United States Patent

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[54] **AUTOMATIC CHEMICAL REACTION SYSTEM**
14 Claims, 12 Drawing Figs.

[52] **U.S. Cl.** **222/52,** 23/259, 222/70, 222/76, 222/132
[51] **Int. Cl.** **B67d 5/08**
[50] **Field of Search** 222/52, 56, 70, 76, 132; 23/259; 137/624.11, 624.18

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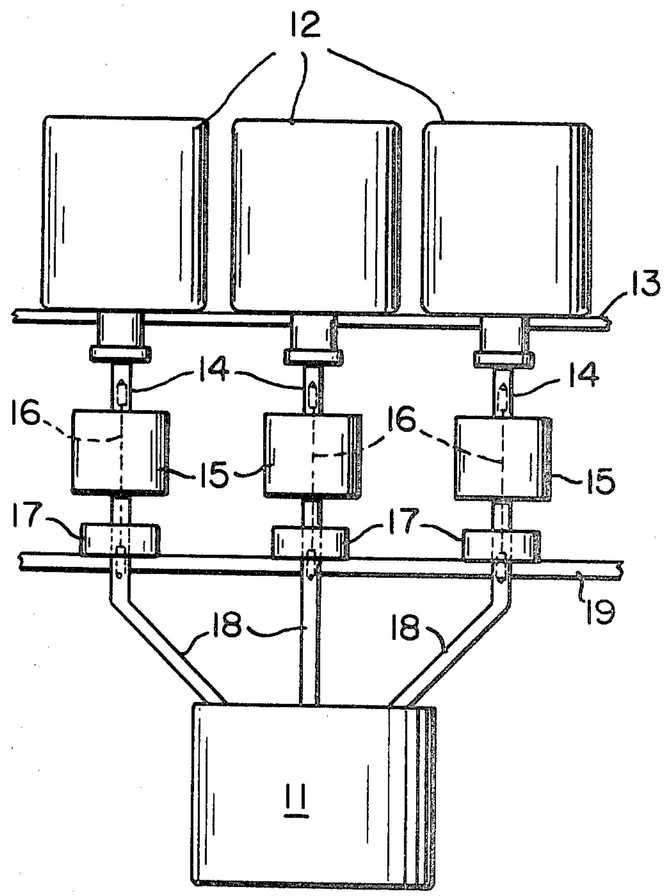
ABSTRACT: A plurality of bottles containing different chemicals are mounted in a cabinet which also contains a tape-operated mechanism that controls the opening and closing of valves that govern flow of the chemicals from the bottles to associated measuring chambers and from the measuring chambers to a reaction vessel. A time-delay mechanism comprising a pulse-generating clock, bimetallic switch arms and heating coils governs the step-by-step advance of the tape. A paddle for mixing the chemicals is rotatably mounted in the reaction vessel; and when a particulate material is used, the paddle may be foraminous and hold the particles.



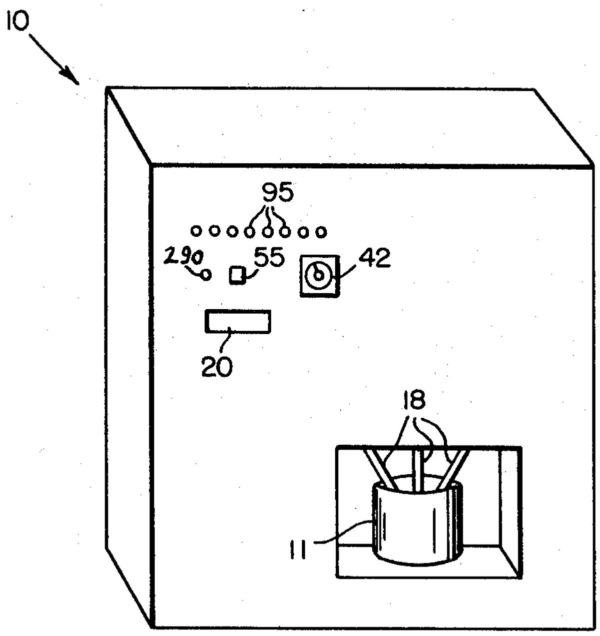


FIG. 1

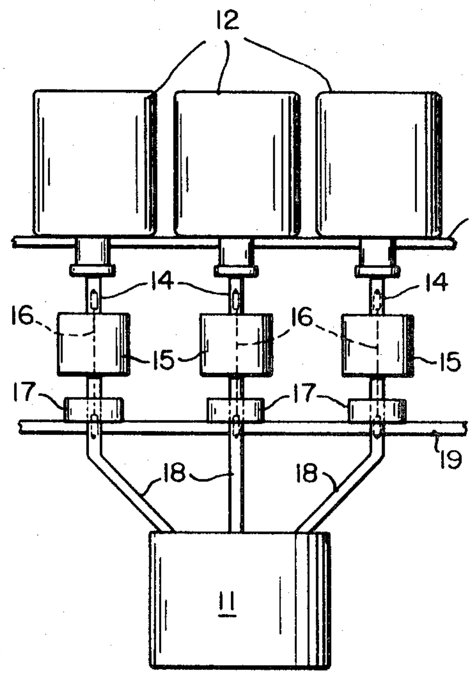


FIG. 2

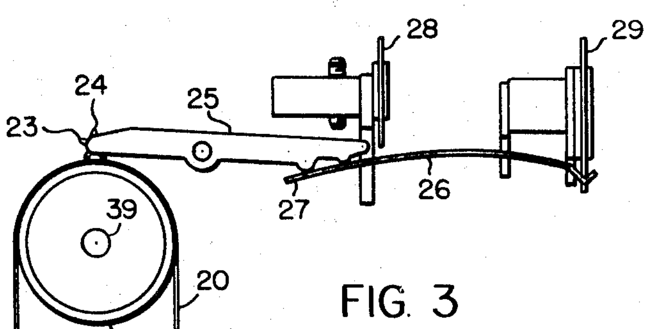


FIG. 3

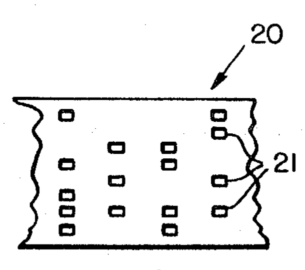


FIG. 5

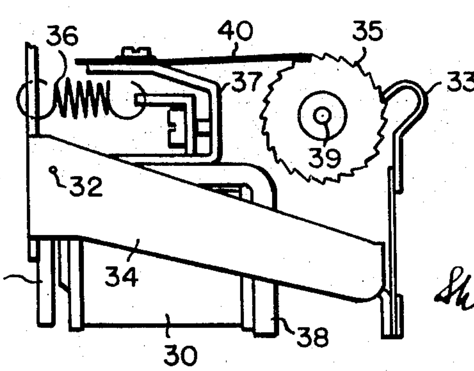


FIG. 4

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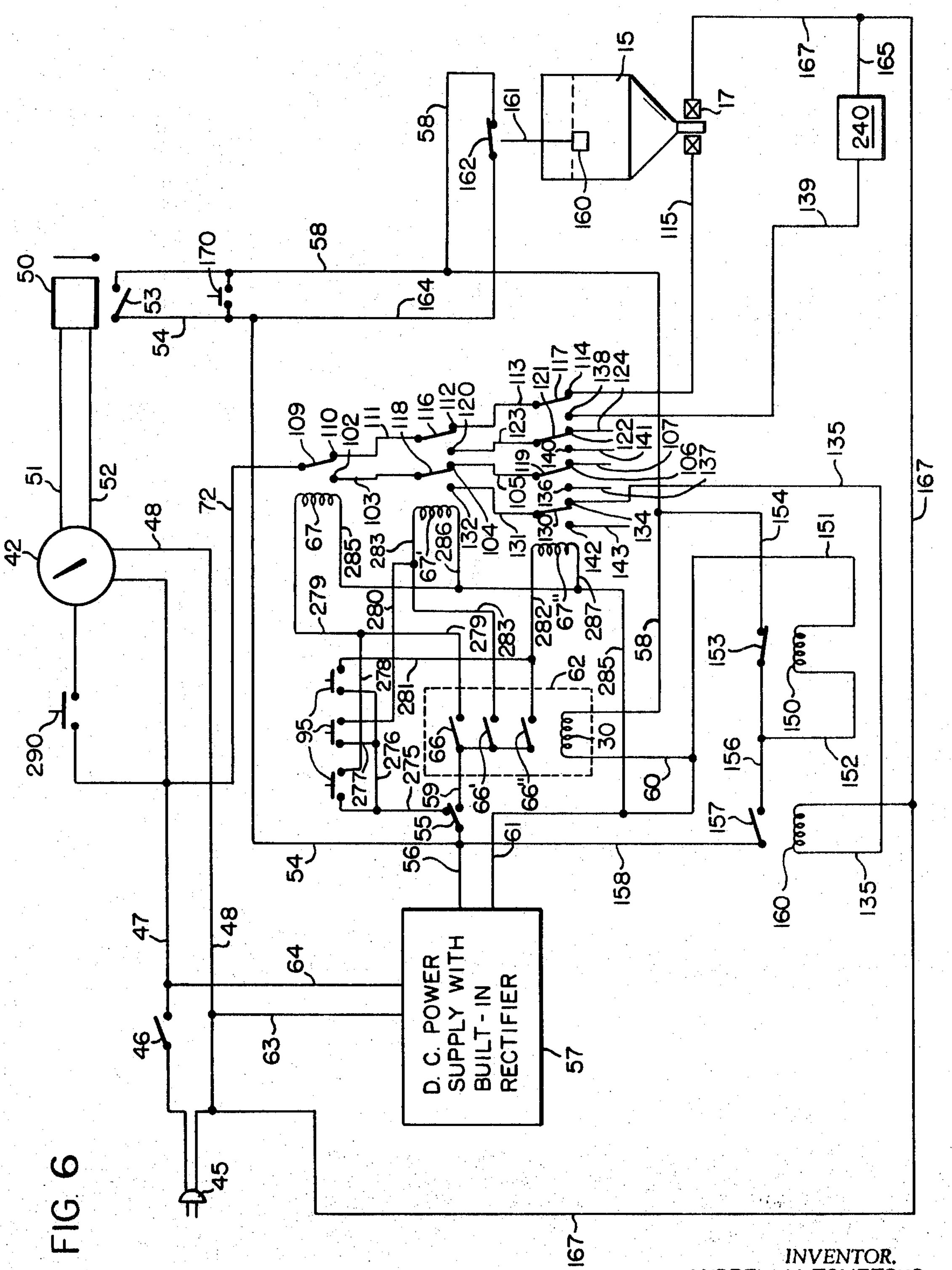

FIG 6

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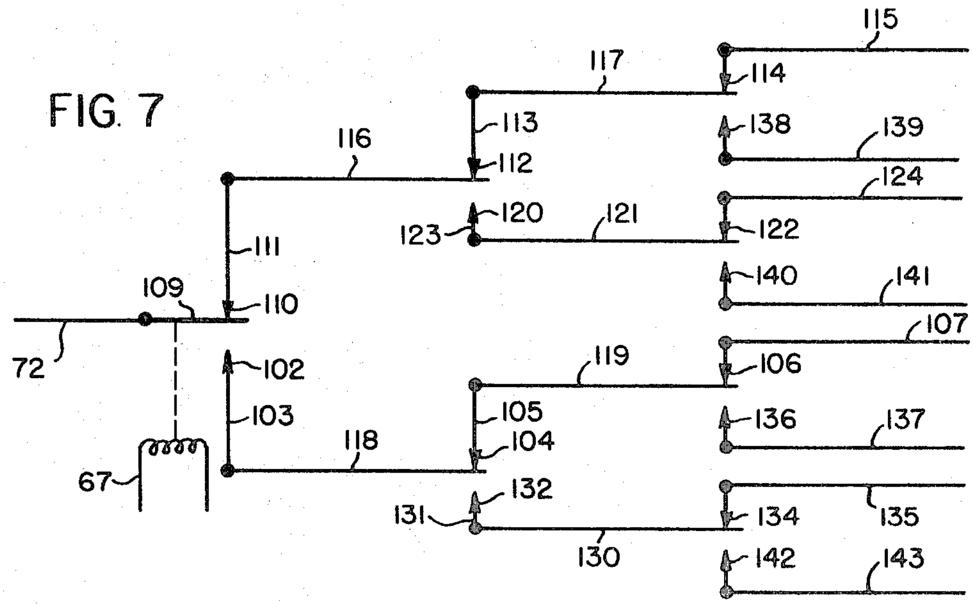
FIG. 7



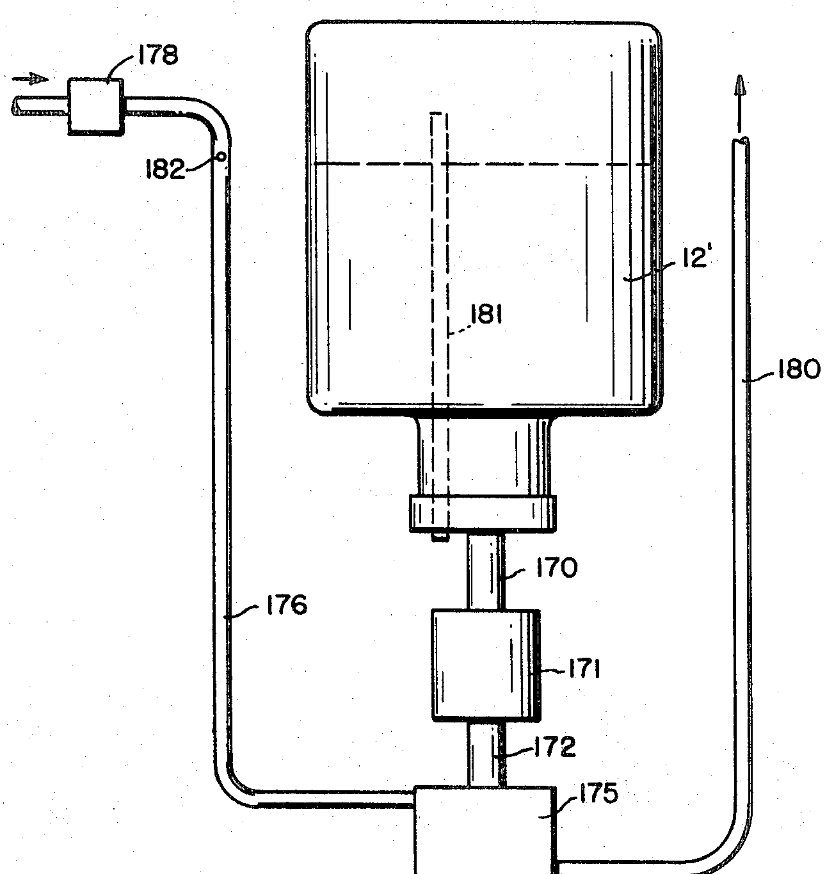


FIG. 8

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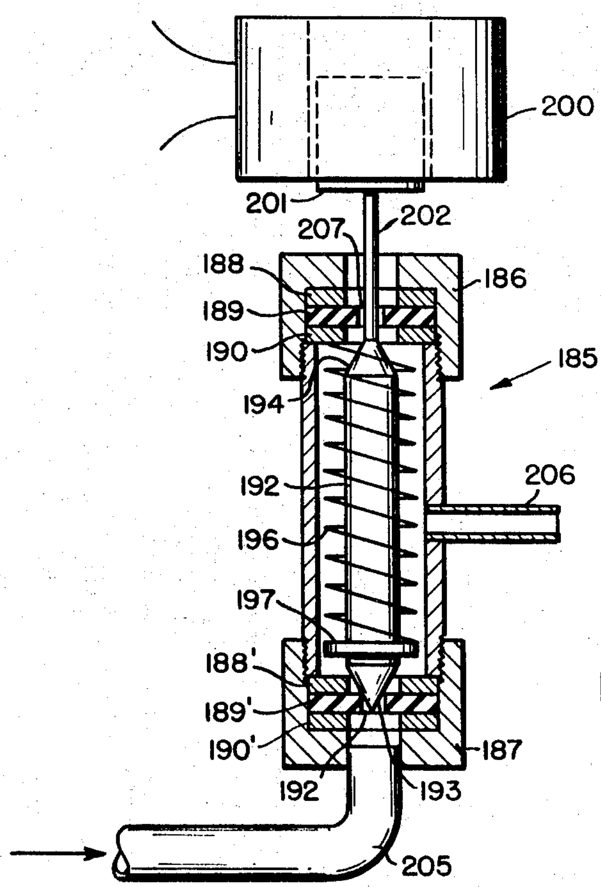

FIG. 9

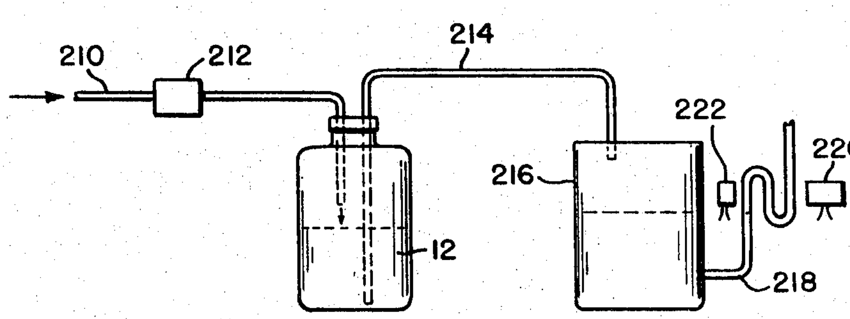

FIG. 10

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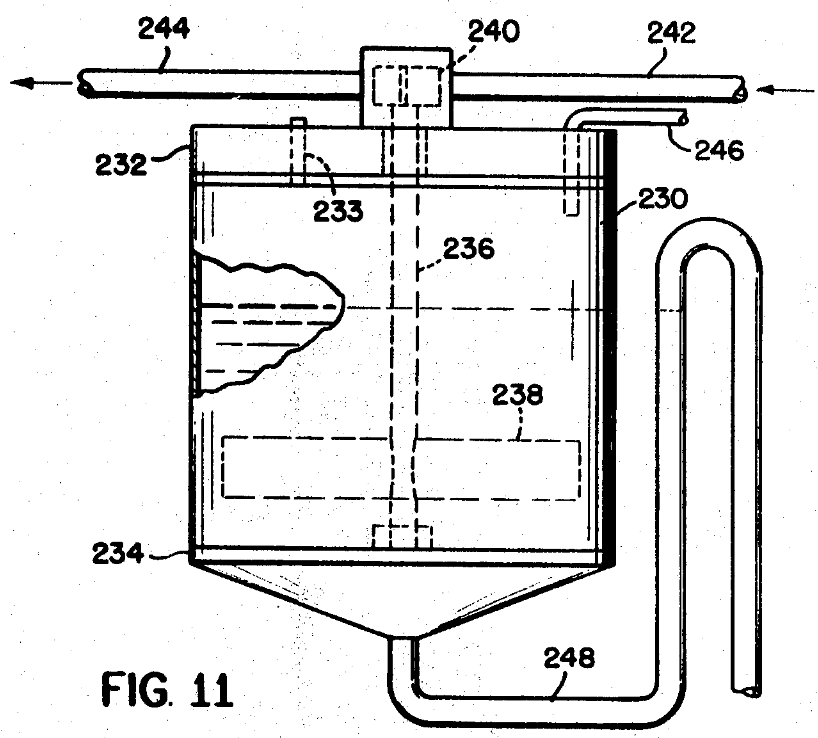

FIG. 11

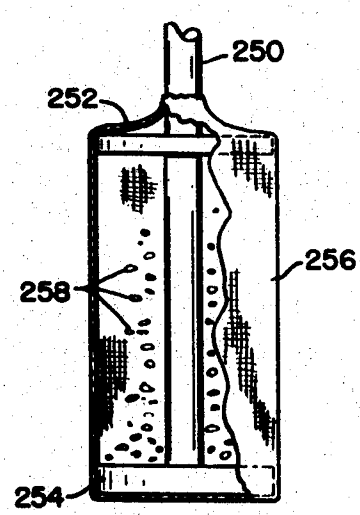

FIG. 12

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AUTOMATIC CHEMICAL REACTION SYSTEM

The present invention relates to apparatus for effecting chemical reactions.

In the chemical laboratories of colleges, and even in the chemical research laboratories of many corporations, the various chemicals that the student or researcher may use in a series of tests or experiments are stored in bottles on open shelves or in ordinary storage cabinets, and the student or researcher has to take the bottles from their storage places, measure out the amount of each chemical required for a test or experiment, return the bottles to their places of storage, and mix the chemicals together. If various chemicals are to be added to the reaction mixture at different times in an experimental or test run, the student or researcher has to be very careful about observing the time lapses and be prompt upon elapse of a time period to perform the next operation required. All this is burdensome, and time-consuming and is especially irksome to a student.

Previously, moreover, separate apparatus had to be used for degradation of a chemical from that used for synthesis thereof.

A primary object of this invention is to provide apparatus for automatically controlling and effecting chemical reactions.

Another object of this invention is to provide apparatus of this character which will relieve the student or researcher of the tedium of continuously watching a long experiment during progress thereof.

In a more specific aspect, the invention has for its purpose to provide apparatus for automatically carrying out the numerous steps involved in either the chemical synthesis or degradation of complex compounds such as polypeptides, and related areas of chemistry.

Other objects of the invention will be apparent hereinafter from the specification and from the recital of the appended claims, particularly when read in conjunction with the accompanying drawings.

In the drawings:

FIG. 1 is a front elevation of a cabinet for housing equipment built in accordance with one embodiment of this invention;

FIG. 2 is a fragmentary rear view looking into the cabinet and showing one arrangement of vessels for holding various chemicals, and the measuring chambers for controlling the volumes of chemicals effluent from each vessel, and the reaction vessel into which the chemicals are flowed, and the walls of the cabinet having been removed for clarity of illustration;

FIG. 3 is a fragmentary, more or less diagrammatic view illustrating the construction and operation of a reader, such as may be employed in this apparatus, for sensing the tape that controls the sequence of operations of the apparatus;

FIG. 4 is a fragmentary view illustrating one means for driving the tape;

FIG. 5 is a fragmentary view of a portion of typical tape used in controlling the sequence of operations;

FIG. 6 is an electrical diagram showing one way in which the apparatus may be wired to accomplish its purpose;

FIG. 7 is a fragmentary electrical diagram further illustrating the manner in which the several solenoids, which operate the control valves for the metering chamber are operated;

FIG. 8 is a more or less diagrammatic view illustrating a modification, showing how a liquid chemical may be fed to the reaction vessel;

FIG. 9 is a fragmentary view, illustrating somewhat diagrammatically another way of supplying a chemical to the reaction vessel;

FIG. 10 is a fragmentary diagrammatic view illustrating still another way of controlling liquid flow to a measuring chamber;

FIG. 11 is a view illustrating a modified form of reaction vessel provided with a mixing paddle; and

FIG. 12 is a view on an enlarged scale of a preferred form of mixing paddle for use when, for instance, a particulate catalyst is to be employed in a reaction.

Referring first to the embodiment of the invention shown in FIGS. 1 and 2, the apparatus is illustrated as housed in a cabinet 10 which contains the vessel 11 in which the chemical reaction takes place. The chemical reagents are stored in liquid or powder form in bottles or other suitable containers 12 supported in inverted position on a shelf 13 above the reaction chamber. The desired amount of each chemical, which is to be employed in a particular reaction, is first introduced into a measuring chamber 15 from a container 12 through a tube 14. From the measuring chamber the measured quantities of liquid or powder flow through solenoid-operated valves 16, such as disclosed in my pending patent application Ser. No. 808,626, into the reaction vessel 11. There the several chemicals from the several measuring chambers are mixed and/or reacted to produce the desired compound or composition.

The valves 16 have, as disclosed in application Ser. No. 808,626, sealing portions at opposite ends. The sealing portion at the lower end of a valve may be seated by gravity, and, when seated, closes off communication between the measuring chamber 15 and the associated duct 18 that leads to reaction vessel 11. The sealing portion at the upper end of a valve 16 is adapted to close off communication between a bottle 12 and the associated measuring chamber 15 when the valve is raised to its upper position. The valve is raised to its upper position when an associated solenoid 17 is energized. The solenoids 17 are supported on a shelf 19 of the cabinet 10 and surround the upper ends of tubes 18 and the lower ends of valves 16 which are slidable in these tubes. The distance between the upper and lower sealing portions of a valve 16 is less than the distance between the respective seats for those portions, so that when a valve is in its lower position, flow of a chemical from the measuring chamber associated with that valve is stopped but the chemical is free to flow from the associated bottle into the measuring chamber. On the other hand, when a valve is in its upper position, the chemical is free to flow from the associated measuring chamber into the reaction vessel but flow of the chemical from the associated bottle into the associated measuring chamber is stopped.

The valves 16 are normally in their down positions to retain the chemicals within the measuring chambers 15, while permitting flow of the chemicals from the bottles 12 into the measuring chambers. When the associated solenoid coil 17 is energized, however, the associated valve 16 will be lifted; and the chemical in the associated measuring chamber will flow from a measuring vessel 15 into the reaction vessel. The amount of a chemical that is delivered at any one time from a measuring chamber 15 into the reaction vessel 11 is determined in this embodiment of my apparatus, therefore, by the time the associated valve 16 is in its lower position.

The volume of liquid to be delivered from a measuring chamber may be determined, however, as will be described in more detail later, by a float 160 (FIG. 6) or other level determining device which, when the desired level of liquid in a measuring chamber is reached, will, through an electrical circuit, deenergize a solenoid to open a valve in a measuring chamber, and allow the measured amount of fluid or powder to flow into the reaction vessel 11.

The amount of a particular chemical to be delivered to the reaction vessel can also be controlled by a photoelectric cell circuit which will open a valve, when the fluid or powder reaches a predetermined level in a measuring chamber, to allow the chemical to flow from the measuring chamber into the reaction vessel. Any other suitable mechanism may also be employed for this purpose.

The valves, which control flow of a chemical from a measuring chamber to the reaction vessel in the various instances mentioned above, may be solenoid-operated, just as is valve 16. The firing of the several solenoids 17 for opening of these valves may be controlled by a tape reader of conventional construction. A perforated tape 20 (FIG. 5) of conventional form having holes 21 (FIG. 4) in it arranged in rows, that extend transversely of the direction of movement of the tape, controls the sequence of the operations of the apparatus. In the instance shown, the tape 20 has a maximum of eight holes 21 in a row.

The tape is advanced over roller 22 (FIG. 3) by any suitable means; and star wheels 23 having teeth 24 are disposed to sense the holes in the tape. Each star wheel (there are eight required for an eight-hole tape) is held against the tape 20 by a sensing arm lever 25 under the urging of a flexible wire contact 26 and leaf spring 27. As the tape is advanced, each star wheel slides along the surface of the tape on two of its five points or teeth 24. When a hole in the tape is encountered, the star wheel rolls into the hole. This allows the associated wire contacts to close against the common plate 28. Electrical connection is thus established between the common plate 28 and a terminal pin 29. This closes the circuit to the solenoid 17 which operates the associated valve. In other words, when a position on the tape is read, that is, when a star wheel drops into a hole on the tape, or a plurality of star wheels drop into several holes on the tape, the corresponding solenoid or solenoids 17 will fire. In effect, then, the number of holes on the tape is decoded and translated into physical operation by the bank of relays.

The tape program 20 can be produced on a conventional computer.

One method of feeding the tape is illustrated in FIG. 4. When drive coil 30 is energized, armature 31 is attracted and pivots about point 32, causing pawl 33, which is carried on the free end of an arm 34, that is secured to the armature, to engage the next tooth on ratchet wheel 35. When the coil 30 is deenergized, coil spring 36, which is secured at one end to the armature and at its other end to a bracket 37, that is fastened to the coil holder 38, returns the armature and stops the ratchet wheel. The ratchet wheel is fixed to the shaft 39 to which the tape roller 22 is secured. A spring dog 40, which is secured to bracket 37, prevents reverse movement of the ratchet.

The tape will advance periodically as determined by the pulse generating clock or reset timer 42 (FIGS. 1 and 6). This clock will be set for the appropriate time between pulses. At the end of a timing cycle a pulse is generated by the clock which will energize coil 30 for a predetermined period, and then deenergize it again. This will advance the tape to the next position.

The pulse emanating from the clock may be 110 volts AC. The reader is set to operate at, for instance, 24 volts DC. The AC pulse is converted to a DC pulse by using the initial AC pulse to energize coil 30 and a relay coil 59 (FIG. 6) which makes secondary DC contacts. The reader will advance one hole for each pulse.

The reader has, as stated, eight positions (holes) across the width of the tape. Five of these holes or positions are used for operating the solenoids 17 that control the valves for the bottles in the cabinet. The other three holes or positions are reserved for other purposes. For instance, one may control the motor that drives the stirrer in the reaction vessel, and the other two may control coils that produce different time delays in indexing of the tape. In this manner it is possible to carry out two or three operations at the same time.

When the star wheel in the reader encounters a hole 21, a contact is made in the position corresponding to the hole on the tape. DC current then flows through the tape reader to one of five relays, each of which corresponds to a position on the tape.

The relays used may have contacts which parallel the binary numbering system. Consequently, using five relays will make possible 32 combinations of relays.

It should be noted that the current arriving at a valve solenoid 17 has passed through all five relays, beginning with the first relay and continuing through the fifth relay to its target, that is, a solenoid-operated valve, stirrer, etc.

FIGS. 6 and 7 illustrate generally how the machine may be wired to accomplish its purpose.

Power may be obtained from a source of AC energy by plugging in a plug 45 and closing a manually operated main line switch 46. This supplies current to the reset pulse timer 42 through lines 47 and 48.

The reset pulse timer controls a relay 50 to which it is connected by lines 51 and 52. The AC pulse energizes the relay coil 50 which, when energized, closes the contact 53. This closes a circuit from a DC power supply 57, with a built-in rectifier, to coil 59 of reader 62 through line 56, line 54, contact 53, line 58, coil 30 (FIGS. 4 and 6), lines 60 and 61. Switch 55 is closed to connect lines 54 and 56 for automatic operation of the apparatus. Manual operation can be effected when this pushbutton switch 55 is open.

The DC power supply is connected by lines 63 and 64 to the power source. It has built-in rectifiers to convert the AC current from the power source to DC current.

The manner in which the several solenoids 17, that operate the several valves 16, are wired, is illustrated in FIG. 7.

There are eight switch arms 66 in the reader 62. Only three have been illustrated in FIG. 5; but the circuit to each is made by operation of the associated respective star wheel 23. When a star wheel 23 makes contact through a hole in the tape 20, it causes the associated contact 29 to close the associated switch arm 66, 66′, 66″. This causes the associated coil 67, 67′, 67″ to be energized.

In the position shown in FIGS. 6 and 7, the current flows from line 72, through contact 109, terminal 110, line 111, movable contact 116, terminal 112, line 113, movable contact 117, terminal 114, and line 115 to one of the solenoid coils 17. When the coil, which shifts switch arm 109 is energized, however, movable contact 109 makes a circuit through terminal 102, line 103, movable contact 118, terminal 104, line 105, movable contact 119, terminal 106, and line 107 to another of the solenoids 17.

When the contact 116 is shifted by the associated coil into engagement with the terminal 120, the circuit is through terminal 120, line 123, movable contact 121, contact 122, and line 124 to another of the solenoids 17. When contacts 109 and 102 are in engagement and the contact 118 is shifted into engagement with the terminal 132, the circuit is from line 72 through contact 109, terminal 102, line 103, contact 118, terminal 132, line 131, contact 130, terminal 134, and line 135 to another of the solenoids 17. When the contact 119 is shifted into engagement with the terminal 136, and line 137 to another of the solenoids 17. When the contact 116 is in engagement with the terminal 112, and the contact 117 is in engagement with the terminal 138, the circuit is made to another solenoid, through the lines 139 and 165, for instance the solenoid which operates the switch which closes the circuit to the motor that drives the stirrer that mixes the chemicals in the reaction vessel 11 (FIG. 1). When the contact 121 is shifted into engagement with the terminal 140, and contact 109 is in engagement with terminal 110, and contact 116 is in engagement with the terminal 120 and contact 121 is in engagement with terminal 140, the circuit is made through the line 140 to another of the solenoids 17. When the contact 109 is in engagement with the terminal 102, and the contact 118 is in engagement with the terminal 132, and the contact 130 is in engagement with the terminal 142, a circuit is made to the solenoid 17 controlling another valve 16 or device through the line 143. Thus, by actuating the various contacts of the several relays of the reader through the operation of the several star wheels 23, the different solenoids 17 can be energized to supply different chemicals in measured amounts to the reaction vessel 11, or to actuate other devices such as a stirrer.

The amount of each chemical delivered to the reaction vessel is determined by a time delay, a measuring chamber, photocell, and/or a liquid level gauge in the associated measuring vessel.

To control the length of time an operation is to be performed, a pair of bimetallic switches 153 and 157 are provided. Switch 157 is a normally open switch while switch 153 is a normally closed switch. Switches 153 and 157 are controlled by heating coils 150 and 160. When contact 109 is engaged with terminal 102, contact 118 is engaged with terminal 132, and contact 130 is in engagement with terminal 134, AC current is supplied to heating coil 160 through lines 162 and

167; and when coil 160 heats up sufficiently switch 157 is closed. This establishes a circuit to coil 30 from line 56 through line 158, now-closed switch 157, line 156, normally closed switch 153, lines 154 and 58, 60 and 61.

Motor 30, when energized, advances the tape 20.

Heating coil 150 is connected to DC lines 56 and 61 by lines 151, 152, 156, switch 157, when closed and line 158. When heating coil 150 heats up sufficiently, the bimetallic switch arm 153 is opened. This breaks the circuit to motor 30 and the tape advance is stopped. When this coil cools down, switch arm 153 closes again. The bimetallic switch arms, therefore, control the amount of advance of the tape each time motor 30 is actuated.

The reader has a ratchet so that when the normally open switch 157 is closed, it presets the pawl, while when the normally closed relay is broken, it advances the ratchet wheel 35; and the tape 20 moves to the next position.

With the apparatus so far described it will be seen that upon closure of the mainline switch 46 and of switch 55, the pulsing timer 42 will be set in operation and the apparatus will be set into operation to deliver for such intervals as the timer closes relay switch 53 chemicals from preselected bottles 12. The bottle or bottles selected for delivery of chemicals during a particular interval will be determined, of course, by what switch arms 66, 66', 66'' are closed by associated pins 29 (FIG. 3) during that interval, to energize associated coils 67, 67', 67'' and associated solenoids 17. When the pulse interval elapses, drive coil 30 will be energized for a time determined by bimetallic switches 157, 153 to advance the tape 20 so that star wheels 23 (FIG. 3) will drop into new holes in the tape to energize anew selected solenoids 17.

In the intervals of pulsing energization of relay 50, not only will the selected solenoids 17 operate to cause delivery of selected chemicals to the reaction vessel, but also the stirrer, etc. may be actuated as described.

For manual control of the operation of the apparatus, the switch 55 may be moved to its open position, shown in FIG. 6, to bypass the reader. In this case, a DC pulse energizes the coils of one or more of the five binary relays. Note that the clock cycle uses AC current to energize the coil; whereas the binary relays use DC current to channel the AC current. The several solenoids 17 may be individually energized at will by the respective associated push buttons 95 which are then connectable with line 56 from the DC power supply 57 through lines 275, 276, 277, and which are connectable with the several coils 67, 67', 67'' through lines 278, 279, 280, 281, 282, 283, so that when switches 66, 66', 66'' of the reader 62 are closed by operation of pins 29 (FIG. 3) power will be supplied to the coils 67, 67', 67'' to energize the associated solenoids 17. Coils 67 are connected to one another by lines 285, 286 and 287, and are connected with line 61 from the DC power supply 57 by line 285.

A manually operable pushbutton switch 170 permits manual control over advance of the tape 20 through manual control of the energization of tape drive coil 30; when switch 170 is closed, a circuit is made from DC power supply 57 through lines 56, 54, 58, 60 and 61.

Each measuring vessel is equipped with a liquid level gauge or some means wired in the circuit of the apparatus for breaking the operating circuit of the apparatus when the liquid in the measuring vessel has risen to a predetermined level. This gauge may take the form of a simple float as shown at 160 in FIG. 6, which has a rod or stem 161 secured to it that opens switch 162 when the liquid in the measuring vessel 11 has risen to the desired predetermined level.

Instead of the arrangement shown in FIG. 1, a fluid metering system such as illustrated in FIG. 8 may be employed. Here a bottle 12' for storing a chemical is shown. It is connected by tube 170 to a conventional check valve 171 which is connected by a pipe 172 to a measuring chamber 175. The check valve, which could be mounted in the neck of bottle 12', if desired, permits flow of the liquid in only one direction.

Either the measuring chamber, or the reaction vessel, or both, can be emptied by using air or an inert gas, such as nitrogen, to force the liquid from the chamber or from the reactor. 176 denotes a tube for supplying air or nitrogen or other inert gas to measuring chamber 175. Mounted in line 176 is electrically operated valve 178. A tube 180 leads from the measuring chamber to the reaction vessel. An air vent tube 181 is mounted in bottle 12' to extend up above the liquid in the vessel.

When the star wheel 23 (FIG. 3) corresponding to a particular container 12' makes contact it closes a circuit to electrically-operated three-way, normally closed valve 178. The valve will then open and the gas under pressure will flow from tube 176 into measuring chamber 175 to force the liquid out of the measuring chamber through line 180 to the reaction vessel. The time-delay relay 153–157 in the circuit, as already described with reference to FIG. 6, will be set for a desired period of gas flow and then shut off. Hence a desired volume of liquid will be obtained from the measuring chamber.

Of course, instead of electrically operating valve 178, it may be manually operated.

FIG. 9 shows still another form of air valve which may be used in the apparatus of the present invention.

This valve comprises a T-tube 185 which has caps 186 and 187, respectively, threaded on the two ends of its transverse leg. Interposed between each cap and the confronting end of the pipe, in each case, are a brass or other metal washer 188, a rubber washer 189, and a brass washer 190. These are held fast and airtight by the caps. A valve member 192, which is formed at its lower end with a conical surface 193 and intermediate its ends with a second conical surface 194, is mounted reciprocably in the pipe 185. A coil spring 196, which surrounds the valve 192, within the pipe 185, and which seats at its lower end against a flange or washer 197, serves to urge the valve member constantly to its lower position to close the opening 198 in the rubber washer 189'. The valve is adapted to be lifted off this seat by energizing the electromagnetic coil 200. A steel block 201 has threaded connection with the rod 202, which forms the upper end of the valve member 192.

Air, or nitrogen, is admissible to the valve body 185 through the tube 205. The arm 206 of the T-unit can be connected to the metering chamber or to a supply bottle. No gas will pass from the tube 205 into the arm 206, however, until the star wheel associated with the unit closes the circuit to energize the coil 200. The time-delay controls the time of deenergization of the coil. When the valve is lifted, the orifice 207 at the upper part of the valve unit, which is normally open, will close; and the gas will exit through the side arm 206.

Still another arrangement is illustrated in FIG. 10. Here the air, or other gas, is supplied to the bottle 12 through a tube 210, which is fitted with a valve 212 to control the flow of the gas therethrough. The tube 210 is connected to enter into the bottle 12, and force liquid therefrom through another tube 214 which conveys the liquid to the measuring chamber 216.

Connected to the measuring chamber 216 is a tube 218, which connects with a U-shaped column 217 that contains iodine or other dark fluid. A light source 220 is disposed at one side of the column; and a photoelectric cell 222 at the opposite side. When the iodine column rises high enough to shut off light from the photoelectric cell, the switch 162 (FIG. 6) will be tripped to advance the tape roll 22 (FIG. 3).

In the case of either the arrangement of FIG. 9 or that of FIG. 10, it is essential to have an immediate release of the air pressure that builds up in the bottle in order to have a sharp dispensing "cutoff." Thus, when the coil 212, or the coil 200, is inactivated, the spring 196 (FIG. 9) will return the valve 192 to its original position blocking airflow into the valve through the orifice 198, but permitting release of pressure from the bottle by directing the air through the orifice 206 and out through the orifice 207.

In some instances, it may be desirable to use a paddle for mixing the chemicals in the reaction vessel. Such an arrangement is shown in FIG. 11. Here, the reaction vessel is denoted at 230. It is closed at its top by a cap 232; and may have a sintered glass bottom 234. Journaled at top and bottom in the cover 232 and bottom plate 234 is a shaft 236, to which is secured a paddle member 238. The shaft may be rotated by a gas-driven turbine 240, which is secured to the top of the shaft. Gas for actuating the turbine may be supplied through the tube 242, and exhausted through the tube 244. A capillary air inlet tube 246 may be mounted in the cap 232 to extend into the reaction vessel to permit flow of liquid therefrom.

Either the reaction vessel 11, or the measuring chamber 15, or both, can be emptied by applying a vacuum which will cause the material therein to be deposited in a suitable container or fraction collector. In the latter case, after the filtration step, the fraction collector may be moved to place an empty container in the receptacle spot. Thus, a vacuum tube 248 may be connected to the bottom of the vessel 230 to draw liquid therefrom.

In some instances, where a particulate catalyst is employed in the reaction, a paddle member may be used such as shown in FIG. 12. This paddle member consists of the shaft 250, which has axially spaced flange portions 252 and 254 thereon, that are covered by a nylon mesh or other foraminous envelope 256. Resin particles 258, may be contained within the envelope, so that as the nylon spool is whirled around, the liquid will flow through the mesh of the nylon envelope, without having the particles scattered around in the reaction vessel. It is easy, then, to lift the mixing spool out of the vessel, when the reaction is done, and substitute another spool, or other particulate material, or, if no particulate catalyst is to be employed, a paddle therefor.

The apparatus can also monitor and control the temperature of the reaction vessel.

A pilot light may be provided to indicate the present state of the system; and a counter may be employed to show the number of cycles that have transpired from start.

A "fail-safe" device may also be incorporated in the circuit which will warn the operator if a problem develops in the apparatus.

In use, particularly in a college or university laboratory, each student, or team of students, will have a cabinet assigned to him in which will be stored the chemicals he or she requires for the series of experiments he is to conduct. In a corporate research laboratory similarly each researcher will have his or her cabinet containing the chemicals required in his or her work.

To operate the apparatus, the operator has a tape 20 punched to give the desired program or sequence of operations; and it will be placed on the roll 22 in the cabinet. The program may be generated on a conventional computer. The pulse generating clock 42 (FIG. 6) will be set for the appropriate time between pulses. For setting timer 42, a reset button 290 may be used.

The apparatus may be set in operation by closing line switch 46. As the roll 22 revolves, the star wheel rides on the tape until it drops into a hole. This closes a circuit to one of the five relays controlling the valve solenoids 17 (FIG. 2), which will cause the associated valve or valves 16 to be shifted to dispense the measured quantity of chemical or chemicals from the measuring chambers 15 into the reaction vessel 11. At the end of a timing cycle (controlled by pulse generating clock 42) the tape 20 will be advanced by the mechanism shown in FIG. 4.

The chemicals in the reaction vessel may be stirred during the reaction; and where a particulate catalyst is used, a foraminous paddle 256 (FIG. 12), which contains the catalytic particles, may be employed to effect the stirring. The reaction vessel may be emptied by air or gas pressure or by applying a vacuum as shown in FIGS. 8, 9, 10 and 11.

Having thus described my invention, what I claim is:

1. Chemical reaction apparatus comprising
a plurality of containers for storing, respectively, different chemicals,
a measuring chamber associated with each container,
a first duct for conducting a chemical from a container to the measuring chamber associated with that container,
a reaction vessel,
a second duct connecting each measuring chamber with the reaction vessel,
valve means controlling flow of a chemical from each container to the associated measuring chamber through the associated first duct and flow of the chemical from a measuring chamber to the reaction vessel through the associated second duct, and
means for automatically controlling the periods and sequence of operation of said valve means,
said automatic control means comprising solenoids associated with the several said valve means and energizable to operate said valve means, and punch tape controlled means for energizing the solenoids, and controlling the periods and sequence of their energization.

2. Chemical reaction apparatus as claimed in claim 1, wherein said automatic control means includes means controlled by the level of liquid chemical in a measuring chamber for operating the valve associated with that chamber.

3. Chemical reaction apparatus as claimed in claim 1, wherein said tape-controlled means includes a punched tape having a plurality of perforations therein spaced from one another in the direction of movement of the tape, means for advancing the tape, and means for reading the tape by engagement with the perforations and controlling the energization of the solenoids.

4. Chemical reaction apparatus as claimed in claim 3, wherein said tape-advancing means is electrically operated and is controlled by a pulse-generating clock adjustable to determine the time between pulses and operative at the end of each time cycle to generate a pulse to actuate said tape-advancing means.

5. Chemical reaction apparatus comprising
a cabinet for storing containers holding different chemicals,
a measuring chamber associated with each container,
a duct for conducting a chemical from a container to the measuring chamber associated with that container,
a valve associated with each duct for controlling flow of the chemical from each container to the measuring chamber associated therewith,
means for automatically controlling the periods and sequence of opening of said valves, and
a duct associated with each measuring chamber for conducting fluid under pressure to each measuring chamber for emptying the measuring chambers, said automatic control means also controlling flow of the pressure fluid in each of the last-named ducts.

6. Chemical reaction apparatus comprising
a plurality of containers for holding, respectively, different chemicals,
a measuring chamber associated with each container,
a duct connecting each container to the associated measuring chamber, and
means for conducting fluid under pressure into each container to force a chemical out of a container into the associated duct,
a valve for controlling flow of the pressure fluid in each duct, and
automatic control means controlling opening and closing of said valve,
said automatic control means including means responsive to the level of chemical in each measuring chamber.

7. Chemical reaction apparatus as claimed in claim 6, wherein each level-responsive means includes a light source, a photoelectric cell operable by said light source, and means responsive to the level of chemical in each measuring chamber for interposing an opaque material between said light source and the associated photoelectric cell.

8. Chemical reaction apparatus comprising
a plurality of containers for storing, respectively, different chemicals,
a measuring chamber associated with each container,

a first duct for conducting a chemical from a container to the measuring chamber associated with that container,
a reaction vessel,
a second duct connecting each measuring chamber with the reaction vessel,
valve means controlling flow of a chemical from each container to the associated measuring chamber through the associated first duct and flow of the chemical from a measuring chamber to the reaction vessel through the associated second duct, and
means for automatically controlling the periods and sequence of operation of said valve means,
a paddle being mounted in the reaction vessel for rotation therein, and said paddle comprising a foraminous container for holding a particulate chemical therein.

9. Chemical reaction apparatus comprising
a plurality of containers for storing, respectively, different chemicals,
a measuring chamber associated with each container,
a first duct for conducting a chemical from a container to the measuring chamber associated with that container,
a reaction vessel,
a second duct connecting each measuring chamber with the reaction vessel,
valve means controlling flow of a chemical from each container to the associated measuring chamber through the associated first duct and flow of the chemical from a measuring chamber to the reaction vessel through the associated second duct, and
means for automatically controlling the periods and sequence of operation of said valve means,
said automatic control means comprising a tape, means connecting said valve means to said tape so that the tape in its movement controls the sequence and duration of operation of said valve means, and means for periodically advancing said tape.

10. Chemical reaction apparatus comprising
a plurality of containers for storing, respectively, different chemicals,
a measuring chamber associated with each container,
a first duct for conducting a chemical from a container to the measuring chamber associated with that container,
a reaction vessel,
a second duct connecting each measuring chamber with the reaction vessel,
valve means controlling flow of a chemical from each container to the associated measuring chamber through the associated first duct and flow of the chemical from a measuring chamber to the reaction vessel through the associated second duct, and
means for automatically controlling the periods and sequence of operation of said valve means,
a pulse timer controlling the periods of automatic operation of said valve means, a movable tape controlling selectively the valve means to be operated during an interval of operation of said tape through means operatively connecting the tape to said valve means, and means for determining the duration of operation of the valve means in any said period.

11. Chemical reaction apparatus comprising
a plurality of containers for storing, respectively, different chemicals,
a measuring chamber associated with each container,
a first duct for conducting a chemical from a container to the measuring chamber associated with that container,
a reaction vessel,
a second duct connecting each measuring chamber with the reaction vessel,
valve means controlling flow of a chemical from each container to the associated measuring chamber through the associated first duct and flow of the chemical from a measuring chamber to the reaction vessel through the associated second duct, and
means for automatically controlling the periods and sequence of operation of said valve means,
a tape, means for reading said tape, means for connecting said tape reader to said valve means to operate said valve means selectively under control of said tape reader, a pulse timer, and means for periodically advancing said pulse timer under control of said pulse timer.

12. Chemical reaction apparatus as claimed in claim 11, wherein said tape is a punched tape, and said reader comprises means engageable in the holes punched in said tape to complete an electric circuit for operating said valve means.

13. Chemical reaction apparatus as claimed in claim 12, having means for controlling the amount of advance of the tape on each pulsation of said timer.

14. Chemical reaction apparatus as claimed in claim 13, wherein said means for controlling the advance of said tape comprises a bimetallic switch, and means for heating the same.

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