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2,631,091

HIGH-PRESSURE CONTACTING APPARATUS

Filed May 21, 1949

2 SHEETS—SHEET 1

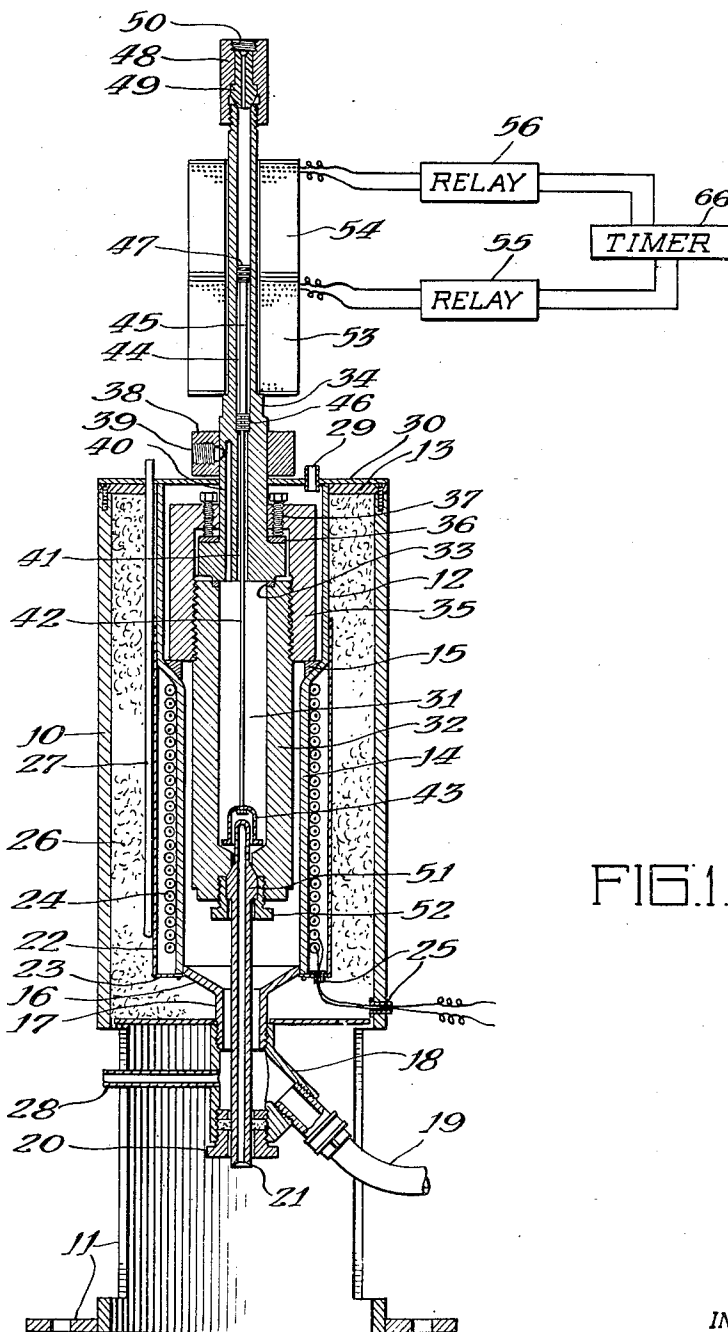


FIG. 1.

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2 SHEETS—SHEET 2

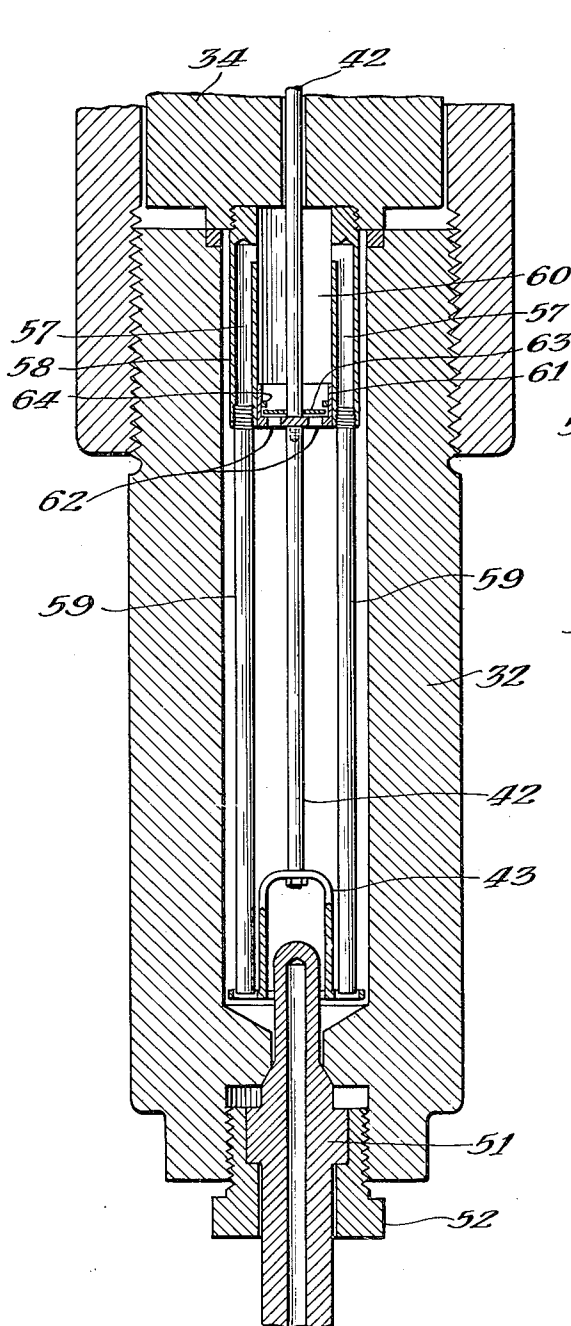


FIG. 2.

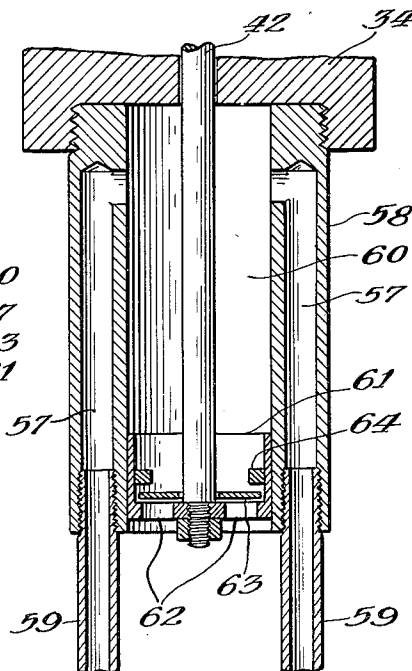


FIG. 3.

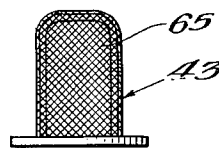


FIG. 4.

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UNITED STATES PATENT OFFICE

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HIGH-PRESSURE CONTACTING APPARATUS

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12 Claims. (Cl. 23-290)

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This invention relates to high pressure contacting apparatus and it pertains, more particularly, to improved apparatus for effecting contact between two separate phases, one of which being a liquid and the other being either gaseous, liquid or solid, under extremely high pressure conditions and under closely controlled temperature conditions.

An object of the invention is to provide an apparatus which is particularly suitable for small-scale or laboratory use in obtaining critically close control in the contacting of two immiscible phases at pressures in the range of 1,000 to 15,000 pounds per square inch and at temperatures up to 750° F. or higher. A particular object is to provide an improved stirring mechanism entirely closed within the reactor space and controllable with a precision which has heretofore been unobtainable in reactors of this type. Another object is to provide a heating and temperature control system from which the contacting apparatus per se is readily separable. A further object is to provide an improved method and means for recycling and/or introducing gases into liquids at extremely high super atmospheric pressure. Other objects will be apparent as the detailed description of the invention proceeds.

In accordance with our invention, an upright open-ended contactor support is provided with heating and temperature control elements so that the contactor itself is not encumbered by the heating element but may simply be inserted into it. The contactor, or reactor, is preferably provided with a thermowell at its base, and it is usually of utmost importance that separate phases immediately adjacent this thermowell be mixed or agitated at a determinable and precisely controllable rate. To accomplish this mixing or contacting, a plunger-type mixer is reciprocally mounted in the contactor so that in its lowermost position it comes in close proximity to the base of the reactor and the thermowell, and in its upper position it approaches the top of a liquid phase in the contactor or emerges from the top of said liquid phase, depending upon whether or not it is desired to obtain a mixing of an upper gaseous phase with a lower liquid phase or phases. The stirrer is positively actuated, both upwards and downwards, by the electromagnetic effect of two separate solenoids on a magnetic core which is fixedly connected to the stirrer. The upper solenoid functions to positively raise the core and stirrer and the lower coil to positively lower the core and stirrer in accordance with an electronic timing mechanism of any type known to the art

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so that both the frequency and the speed of the upward and downward movements can be controlled with great precision. The positive downward stroke of the core and mixer is perhaps of even greater effectiveness than the upward movement thereof but it is essential that movement in both directions be positively actuated and carefully controlled.

The invention provides not only for positive and closely controlled stirring or mixing action in both upward and downward directions, but it also provides a means for simultaneously or alternatively recycling a gaseous phase from the top of the reactor or contactor back to the bottom thereof. In this embodiment of the invention, the plunger connected to the core carries a piston with a flat plate ring valve, ball valve, or the like; and a gas conduit is provided for conducting gas from the upper part of the chamber to the base thereof. The sharp downward movement of the core causes the valve to open and predetermined amount of gas to be trapped above the valve; the sharp or controlled upward movement of the core forces the entrapped gas through the conduit to the base of the contactor or reactor.

The invention will be more clearly understood from the following detailed description of a specific example thereof, read in conjunction with the accompanying drawings, in which Figure 1 is a schematic view of our improved apparatus in vertical section.

Figures 2, 3 and 4 are detailed views illustrating gas recycle features.

As an example of the apparatus of our invention we will describe a laboratory reactor designed to obtain closely controlled contacting of gases with liquids at a predetermined temperature which may be as high as 750° F. or more and at an extremely high pressure which may be in the range of 2,000 to 15,000 pounds per square inch. The apparatus consists essentially of two readily separable parts, namely: (A) a heater and support; and (B) a reactor with its associated contacting means. The heater and support consists of a section of 6-inch (schedule 40) steel pipe 10, about 18 inches long, welded to a flanged support 11, which may be bolted to a work bench or the like. A 3¼ inch I. D. stainless steel tube 12, ½ inch thick and about 4¼ inches long is welded to flange 13, which may be screwed to the top of pipe 10. To the rounded bottom portion of pipe 12 is welded a 3-inch O. D. stainless steel tube 14, about ⅜ inch thick and 6½ inches long, lugs 15 being provided at the juncture of these two pipes for supporting the reactor assembly. A

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conical base 16 is welded to the base of tube 14 at its upper end and at its base to a $\frac{3}{4}$ inch stainless steel pipe 17, 1 inch long, the latter being provided with a Y-pipe connection 18 for drain hose 19, and also being provided with a ring and packing nut assembly 20 for receiving the thermowell casing 21 of the reactor. A 24 gauge 18-8 stainless steel sheath 22 extends downwardly from pipe 12 and together with the annular base 23 of similar metal forms a chamber for heating coils 24, which in this case, consists of approximately 20 feet of No. 22 Nichrome wire insulated with one-quarter inch diameter fishspine beads. The ends of the wire are passed through porcelain tubes 25 through base 23 and through pipe 10 for connection to a power source, this particular heater 24 being of about 600-watt capacity.

The space between pipe 10 and the stainless steel sheath 22 is preferably filled with magnesia, vermiculite, or other suitable insulation material 26, and the temperature of this space may be measured by a thermocouple or other suitable means introduced into thermowell 27. In order that the reactor may be quickly quenched, a gaseous cooling fluid may be introduced by communication 28 passing up through the annulus between the exterior of the reactor 31 and the inside wall of the heater tube 14. Liquid coolants, such as cold water, may be introduced through conduit 29 inserted in loose split cover 30 to deluge down over the reactor and flow out through Y-connection 18 and hose 19. The apparatus so far described constitutes the heater and support or outer shell of our improved apparatus, which is preferably fixedly mounted. Other types of heater and support may be provided for special purposes: A water, steam or oil bath for more critical temperature control; a specially fabricated "Glass-Col" heating mantle for lightweight flexibility in use.

The reactor or contractor 31 is formed chiefly by pressure vessel walls 32 which may be of about $\frac{3}{4}$ inch thick with an inside diameter of about $1\frac{1}{2}$ inches and a height of about 6 inches. The notched upper and inner edge of the reactor is provided with a gasket 33 of copper or other suitable metal against which the reactor head 34 is secured by closure cap 35, the pressure exerted by the inner flange thereof being equalized by a thrust plate ring 36 and suitable bolts 37 threaded through said flange. The reactor head 34 may be provided with a suitable high pressure connecting ring fitting 38 carrying 2 or more ports, one of which, 39, is shown connected to a small separate conduit or bore 40 in the head. The other ports (not shown) connect directly to an axial conduit or bore 41. Reactants or any fluid may be introduced into the reactor through any of these ports, and one of them is preferably attached to a refrangible safety disk holder with disk (not shown) designed to burst at 50% over the reaction pressure. Bore 41 is provided for the stem 42 of mixer 43 and in the upper part of the reactor head 34, bore 41 leads into a larger diameter bore 44 to accommodate magnetic core 45 which is secured at its base to stem 42. A lower spring 46 cushions the lowermost movement of the core, stem and stirrer blade 43 and prevents actual impingement of the latter against the base of the reactor, and upper spring 47 similarly cushions the uppermost movement of magnetic core 45.

The top of the reactor head 34 is closed by a

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connector 48 which forces the coned gland 49 into a cone seat cut in the top end of the reactor head. A standard high pressure cone seat fitting 50 in the top of the connector and gland then connects the reactor to a pressure gauge (not shown). The reactor head 34, stem 42 and springs 46 and 47 must be made of non-magnetic material, preferably stainless steel of austenitic structure, although copper, brass, and the like, non-magnetic materials may be used as well.

While the reactor closure here described is of the compression type, our invention is in no way restricted to this type closure, and any suitable closure such as the self-sealing type may be preferred especially at the higher pressures of 5000 p. s. i. and up.

The thermowell casing 21 extends upwardly into the bottom of the reactor, said casing having a shoulder 51 which is held in place by a gland nut 52 which is threaded to engage threads on a projecting lower end of the reactor walls. In reactors of larger size, the thermowell casing may depend from the reactor head instead of extending through its base.

Surrounding the upper end of the reactor head 34 are two solenoids 53 and 54. The current to the solenoid 53 is controlled by relay 55 and the current to solenoid 54 is controlled by relay 56. The current is applied alternately to each relay by a timer 56 which permits individual control of the "on" time of each. (A convenient commercially available relay and timer combination is offered by G. C. Wilson and Company, Chatham, N. J., as Repeat Cycle Timer Model No. 1, with $\frac{1}{16}$ standard cycle time.) Any dual control timer which permits separate control of each cycle in the range 0.05 to 5 seconds would be suitable. By having two separate solenoids and having each solenoid separately controlled by an electronic timer, the movement of the stirrer is positively actuated in both upward and downward directions and it may be controlled with great precision. Various sizes and shapes of stirrers may, of course, be employed depending upon the viscosity of the fluids to be mixed. The movement of the mixer may likewise be controlled by changing the length of stem 42 or by employing suitable stops so that it remains constantly within a liquid phase in the reactor when it is desired to contact 2 immiscible liquids and so that it may move above the liquid level when it is desired to obtain contact between a gas and a liquid.

For obtaining intimate contact between a gas and a liquid and, particularly for recycling a gas from the upper part of the reactor to the bottom thereof, a modification of our apparatus may be employed as illustrated in Figure 2 and Figure 3 which shows an enlarged view of the essential parts. In this case, one or more conduits 57 are provided in the walls of a small cylinder 58 which may be removably attached by screw thread to the reactor head 34 or may be integrally machined into the base of the reactor head 34, and tubes 59 extend these conduits to the base of the reactor. In this case, a larger opening 60 is provided in the reactor head or attached cylinder 58 to serve as a gas receiver and a piston 61 is secured to stem 42, the piston being provided with one or more valves illustrated by opening 62, plate ring 63, and plate retaining ring 64. With this apparatus when stem 42 is moved sharply in a downward direction the plate ring is dislodged from openings 62 (and retained by retain-

ing ring 64) so that the space above the piston 61 is filled with gas. During upward movement of stem 42 the entrapped gas is forced through conduits 57 and 59 into the base of the reactor. Mixer 43 may optionally be attached to the piston 61 by extension of rod 42 to stir liquid phase during gas recycling operation, mixer blades 43 being suitably apertured or spaced to avoid contact with tubes 59.

A further modification of our apparatus for recycling gas from the upper part of the reactor to, and intimate dispersion of gas in, the liquid phase may be employed as illustrated in Figure 4. In this case, the stirrup type mixer 43 in Figure 1 is modified by placing an inverted cup 65 of suitable mesh screen over the connecting stirrup of mixer 43 and adjusting the stroke of the mixer rod 42 so that the mixer 65 is lifted above the liquid surface into the gas phase where it fills with the gas. The meshes of the screen are closed by liquid film due to surface tension effects, thus capturing a given volume of gas in the inverted cup. On the down stroke, the sudden submergence of mixer 65 causes the gas to bubble out through the meshes and escape to the surface in a fine spray with attendant beneficial effects of intimate contact of gas and liquid phases. Other shapes and forms of mixers, suitably perforated or meshed, may be used, the above being cited merely as an example of the principle involved.

It will thus be seen that we have accomplished the objects of our invention. Furthermore, we have provided an apparatus with an improved safety feature by surrounding the reactor with the heating and insulating chamber, which would absorb at least a substantial part of the shock should the contactor burst on account of failure of the safety disk to function. The stainless steel liner tube of the support permits water cooling of the reactor in case of emergency or if a reactor requires "quenching," the design also permits rapid air cooling of the reactor. A most important feature, however, is precision with which mixing can be accomplished by positively and closely controlled movement of the magnetic core in both upward and downward direction by the electronically controlled pair of solenoids. Obviously, many variations in structure may be made. The solenoids may be slideably mounted on the narrowed, non-magnetic portion of the reactor head and held in any desired position by set screws. The reciprocating motion of the core may be translated by any known mechanism to provide rotary stirring. In sufficiently large reactors, the thermowell casing may suspend from the reactor head instead of being inserted at the base. These and other modifications will be apparent to those skilled in the art from the above description.

We claim:

1. Apparatus for contacting separate phases at controlled temperature and high pressure, which apparatus comprises a pressure vessel having an open top and closed base, a thermowell casing extending into the lower part of said vessel and removably secured therein, an elongated closure element for said pressure vessel of non-magnetic material provided with a bore communicating with said vessel and in substantial alignment therewith, a mixer element in said vessel, a movable magnetic core in said bore, a non-magnetic stem connecting said core to said mixer; a lower solenoid coaxial with said core in its lower position, an upper solenoid coaxial with said core in its upper position, said solenoids being in opposed

tandem arrangement and adapted alternately to lift and to depress the core, a timer for separately controlling the energizing of each solenoid, and means for securing said closure element on said

5 vessel.

2. The apparatus of claim 1 in which the mixer element consists essentially of a reciprocable element in close proximity in its lower position to the thermowell casing and to the base of the

10 vessel.

3. The apparatus of claim 1 wherein the mixer consists essentially of a reciprocable element comprised of an inverted foraminous metal cup.

4. The apparatus of claim 1 wherein the mixer element comprises a valved piston and a conduit communicating from a space above said piston to the bottom of the vessel.

5. The apparatus of claim 1 wherein the pressure vessel is provided with an opening at its base and a gland nut for mounting the thermowell casing through said base.

6. The apparatus of claim 1 wherein the elongated closure element is provided with a second longitudinal bore which serves as a conduit leading from the exterior of said closure element into said vessel, a high pressure connecting ring fitting surrounding the elongated closure element adjacent the outlet end of said second bore, and at least one port in said ring fitting which communicates with the said outlet end of said second bore.

7. Contacting apparatus which comprises a pressure vessel with an open top, an axially elongated closure element for closing the top of said vessel, said closure element being provided with a bore communicating with said vessel and in substantial axial alignment therewith, a mixer element in said vessel, a movable magnetic core in said bore, a stem connecting said core to said mixer element, a plurality of separate solenoids surrounding the upper part of the elongated closure element at substantially the level of said core, said solenoids being arranged in opposed tandem relation co-axially with the upper end of said closure element, and a timer for controlling the energizing of each solenoid.

8. Apparatus for contacting separate phases at controlled temperature and high pressure, which apparatus comprises a pressure vessel, a thermowell casing extending into the base of said vessel with an open top and a closed base, a threaded opening through said base, and removably secured through said threaded opening, an elongated closure element for closing the top of said pressure vessel, said closure element having an elongated upper portion of non-magnetic material, a bore in said elongated upper portion communicating with said vessel and in substantial alignment therewith, a mixer element in said vessel, a movable magnetic core in said bore, a non-magnetic stem connecting said core to said mixer element, a lower solenoid substantially surrounding said core in its lower position, an upper solenoid substantially surrounding said core in its upper position, a timer for separately controlling the energizing of each solenoid, a closure cap for securing said head on said vessel, and a heater support for said pressure vessel, said heater support comprising an annular insulated jacket, an upper inner wall of slightly larger inside diameter than the outside diameter of the securing means, a cap inner wall of slightly larger inside diameter than the outside diameter of the said pressure vessel, supports at the top of the lower walls for engagement with the lower part of the securing means, an electri-

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cal heating element surrounding the lower inner walls, a connection for introducing a fluid into the space between the lower walls, a connection for withdrawing fluid from said space, and a packing unit assembly for sealing the space between the lower walls and the thermowell casing.

9. An apparatus for effecting contact of separate phases in a closed high pressure vessel which apparatus comprises an open top pressure vessel, a non-magnetic elongated closure element for said pressure vessel provided with a bore in communication with said vessel and in substantial axial alignment therewith, a mixer element in said vessel, a movable magnetic core within said non-magnetic bore, a stem connecting said core and said mixer element, a pair of separately operable opposed solenoids coaxial with the elongated closure element of the pressure vessel in the region of said core, a relay means for controlling the energizing of said solenoids, and a heater-support for said pressure vessel including means for fixing a removable electrical heating element about the outer wall of said vessel.

10. Contacting apparatus which comprises a pressure vessel having side and bottom walls but open at its top, a closure element for the top of said pressure vessel, said closure element having an elongated upper portion, said elongated upper portion of said closure being of non-magnetic material and being provided with a bore which extends downwardly through said closure element and communicates with said vessel in substantial alignment therewith, mixing means in said vessel, a movable magnetic core in said bore, connecting means between said core and said mixing means whereby the latter is actuated by the former, and solenoid means for positively reciprocating said core within said bore, said solenoid means including a pair of solenoids arranged in opposed tandem relation co-axially about said bore.

11. The apparatus which comprises a pressure vessel having side and bottom walls but open at its top, a closure element for the top of said pressure vessel, said closure element having an elongated upper portion, said elongated upper portion of said closure being of non-magnetic ma-

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terial and being provided with a bore which extends downwardly through said closure element and communicates with said vessel in substantial alignment therewith, a reciprocable member in said vessel, a foraminous metal cup carried by said reciprocable member, a movable magnetic core in said bore, connecting means between said core and said reciprocable member whereby the latter is actuated by the former, and solenoid means for positively reciprocating said core within said bore, said solenoid means including a pair of solenoids arranged in opposed tandem relation co-axially about said bore.

12. The apparatus which comprises a pressure vessel having side and bottom walls but open at its top, a closure element for the top of said pressure vessel, said closure element having an elongated upper portion, said elongated upper portion of said closure being of non-magnetic material and being provided with a bore which extends downwardly through said closure element and communicates with said vessel in substantial alignment therewith, a valved piston means in said vessel, conduit means communicating from a point above said piston to a point adjacent the bottom of the said vessel, a movable magnetic core in said bore, connecting means between said core and said piston means whereby the latter is actuated by the former, and solenoid means for positively reciprocating said core within said bore, said solenoid means including a pair of solenoids arranged in opposed tandem relation coaxially about said bore.

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