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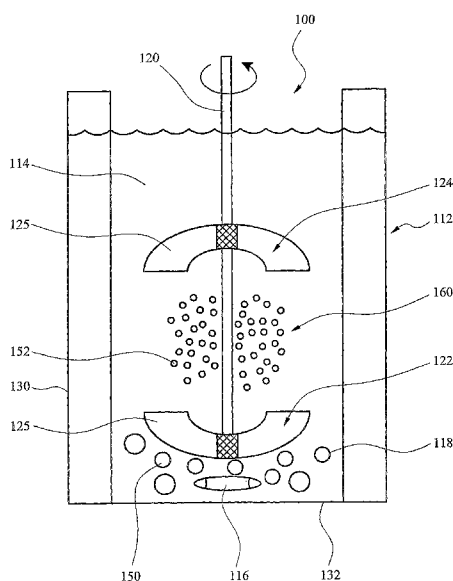
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(54) Title: MIXING APPARATUS



(57) Abstract: A mixing apparatus (100) for mixing at least two fluids, the mixing apparatus (100) comprising a shaft (120) rotatable about its longitudinal axis (121), a first (122) and a second (124) radially extending impeller mounted on the shaft (120) and respectively axially spaced apart, characterised in that the first impeller (122) comprises a plurality of curved blades (125) operable to move said fluids in an axial direction towards the second impeller (124), and the second impeller (124) comprises a plurality of curved blades (125) operable to move said fluids in an axial direction towards the first impeller (122).

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MIXING APPARATUS

The invention relates to a mixing apparatus. In particular, but not exclusively, the invention relates to
5 an apparatus for dispersing gas in liquid.

Many industrial processes incorporate an impeller driven mixing system, for example, processes for fermentation, hydrogenation, chlorination, oxidation, and carbonylation.

10

Impeller driven mixing systems generally incorporate one impeller mounted on a rotating shaft. Such systems may be said to have an axial longitudinal flow parallel to the axis of the rotating shaft and/or a radial flow parallel
15 to the radially extending blades mounted on the shaft. The impeller may thus be a radial flow impeller which projects fluid in a radial direction towards a wall of a chamber in which the impeller is housed, for example, a Chemineer BT-6™ turbine, or alternatively, the impeller
20 may be an axial flow impeller which comprises radially extending blades that are pitched at an angle so as to direct the fluid flow in an axial direction. Examples of axial flow impellers include marine impellers and hydrofoil impellers. Mixed flow systems are known, where
25 the impeller causes flow in both axial and radial directions. An example of a mixed flow impeller is the 45° pitched blade turbine. Such mixing systems can be used in liquid-liquid, liquid-solid, or liquid-gas reactions.

30 A dual impeller system for dispersing a gas in a liquid, in a vessel, is known. Specifically, in a paper by Kuboi, entitled "The Power Drawn by Dual Impeller Systems Under Gassed and Ungassed Conditions" Fourth European Conference

on Mixing, April 27-29 1982, the combination of two axially spaced 45° pitched blade turbines is disclosed whereby a first impeller is mounted below a second impeller on a common shaft. The impeller blades are orientated such that the first impeller projects liquid upwards and outwards and the second impeller projects liquid downwards and outwards. Under gassed conditions, as gas is introduced into the vessel, the first impeller causes the incoming flow of gas bubbles to split so that some of the flow is driven radially outwards towards the walls of the vessel, and some is driven axially upwards towards the second impeller.

The effectiveness of the two impeller system under gassing conditions depends upon the speed of rotation of the impellers. At lower speeds it is possible that the bubbles that have been projected upwards by the first impeller are not affected by the second impeller. The second impeller is unable to overcome buoyancy forces and therefore the second impeller plays no part in the dispersion of gas in the liquid. Only when the rotational speed of the second impeller is increased are the buoyancy forces overcome. Disadvantageously, non-uniform dispersion of the gas in the liquid results. This is shown in Figure 1 of the application. In order to achieve uniform dispersion, further disadvantageously, the rotational speed of the second impeller has to be sharply increased.

It is highly desirable to be able to achieve complete uniform dispersion in an industrial process. In industrial processes where gas/liquid mass transfer is an essential feature, this aids controllability and enhances

reactor performance. In such processes, when the gas is well dispersed, a region of reasonably uniform turbulent

kinetic eddy dissipation can exist in the liquid between
5 and around the impellers which controls coalescence and
breakup of the bubbles. If the gas dispersion is highly
non-uniform, undesirable coalescence can result in a sharp
increase in bubble size and therefore, the surface area
for interface mass transfer to occur is reduced.
10 Advantageously, a controlled turbulent kinetic energy
dissipation field results in a narrow bubble size
distribution over a range of impeller specific powers.

It is an object of the present invention to provide a
15 mixing apparatus which enables controllable mixing of
fluids or solids whilst simultaneously providing an
effective mixing environment.

According to a first aspect of the present invention there
20 is provided a mixing apparatus for mixing at least two
fluids, the mixing apparatus comprising a shaft rotatable
about its longitudinal axis, a first and a second radially
extending impeller mounted on the shaft and respectively
axially spaced apart, characterised in that the first
25 impeller comprises a plurality of curved blades operable
to move said fluids in an axial direction towards the
second impeller, and the second impeller comprises a
plurality of curved blades operable to move said fluids in
an axial direction towards the first impeller.

30

Preferably, the blades on each impeller are inward pumping
into the space between the impellers. In the case of a
substantially vertical shaft, the lower impeller is,

therefore, upward pumping and the upper impeller is downward pumping. Preferably, the blades of each impeller are hydrofoil blades. A suitable hydrofoil blade is the Chemineer Maxflo™ W. Alternatively, the Lightnin A315™, 5 A320™ or A340™ may be used.

Advantageously, due to the opposing axial flows created by the first impeller and the second impeller, a region of high turbulence is observed in a central mixing zone 10 between said impellers. The high turbulence is maintained in this zone, and so there is little variation in the turbulence energy dissipation. Consequently, there is minimal variation in bubble size which results in a narrow size distribution of the bubbles in the central mixing 15 zone. Advantageously, a narrow bubble size distribution enables the process or chemical reaction to be more easily controlled. This region provides a zone where the at least two fluids are brought together to be mixed. A chemical reaction can therefore be facilitated in the 20 central mixing zone. The fluids may be liquid-solid, liquid-liquid, or liquid-gas. Preferably, the at least two fluids comprise a liquid and a gas.

It is advantageous to provide a gas/liquid mixing 25 environment where the bubble size is largely independent of the impeller specific power. In such a system the liquid mixing time may be varied independently of the bubble size.

30 Preferably, the first impeller and the second impeller each comprise two or more curved blades, more preferably, three or more curved blades. Most preferably, are impellers with four curved blades. The provision of an

impeller with a large number of curved blades increases the shear forces acting to break up large bubbles. The small bubbles produced have a smaller average bubble diameter than those produced with a first impeller and/or
5 a second impeller with fewer curved blades and therefore, the available surface area for a reaction to occur is increased.

Preferably, the diameter of the first impeller is the same
10 as the diameter of the second impeller. Preferably, the diameter of the or each impeller is substantially half the diameter of the vessel in which said impeller is mounted.

Advantageously, the smaller the diameter of the impeller,
15 the greater the shear force created for a given power and therefore, a large number of small bubbles are produced which leads to an increase in available surface area for a reaction to occur.

20 Preferably, the axial distance between the first impeller and the second impeller is at least one impeller diameter apart. In this configuration, the turbulence created by the opposing impellers is an equilibrium in the central mixing zone which allows for prediction of bubble size and
25 therefore control of the reaction occurring.

It is preferable that the total power drawn by the combination of impellers is low. Preferably, the impellers operate at a low power number, preferably
30 substantially between 1 to 5, more preferably, substantially between 1 to 3, most preferably substantially 1.75. In so doing, the system consumes less energy than conventional systems which operate at power

numbers of typically 3.2. The power may be measured using conventional equipment, for example, strain gauges.

Preferably, when operating at a low power number complete
5 uniform dispersed phase distribution is achieved. This is highly desirable and is because of the energy efficiency of the blades, preferably hydrofoil blades.

Without being restricted by theory, one possible
10 explanation for the efficiency of the invention is that the use of hydrofoil blades reduces tip vortices and converts more of the shaft energy into flow rather than turbulent kinetic energy, aiding complete dispersion.

15 Preferably, the specific power used when the first impeller and the second impeller rotate is substantially between 50W/m^3 to 900W/m^3 , more preferably, substantially between 100W/m^3 to 800W/m^3 .

20 Preferably, when dual opposed Maxflo type impellers are used in the system, the preferred specific power is substantially between 50W/m^3 to 900W/m^3 . Preferably, when BT-6 type impellers are used, the preferred specific power is substantially between 400W/m^3 to 3200W/m^3 . At such
25 specific powers, a narrow bubble size distribution is maintained and the reaction is controlled.

Preferably, when dual opposed Maxflo impellers are used, the arithmetic mean size (d_{10}) is substantially between
30 $250\mu\text{m}$ to $550\mu\text{m}$ and the surface volume mean diameter (d_{32}) is substantially between $400\mu\text{m}$ to $750\mu\text{m}$. Preferably, when operating at substantially 750rpm , the d_{10} is substantially between $250\mu\text{m}$ to $350\mu\text{m}$, most preferably

substantially $296\mu\text{m}$, and preferably, the d_{32} is substantially between $400\mu\text{m}$ to $500\mu\text{m}$, most preferably substantially $450\mu\text{m}$. Preferably, when operating at substantially 991rpm , the d_{10} is substantially between
5 $300\mu\text{m}$ to $400\mu\text{m}$, most preferably substantially $330\mu\text{m}$, and preferably, the d_{32} is substantially between $460\mu\text{m}$ to $560\mu\text{m}$, most preferably substantially $510\mu\text{m}$. Preferably, when operating at substantially 1200rpm , the d_{10} is substantially between $350\mu\text{m}$ to $450\mu\text{m}$, most preferably
10 substantially $394\mu\text{m}$, and preferably, the d_{32} is substantially between $450\mu\text{m}$ to $550\mu\text{m}$, most preferably substantially $500\mu\text{m}$.

Preferably, when BT-6 type impellers are used, the d_{10} is
15 substantially between $250\mu\text{m}$ to $1500\mu\text{m}$. Particularly, when operating at substantially 251rpm , preferably the d_{10} is substantially between $550\mu\text{m}$ to $650\mu\text{m}$, most preferably substantially $633\mu\text{m}$, and the d_{32} is preferably substantially between $800\mu\text{m}$ to $1000\mu\text{m}$, most preferably
20 substantially $978\mu\text{m}$. Preferably, when operating at substantially 380rpm , the d_{10} is preferably substantially between $800\mu\text{m}$ to $900\mu\text{m}$, most preferably substantially $841\mu\text{m}$, and the d_{32} is preferably substantially between $1000\mu\text{m}$ to $1500\mu\text{m}$, most preferably substantially $1345\mu\text{m}$.
25 At substantially 500rpm , the d_{10} is preferably substantially between $500\mu\text{m}$ to $600\mu\text{m}$, most preferably substantially $597\mu\text{m}$, and the d_{32} is preferably substantially between $700\mu\text{m}$ to $800\mu\text{m}$, most preferably substantially $721\mu\text{m}$. Preferably, when operating at
30 substantially 765rpm , the d_{10} is preferably substantially between $300\mu\text{m}$ to $400\mu\text{m}$, most preferably substantially $378\mu\text{m}$, and the d_{32} is preferably substantially between $400\mu\text{m}$ to $500\mu\text{m}$, most preferably substantially $445\mu\text{m}$.

In a reactor where gas is sparged into an agitated liquid medium, preferably, the sparged gas rate is substantially between 0.05 to 1.0 m³/s, preferably substantially between 5 0.1 to 0.5m³/s, most preferably substantially 0.13m³/s at an impeller speed of preferably between 50rpm to 1200rpm, most preferably substantially 50rpm to 200rpm.

A key parameter used in the design of gas-liquid mixing 10 systems is the critical dispersion speed. This is the minimum impeller speed required to ensure uniform dispersion of the gas bubbles. The critical dispersion speed for achieving dispersion in a dual opposed flow hydrofoil system in a vessel having a diameter of 15 preferably substantially between 1 to 10m, more preferably, substantially between 2 to 5m, is preferably substantially between 1 to 100rpm, preferably substantially between 5 to 50rpm, more preferably substantially between 10 to 20rpm, most preferably 20 substantially 14rpm.

Accordingly, in a further aspect of the present invention there is provided a chemical reaction system comprising a liquid phase and a mixing apparatus according to the first 25 aspect of the invention for mixing a fluid into the liquid phase. Preferably, the fluid is a solid or most preferably, the fluid is a gas.

Preferably, the liquid phase comprises at least one liquid 30 phase reactant for reacting with a gas introduced into the liquid phase, as well as at least one liquid phase reaction product. Preferably, the liquid phase includes a gas introduced therein. Preferably, the said gas

comprises one or more reactants capable of reacting with the said one or more reactants. Preferably, the liquid phase comprises a catalyst system. Preferably, the reaction system is a carbonylation reaction system such as

5 one described in co-pending United Kingdom patent application GB 0516556.8 or as referred to in European patents and patent applications EP-A-0055875, EP-A-04489472, EP-A-0106379, EP-A-0235864, EP-A-0274795, EP-A-0499329, EP-A-0386833, EP-A-0441447, EP-A-0489472, EP-A-

10 0282142, EP-A-0227160, EP-A-0495547, EP-A-0495548, EP-A-1651587, EP-A-1565425, EP-A-1554039, EP-A-1534427, EP-A-1527038, EP-A-1204476, WO2005118519 and WO2005079981.

Preferably, the reaction system is a carbonylation process

15 comprising carbonylating an ethylenically unsaturated compound with carbon monoxide in the presence of a source of hydroxyl groups, preferably, methanol, and a catalyst system comprising (a) a bidentate phosphine, arsine or stibine ligand, and (b) a catalytic metal selected from a

20 group 8, 9 or 10 metal or a compound thereof, preferably palladium. Preferably, the phosphine ligand is selected from 1,2-bis-(di-tert-butylphosphinomethyl)benzene, 1,2-bis-(di-tert-pentylphosphinomethyl)benzene, 1,2-bis-(di-tert-butylphosphinomethyl)naphthalene,

25 1,2-bis(diadamantylphosphinomethyl)benzene, 1,2-bis(di-3,5-dimethyladamantylphosphinomethyl)benzene, 1,2-bis(di-5-tert-butyladamantylphosphinomethyl)benzene, 1,2-bis(1-adamantyl tert-butyl-phosphinomethyl)benzene, 1-(diadamantylphosphinomethyl)-2-(di-tert-

30 butylphosphinomethyl)benzene, 1-(di-tert-butylphosphinomethyl)-2-(dicongressylphosphinomethyl)benzene, 1-(di-tert-butylphosphinomethyl)-2-(phospha-adamantyl-P-

methyl)benzene, 1-(diadamantylphosphinomethyl)-2-(phospha-
 adamantyl-P-methyl)benzene, 1-(tert-
 butyladamantylphosphinomethyl)-2-(di-
 adamantylphosphinomethyl)benzene and 1-[(P-(2,2,6,6,-
 5 tetra-methylphosphinan-4-one)phosphinomethyl)]-2-(phospha-
 adamantyl-P-methyl)benzene, wherein "phospha-adamantyl" is
 selected from 2-phospha-1,3,5,7-tetramethyl-6,9,10-
 trioxadamantyl, 2-phospha-1,3,5-trimethyl-6,9,10-
 trioxadamantyl, 2-phospha-1,3,5,7-tetra(trifluoromethyl)-
 10 6,9,10-trioxadamantyl or 2-phospha-1,3,5-
 tri(trifluoromethyl)-6,9,10-trioxadamantyl; 1,2-bis-
 (dimethylaminomethyl)ferrocene, 1,2-bis-
 (ditertbutylphosphinomethyl)ferrocene, 1-hydroxymethyl-2-
 dimethylaminomethylferrocene, 1,2-bis-
 15 (ditertbutylphosphinomethyl)ferrocene, 1-hydroxymethyl-
 2,3-bis-(dimethylaminomethyl)ferrocene, 1,2,3-tris-
 (ditertbutylphosphinomethyl)ferrocene, 1,2-bis-
 (dicyclohexylphosphinomethyl)ferrocene, 1,2-bis-(di-iso-
 butylphosphinomethyl)ferrocene, 1,2-bis-
 20 (dicyclopentylphosphinomethyl)ferrocene, 1,2-bis-
 (diethylphosphinomethyl)ferrocene, 1,2-bis(di-
 isopropylphosphinomethyl)ferrocene, 1,2-bis-
 (dimethylphosphinomethyl)ferrocene, 1,2-bis-(di-(1,3,5,7-
 tetramethyl-6,9,10-trioxa-2-phospha-
 25 adamantylmethyl))ferrocene, 1,2-bis-
 (dimethylaminomethyl)ferrocene-bismethyl iodide, 1,2-
 bis(dihydroxymethylphosphinomethyl)ferrocene, 1,2-
 bis(diphosphinomethyl)ferrocene, 1,2-bis- α,α -(P-(2,2,6,6,-
 tetramethylphosphinan-4-one))dimethylferrocene, and 1,2-
 30 bis-(di-1,3,5,7-tetramethyl-6,9,10-trioxa-2-phospha-
 adamantylmethyl))benzene; cis-1,2-bis(di-t-
 butylphosphinomethyl)-4,5-dimethyl cyclohexane; cis-1,2-

bis(di-t-butylphosphinomethyl)-5-methylcyclopentane;
 cis-1,2-bis(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-
 trioxa-adamantyl)-4,5-dimethylcyclohexane; cis-1,2-bis(2-
 phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-
 5 adamantyl) 5-methylcyclopentane; cis-1,2-bis(di-
 adamantylphosphinomethyl)-4,5 dimethylcyclohexane; cis-
 1,2-bis(di-adamantylphosphinomethyl)-5-methyl
 cyclopentane; cis-1- (P,P adamantyl, t-butyl
 phosphinomethyl)-2-(di-t-butylphosphinomethyl)-4,5-
 10 dimethylcyclohexane; cis-1- (P,P adamantyl, t-butyl
 phosphinomethyl)-2-(di-t-butylphosphinomethyl)-5-
 methylcyclopentane; cis-1- (2-phosphinomethyl-1,3,5,7-
 tetramethyl-6,9,10-trioxa-adamantyl) - 2 - (di-t-
 butylphosphinomethyl)4,5- dimethylcyclohexane; cis-1- (2-
 15 phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-
 adamantyl) - 2 - (di-t-butylphosphinomethyl)-5-methyl
 cyclopentane; cis-1-(2-phosphinomethyl-1,3,5,7-
 tetramethyl-6,9,10-trioxa-adamantyl) -2-
 (diadamantylphosphinomethyl)-5-methyl cyclohexane; cis-1-
 20 (2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-
 adamantyl)-2-(diadamantylphosphinomethyl)-5-methyl
 cyclopentane; cis-1-(2-phosphinomethyl-1,3,5,7-
 tetramethyl-6,9,10-trioxa-adamantyl) -2-
 (diadamantylphosphinomethyl)cyclobutane; cis-1-(di-t-
 25 butylphosphinomethyl)-2- (diadamantylphosphinomethyl)-
 4,5-dimethyl cyclohexane; cis-1-(di-t-
 butylphosphinomethyl)-2- (diadamantylphosphinomethyl)-5-
 methyl cyclopentane; cis-1,2-bis(2-phospha-1,3,5-
 trimethyl-6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-4,5-
 30 dimethyl cyclohexane; cis-1,2-bis(2-phospha-1,3,5-
 trimethyl-6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-5-
 methyl cyclopentane; cis-1-(2-phospha-1,3,5-trimethyl-
 6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-2-(di-t-

butylphosphinomethyl)-4,5-dimethyl cyclohexane; cis-1-(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-2-(di-t-butylphosphinomethyl)-5-methyl cyclopentane; cis-1-(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-2-(diadamantylphosphinomethyl)-4,5-dimethyl cyclohexane; cis-1-(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-2-(diadamantylphosphinomethyl)-5-methyl cyclopentane; cis-1,2-bis-perfluoro(2-phospha-1,3,5,7-tetramethyl-6,9,10-trioxatricyclo{3.3.1.1[3.7]}decyl)-4,5-dimethyl cyclohexane; cis-1,2-bis-perfluoro(2-phospha-1,3,5,7-tetramethyl-6,9,10-trioxatricyclo{3.3.1.1[3.7]}decyl)-5-methyl cyclopentane; cis-1,2-bis-(2-phospha-1,3,5,7-tetra(trifluoro-methyl)-6,9,10-trioxatricyclo{3.3.1.1[3.7]}decyl)-4,5-dimethyl cyclohexane; cis-1,2-bis-(2-phospha-1,3,5,7-tetra(trifluoro-methyl)-6,9,10-trioxatricyclo{3.3.1.1[3.7]}decyl)-5-methyl cyclopentane; cis-1,2-bis(di-t-butylphosphinomethyl)cyclohexane; cis-1,2-bis(di-t-butylphosphinomethyl)cyclopentane; cis-1,2-bis(di-t-butylphosphinomethyl)cyclobutane; cis-1,2-bis(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxaadamantyl)cyclohexane; cis-1,2-bis(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxaadamantyl)cyclopentane; cis-1,2-bis(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxaadamantyl)cyclobutane; cis-1,2-bis(diadamantylphosphinomethyl)cyclohexane; cis-1,2-bis(diadamantylphosphinomethyl)cyclopentane; cis-1,2-bis(diadamantylphosphinomethyl)cyclobutane; cis-1-(P,P-adamantyl, t-butyl-phosphinomethyl)-2-(di-t-butylphosphinomethyl)cyclohexane; cis-1-(P,P-adamantyl, t-butyl-phosphinomethyl)-2-(di-t-butylphosphinomethyl)cyclopentane; cis-1-(P,P-adamantyl,

t-butylphosphinomethyl)-2-(di-t-butylphosphinomethyl)cyclobutane; cis-1-(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-adamantyl) - 2 - (di-t-butylphosphinomethyl) cyclohexane;
 5 cis-1-(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-adamantyl) - 2 - (di-t-butylphosphinomethyl)cyclopentane; cis-1-(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-adamantyl) - 2 - (di-t-butylphosphinomethyl) cyclobutane;
 10 cis-1-(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-adamantyl) -2-(diadamantylphosphinomethyl)cyclohexane; cis-1-(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-adamantyl)-2-(diadamantylphosphinomethyl)cyclopentane;
 15 cis-1-(2-phosphinomethyl-1,3,5,7-tetramethyl-6,9,10-trioxa-adamantyl) -2-(diadamantylphosphinomethyl)cyclobutane; cis-1-(di-t-butylphosphinomethyl)-2-(diadamantylphosphinomethyl)cyclohexane; cis-1-(di-t-butylphosphinomethyl)-2-(diadamantylphosphinomethyl)cyclopentane;
 20 cis-1-(di-t-butylphosphinomethyl)-2-(diadamantylphosphinomethyl)cyclobutane; cis-1,2-bis(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-
 25 {3.3.1.1[3.7]}decyl)cyclohexane; cis-1,2-bis(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-
 {3.3.1.1[3.7]}decyl)cyclopentane; cis-1,2-bis(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-
 {3.3.1.1[3.7]}decyl)cyclobutane; cis-1-(2-phospha-
 30 1,3,5-trimethyl-6,9,10-trioxatricyclo-
 {3.3.1.1[3.7]}decyl)-2-(di-t-butylphosphinomethyl)cyclohexane; cis-1-(2-phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-

{3.3.1.1[3.7]}decyl)-2-(di-
 butylphosphinomethyl)cyclopentane; cis-1-(2-phospha-1,3,5-
 trimethyl-6,9,10-trioxatricyclo-{3.3.1.1[3.7]}decyl)-2-
 (di-*t*-butylphosphinomethyl)cyclobutane; cis-1-(2-phospha-
 5 1,3,5-trimethyl-6,9,10-trioxatricyclo-
 {3.3.1.1[3.7]}decyl)-2-
 (diadamantylphosphinomethyl)cyclohexane; cis-1-(2-phospha-
 1,3,5-trimethyl-6,9,10-trioxatricyclo-
 {3.3.1.1[3.7]}decyl)-2-
 10 (diadamantylphosphinomethyl)cyclopentane; cis-1-(2-
 phospha-1,3,5-trimethyl-6,9,10-trioxatricyclo-
 {3.3.1.1[3.7]}decyl)-2-
 (diadamantylphosphinomethyl)cyclobutane; cis-1,2-bis-
 perfluoro(2-phospha-1,3,5,7-tetramethyl-6,9,10-
 15 trioxatricyclo{3.3.1.1[3.7]}-decyl)cyclohexane; cis-1,2-
 bis-perfluoro(2-phospha-1,3,5,7-tetramethyl-6,9,10-
 trioxatricyclo{3.3.1.1[3.7]}decyl)cyclopentane; cis-1,2-
 bis-perfluoro(2-phospha-1,3,5,7-tetramethyl-6,9,10-
 trioxatricyclo{3.3.1.1[3.7]}decyl)cyclobutane; cis-1,2-
 20 bis- (2-phospha-1,3,5,7-tetra(trifluoro-methyl)-6,9,10-
 trioxatricyclo{3.3.1.1[3.7]}decyl)cyclohexane; cis-
 1,2-bis- (2-phospha-1,3,5,7-tetra(trifluoro-methyl)-
 6,9,10-trioxatricyclo{3.3.1.1[3.7]}decyl)cyclopentane;
 and
 25 cis-1,2-bis- (2-phospha-1,3,5,7-tetra(trifluoro-methyl)-
 6,9,10-trioxatricyclo{3.3.1.1[3.7]}decyl)cyclobutane; (2-
 exo, 3-exo)-bicyclo[2.2.1]heptane-2,3-bis(di-*tert*-
 butylphosphinomethyl) and (2-endo, 3-endo)-
 bicyclo[2.2.1]heptane-2,3-bis(di-*tert*-
 30 butylphosphinomethyl).

The bubble size produced by the invention may be small and therefore a large surface area is provided for interface mass transfer to take place. Furthermore, because the bubble size distribution is narrow with small deviation,
5 the carbonylation reaction can be controlled.

All of the features described herein may be combined with any of the above aspects, in any combination unless such combinations are mutually exclusive.

10

An embodiment of the invention will now be described, by way of example only, with reference to the accompanying drawings, wherein:

15 Figure 1 is a schematic sectional side view of a prior art mixing apparatus;

Figure 2 is a schematic sectional side view of a mixing apparatus according to the invention;

20

Figure 3 is a schematic sectional side view of a mixing apparatus according to the invention in use; and

Figure 4 is a further schematic sectional side view of a
25 mixing apparatus according to the invention in use.

Figure 1 shows a prior art mixing apparatus 10 in use in a vessel 12 containing a liquid 14. The vessel 12 has an inlet 16 through which a gas 18 is sparged into the liquid
30 14. The mixing apparatus 10 comprises a vertical elongate shaft 20 rotatable about a longitudinal axis 21 on which are fixedly mounted a first impeller 22 and a second impeller 24 in spaced apart relation. The first impeller

22 is mounted above the second impeller 24. Both the first and the second impellers 22,24 are 45° pitched blade turbines.

5 In use, said impellers must rotate at the same speed. The first impeller 22 causes incoming gas bubbles 26 to flow in a both an axial and radial direction. The axial component of the flow creates momentum, which along with buoyancy, prevents the second impeller 24 from operating
10 effectively. The momentum and buoyancy is only overcome by increasing the speed of the impellers 22, 24. This increase in speed causes full dispersion of the gas 18 in the liquid 14 as shown in Figure 1 by the lines A. Non-uniform dispersion of the liquid 14 in the gas 18 is
15 undesirable because the mixing process cannot be controlled.

Figure 2 shows a mixing apparatus 100 according to the present invention. The mixing apparatus 100 comprises a
20 vertical elongate shaft 120 rotatable about a longitudinal axis 121 on which are fixedly mounted a first impeller 122 and a second impeller 124 in a spaced apart arrangement. Both the first and the second impeller 122, 124 comprise a number of hydrofoil blades 125. Each impeller 122,124
25 comprises four radially extending blades 125 fixedly mounted on the shaft 120 for co-operating rotation about the longitudinal axis of the shaft, in use. Each blade 125 on each impeller 122,124 is a hydrofoil blade arranged so as to impel the surrounding fluid axially in the
30 direction of the other impeller. The first or lower impeller 122 is thus an up-pumping impeller and the second or upper impeller 124 is a down-pumping impeller. Although only two blades 125 can be seen in the figure, it

will be understood by a skilled person, that any number of blades may be used on each of the said impellers, for example, 3, 4 or even 6 blades. Particularly suitable commercially available impellers are those known as
5 Maxflo™ W, A315, A320 or A340 impellers.

The first impeller 122 is mounted on the shaft 120 so that the concave face of the blades 125 face in an upward direction. The second impeller 124 is spaced along the
10 shaft 120 and is mounted so that the concave face of the blades 125 face in a downwards direction. The distance between the first impeller 122 and the second impeller 124 is approximately the diameter of either of the said impellers 122, 124.

15

Figure 3 shows the mixing apparatus 100 in a cylindrical vessel 112. A gas inlet 116 is located in the bottom-wall 132 of the vessel 112 adjacent to the base 132. It will be appreciated that the vessel 112 can be of any suitable
20 alternative configuration, for example, it may be a hopper. The mixing apparatus 100 is centrally suspended in the vessel 112.

Although only one mixing apparatus 100 is shown in Figure
25 3, it will be appreciated that any number of mixing apparatus 100 could be used in the chamber 112. For example, two, three, or four mixing apparatus 100 may be mounted in the chamber 112.

30 It will also be understood that any number of first or second impellers 122,124 may be mounted on the shaft 120 and in keeping with the object of the invention. For example, the arrangement 200 shown in Figure 4, wherein a

series of first impellers 222 and a series of second impellers 224 are mounted on the shaft 220. Any number of impellers 222,224 may be provided on the shaft 220.

5 An alternative configuration would comprise pairs of impellers mounted on the shaft. Each pair would comprise a first impeller and a second impeller. There may be a number of pairs of impellers on any given shaft, for example, two, three, or four pairs. In such an
10 arrangement, the fluid, for example, gas, may be introduced into the chamber through the base or the side wall thereof, being directed under and towards the first impeller.

15 The first or the second impeller may be driven by a separate drive means so that the rotational speed of, for example, the first impeller, may be different to the rotational speed of the said other impeller.

20 The diameter of the first and second impellers may not necessarily be the same in any one of the embodiments shown. Further, the optimum distance between two impellers is dependent upon the geometry of the vessel and the diameter of said impellers.

25

In use, as in the illustrated carbonylation reaction, the vessel 112,212 is filled with fluid 114,214. A gas 118,218 is then directed into the vessel 112,212 via gas inlet 116,216. The shaft 120,220 is rotated by a suitable
30 drive means (not shown), so as to cause the first impeller 122,222 and the second impeller 124,224 to rotate about the longitudinal axis 121,221 of the shaft 120,220 in a liquid 114.

The gas 118,218 enters the vessel 112,212 as large bubbles 150,215. Rotation of the first impeller 122,222 causes the large bubbles 150,215 to move in an axial direction
5 towards the blades 125,225. The large bubbles 150,250 impact on the blades 125,225 and are broken up into a number of small bubbles 152,252 in the region of high turbulent energy dissipation. Small bubbles follow the liquid flow path which is initially axial then radial.

10

The second impeller 124,224 causes axial flow in a downwards direction towards the first impeller 122,222. Due to the opposing axial flow paths created by said impellers, a central zone 160,260, or high turbulent
15 energy dissipation zone, is established. The central zone 160,260 comprises an area of comparatively uniform high turbulent energy dissipation with high interfacial area to allow reaction between reactants in the liquid 114,214 and in reactants in the gas 118,218. Further, because the
20 turbulent energy dissipation in the central zone 160,260 is maintained without much variation, a narrow size distribution of small bubbles is produced. Such a narrow size distribution enables the reactor performance to be predicted and controlled.

25

The mixing apparatus 100,200 is particularly suited to carbonylation processes.

Table 1 gives an example of resultant bubble size for a
30 dual opposed Maxflo impeller system operating at various speeds, and at 4.2mm/sec. The bubble size was determined using a typical image capture camera.

N (rpm)	750	991	1200
d ₁₀ (μm)	296	330	394
d ₃₂ (μm)	450	510	500

Table 1

It is a distinct advantage to be able to control the
5 reaction occurring between two fluids, particularly a
liquid and a gas. It is also especially favorable to be
able to promote effective and efficient mixing and
interphase mass transfer between at least two fluids.
When applied to industrial processes, such advantages are
10 of high commercial value.

The reader's attention is directed to all papers and
documents which are filed concurrently with or previous to
this specification in connection with this application and
15 which are open to public inspection with this
specification, and the contents of all such papers and
documents are incorporated herein by reference.

All of the features disclosed in this specification
20 (including any accompanying claims, abstract and
drawings), and/or all of the steps of any method or
process so disclosed, may be combined in any combination,
except combinations where at least some of such features
and/or steps are mutually exclusive.

25

Each feature disclosed in this specification (including
any accompanying claims, abstract and drawings), may be
replaced by alternative features serving the same,
equivalent or similar purpose, unless expressly stated
30 otherwise. Thus, unless expressly stated otherwise, each

feature disclosed is one example only of a generic series of equivalent or similar features.

The invention is not restricted to the details of the foregoing embodiment(s). The invention extends to any novel one, or any novel combination, of the features disclosed in this specification (including any accompanying claims, abstract and drawings), or to any novel one, or any novel combination, of the steps of any method or process so disclosed.

CLAIMS

1. A mixing apparatus for mixing at least two fluids, the mixing apparatus comprising a shaft rotatable about its longitudinal axis, a first and a second radially extending impeller mounted on the shaft and respectively axially spaced apart, characterised in that the first impeller comprises a plurality of curved blades operable to move said fluids in an axial direction towards the second impeller, and the second impeller comprises a plurality of curved blades operable to move said fluids in an axial direction towards the first impeller.

2. A mixing apparatus as claimed in claim 1, characterised in that the blades on each impeller are inward pumping into the space between the impellers.

3. A mixing apparatus as claimed in claim 1 or 2, characterised in that the at least two fluids comprise a liquid and a gas.

4. A mixing apparatus as claimed in any one of the preceding claims, characterised in that the first impeller and the second impeller each comprise two or more curved blades.

5. A mixing apparatus as claimed in any one of the preceding claims, characterised in that the diameter of the first impeller is the same as the diameter of the second impeller.

6. A mixing apparatus as claimed in any one of the preceding claims, characterised in that the axial distance

between the first impeller and the second impeller is at least one impeller diameter apart.

7. A mixing apparatus as claimed in any one of the preceding claims, characterised in that said impellers operate at a power number substantially 1.75.

8. A mixing apparatus as claimed in any one of the preceding claims, characterised in that the specific power used when the first impeller and the second impeller rotate is substantially between 100W/m^3 to 800W/m^3 .

9. A mixing apparatus as claimed in any one of the preceding claims, characterised in that when dual opposed Maxflo impellers are used, the arithmetic mean size (d_{10}) is substantially between $250\mu\text{m}$ to $550\mu\text{m}$ and the surface volume mean diameter (d_{32}) is substantially between $400\mu\text{m}$ to $750\mu\text{m}$.

10. A mixing apparatus as claimed in any one of the preceding claims, characterised in that when BT-6 type impellers are used, the d_{10} is substantially between $250\mu\text{m}$ to $1500\mu\text{m}$.

11. A mixing apparatus as claimed in claim 10, characterised in that when operating at substantially 765rpm, the d_{10} is substantially $378\mu\text{m}$, and the d_{32} is substantially $445\mu\text{m}$.

12. A mixing apparatus as claimed in any one of the preceding claims, characterised in that the sparged gas rate is substantially between 0.05 to $1.0\text{ m}^3/\text{s}$.

13. A mixing apparatus as claimed in claim 12, characterised in that the sparged gas rate is substantially $0.13\text{m}^3/\text{s}$ at an impeller speed of substantially 50rpm to 200rpm.

5

14. A mixing apparatus as claimed in any one of the preceding claims, characterised in that a critical dispersion speed in a vessel having a diameter of substantially between 2 to 5m, is substantially between 10
10 to 20rpm.

15. A chemical reaction system comprising a liquid phase and a mixing apparatus according to the first aspect of the invention for mixing a fluid into the liquid phase.

15

16. A chemical reaction system as claimed in claim 15, characterised in that the liquid phase comprises at least one liquid phase reactant for reacting with a gas introduced into the liquid phase, as well as at least one
20 liquid phase reaction product.

17. A chemical reaction system as claimed in claims 15 or 16, characterised in that the liquid phase comprises a catalyst system.

25

18. A chemical reaction system as claimed in any one of claims 15 to 17, characterised in that the reaction system is a carbonylation process comprising carbonylating an ethylenically unsaturated compound with carbon monoxide in
30 the presence of a source of hydroxyl groups, and a catalyst system comprising (a) a bidentate phosphine, arsine or stibine ligand, and (b) a catalytic metal

selected from a group 8, 9 or 10 metal or a compound thereof.

19. A mixing apparatus substantially as hereinbefore
5 described with reference to any one of the accompanying drawings.

-1/4-

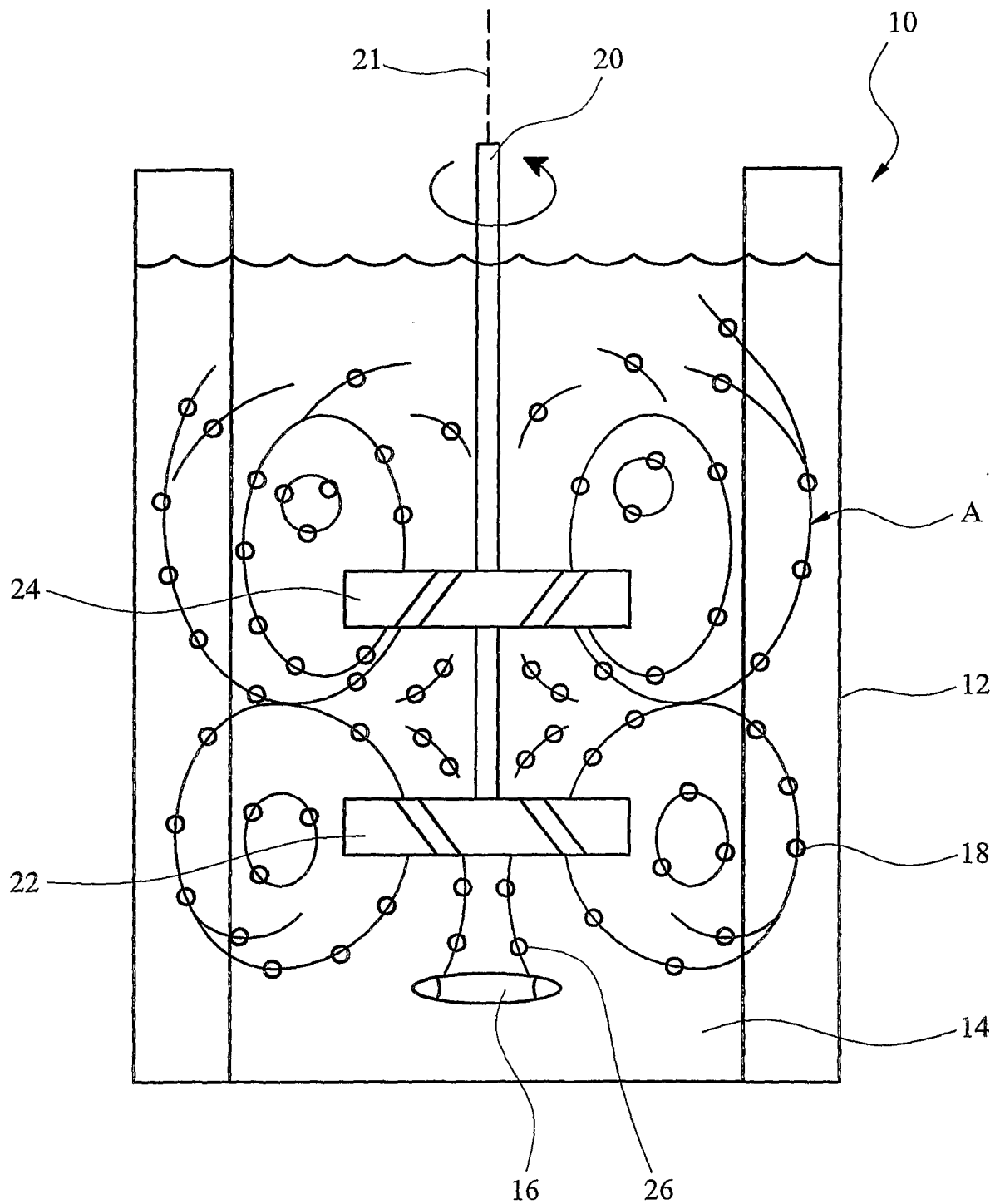
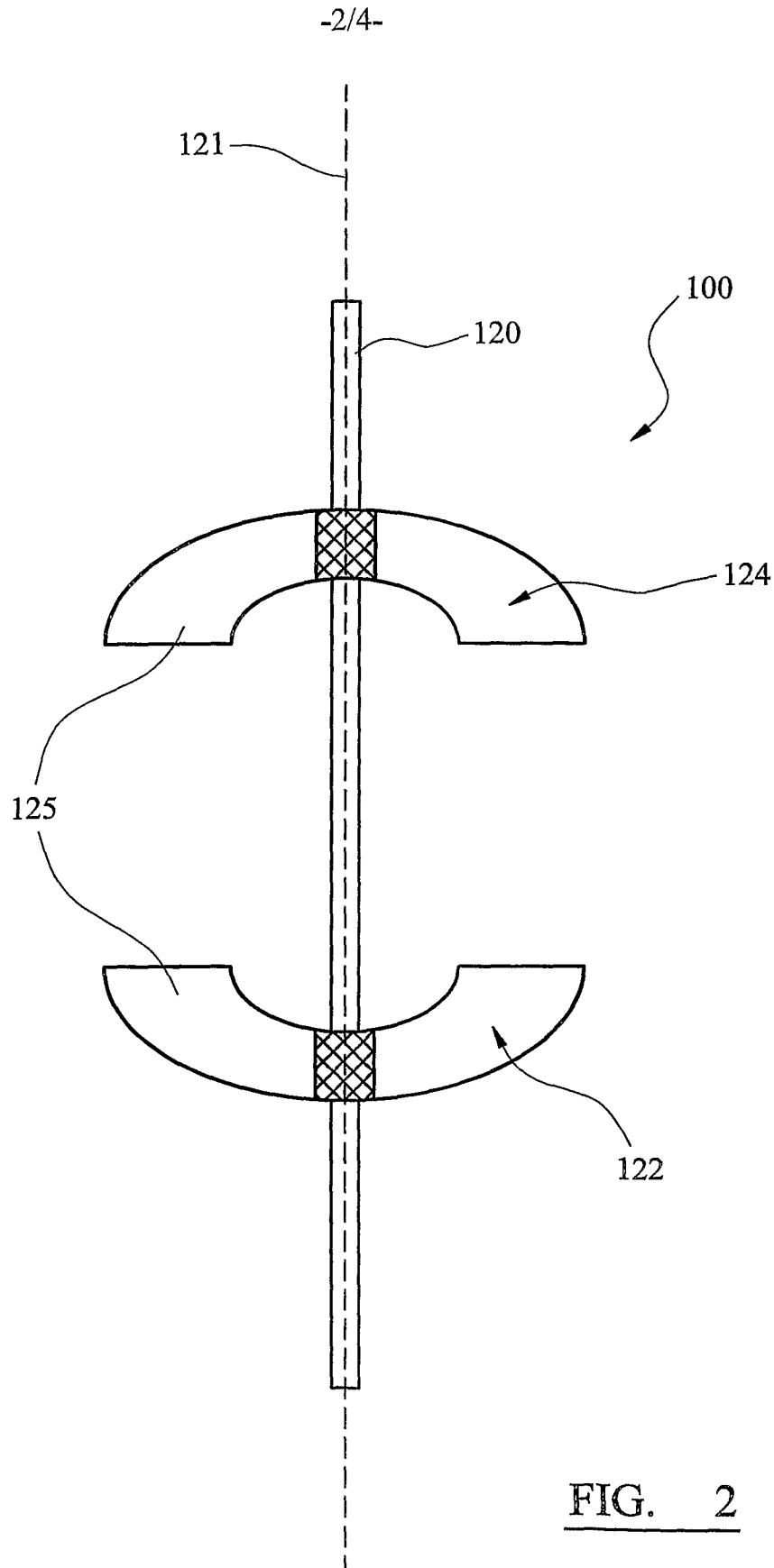


FIG. 1

PRIOR ART



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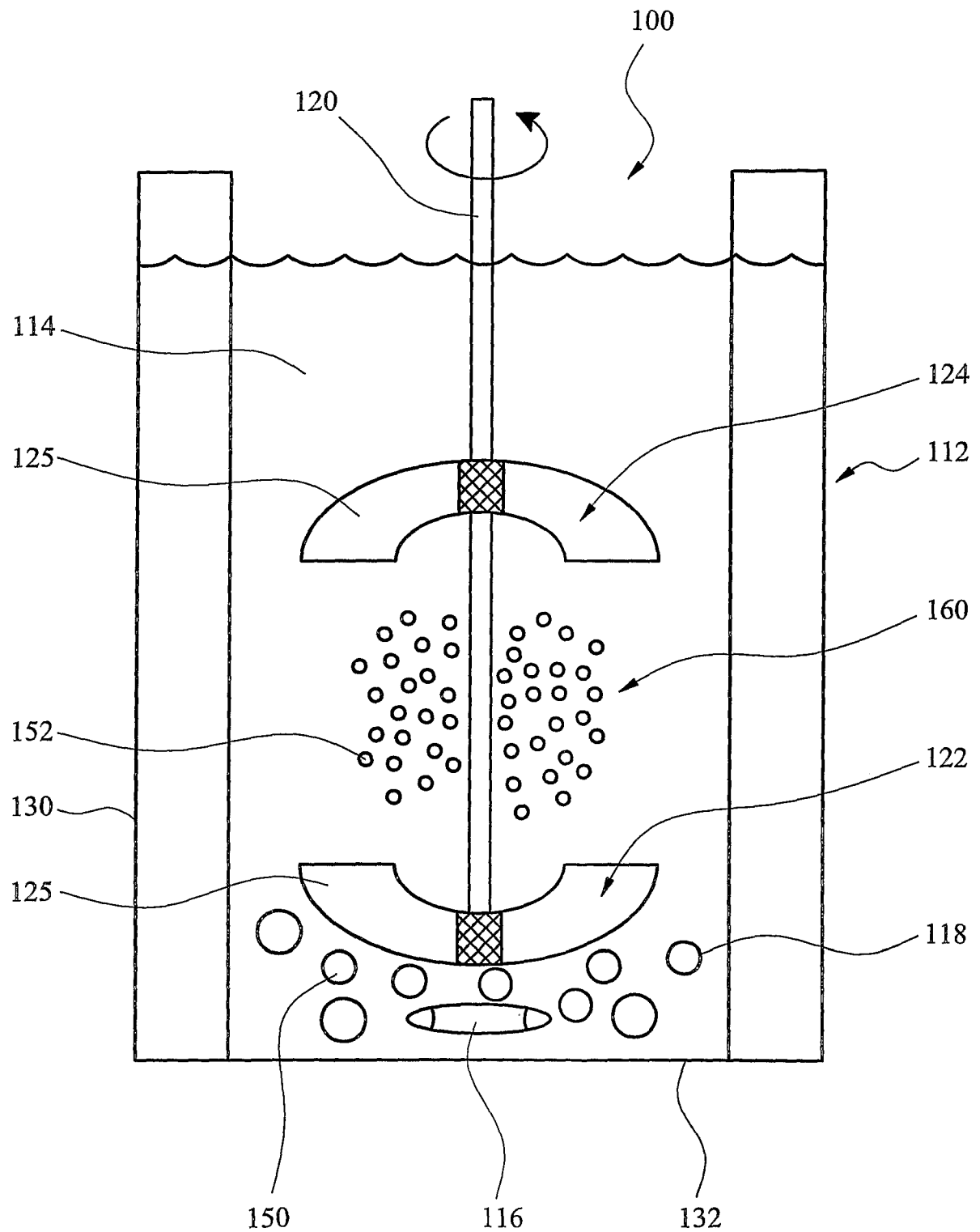


FIG. 3

-4/4-

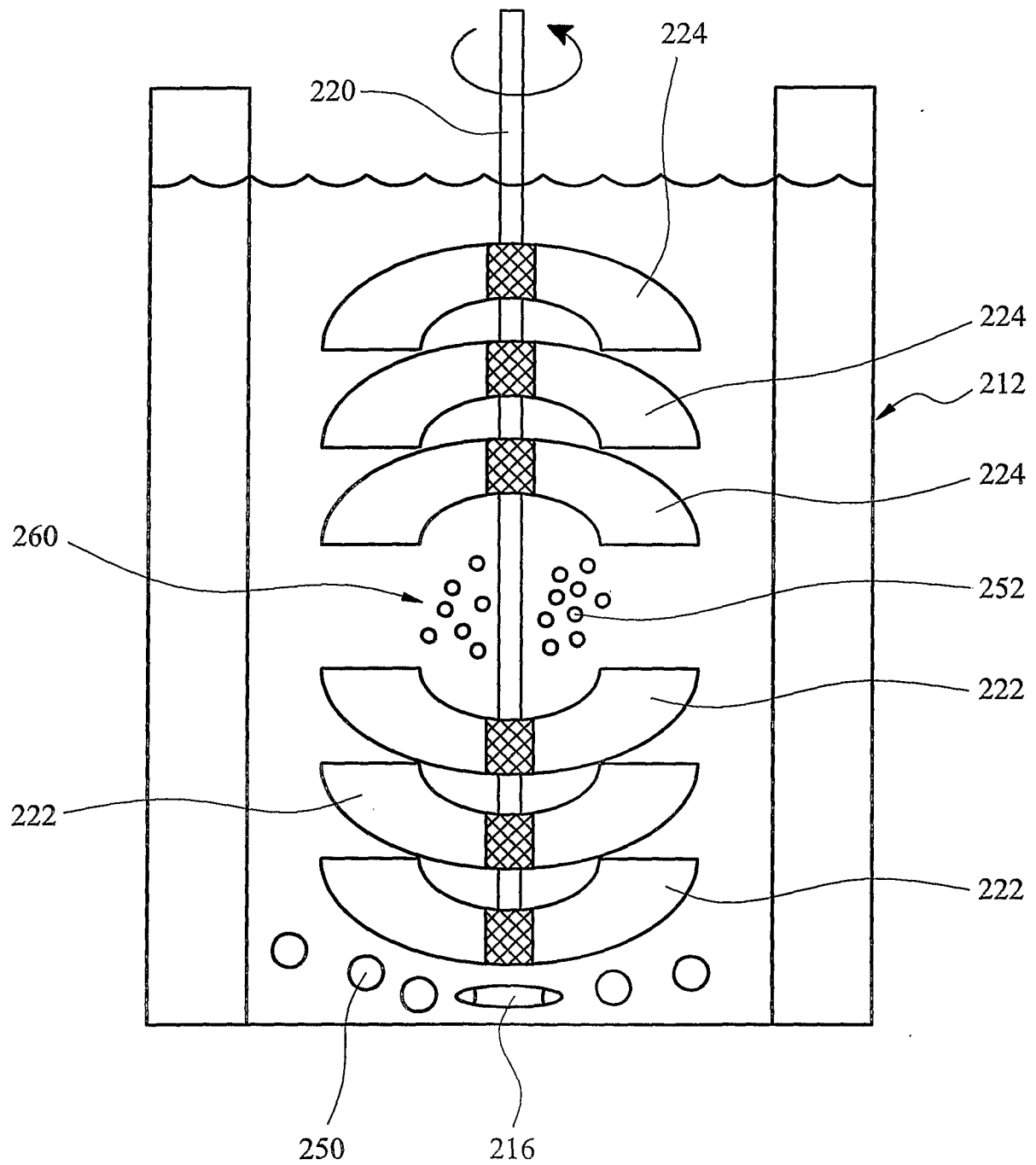


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2007/001524

A. CLASSIFICATION OF SUBJECT MATTER

INV. B01F3/04 B01F7/00 C07C51/14 B01J19/18

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B01F C07C B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2004/042942 A1 (SICCARDI EDWARD S [US]) 4 March 2004 (2004-03-04) paragraphs [0008] - [0011], [0022], [0024], [0025] claims 1,2,7 abstract; figures 2-4	1-14
X	DE 203 06 404 U1 (MAVAG VERFAHRENSTECHNIK AG NEU [AT]) 26 June 2003 (2003-06-26) page 3, line 1 - page 4, line 13 abstract; figure 13	1-14
X	CH 332 926 A (KREUZER EMIL [CH]) 30 September 1958 (1958-09-30) page 1, lines 1-57 figure 1	1-14
	----- -/-- -----	

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

6 August 2007

Date of mailing of the international search report

20/08/2007

Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2007/001524

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 44 21 478 A1 (NETZSCH ERICH HOLDING [DE]) 21 December 1995 (1995-12-21) page 1, lines 3-11 page 3, lines 45-66 page 4, lines 26-29 figure 2B -----	1-14
X	US 4 401 645 A (GISLER HENRY J [US]) 30 August 1983 (1983-08-30) column 1, lines 8-21 column 4, line 9 - column 5, line 21 abstract; figures 1,2 -----	1-14
A	US 4 004 786 A (STEPHENS WILL R) 25 January 1977 (1977-01-25) abstract; figures 1-4 -----	1-14

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Claims Nos.: 15-19

Independent claim 15 has not been defined by technical features referring to a chemical reaction system, but contains a broad and general reference to a "...first aspect of the invention..." which appears to be described in the description on p. 3 lines 19 to 29.

Furthermore, independent apparatus claim 19 has also not been defined by technical features, but simply consists of a reference to the drawings.

According to Rule 6.2(a) PCT, claims should not contain such references to the description or the drawings except where absolutely necessary, which is not the case here. Consequently, claim 15 (as well as claims 16 to 18 being dependent on claim 15) and claim 19 lack clarity, Art. 6 PCT.

This non-compliance with the substantive provisions is to such an extent that no meaningful search could be performed for these claims 15 to 19.

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guideline C-VI, 8.5), should the problems which led to the Article 17(2) declaration be overcome.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/GB2007/001524

Box II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☒ Claims Nos.: 15-19
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2007/001524

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2004042942	A1	04-03-2004	NONE
DE 20306404	U1	26-06-2003	NONE
CH 332926	A	30-09-1958	NONE
DE 4421478	A1	21-12-1995	NONE
US 4401645	A	30-08-1983	NONE
US 4004786	A	25-01-1977	NONE