions which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure through a reaction zone substantially bounded by two spaced relatively smooth surfaces, said reaction zone being of cross-section and length to accommodate the desired throughput at the desired space velocity with non-turbulent flow and maintaining said boundary surfaces in relative motion to one another by movement of at least one of said surfaces in a direction parallel to an element thereof.

16. A method for conducting a homogeneous fluid phase reaction which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure and under conditions of eddy flow through a reaction zone substantially bounded by two spaced smooth surfaces in relative motion to one another in a direction parallel to an element thereof.

17. A method for conducting a homogeneous fluid phase reaction which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure and under conditions of eddy flow through a reaction zone defined by two spaced relatively smooth surfaces of revolution, which surfaces are maintained in motion relative to one another in a direction parallel to an element of one of them.

18. A method for conducting a homogeneous fluid phase reaction which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure and under conditions of eddy flow through an elongated annular reaction zone substantially defined by two spaced relatively smooth surfaces of revolution, which surfaces are maintained in substantially constant relative angular motion about an axis of rotation.

19. A method for conducting a homogeneous fluid phase reaction which comprises passing the homogeneous reactant fluid under reaction conditions of temperature and pressure and under conditions of eddy flow through an elongated annular reaction zone substantially defined by relatively smooth surfaces of two concentric cylinders, which surfaces are maintained in relative motion to one another by rotation of one of said cylinders about its axis.



INVENTOR

Certified to be the drawings referred to in the specification hereunto annexed.

San Francisco, Calif. Sept. 27 1946