

Nov. 17, 1970

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3,540,982

LABORATORY APPARATUS FOR EXPERIMENTAL WORK IN PULPING, BLEACHING
AND CHEMICAL TREATMENTS OF CELLULOSES

Filed Jan. 27, 1969

4 Sheets-Sheet 1

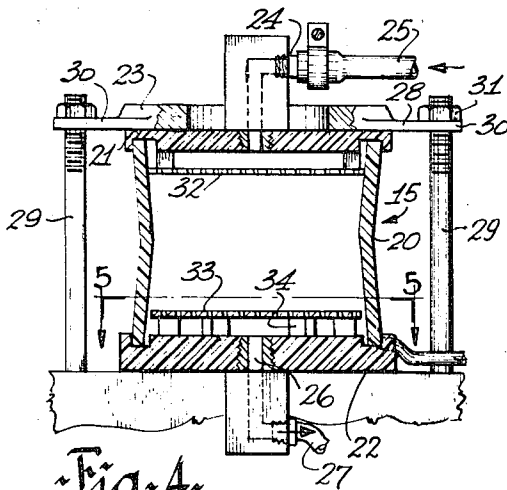


Fig. 4

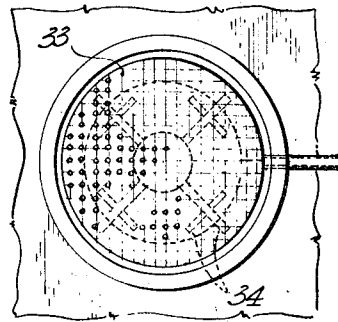


Fig. 5

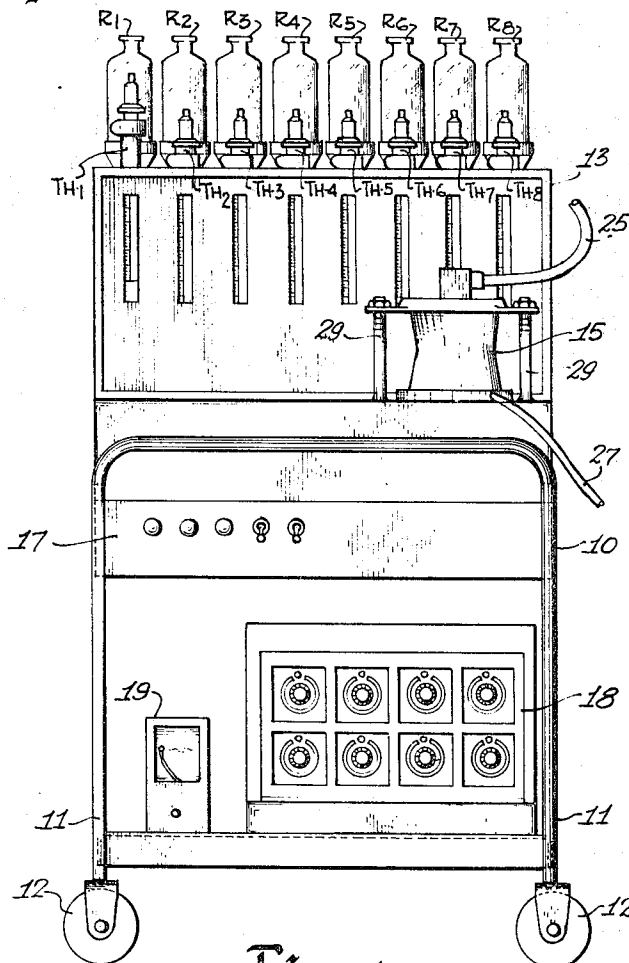


Fig. 1

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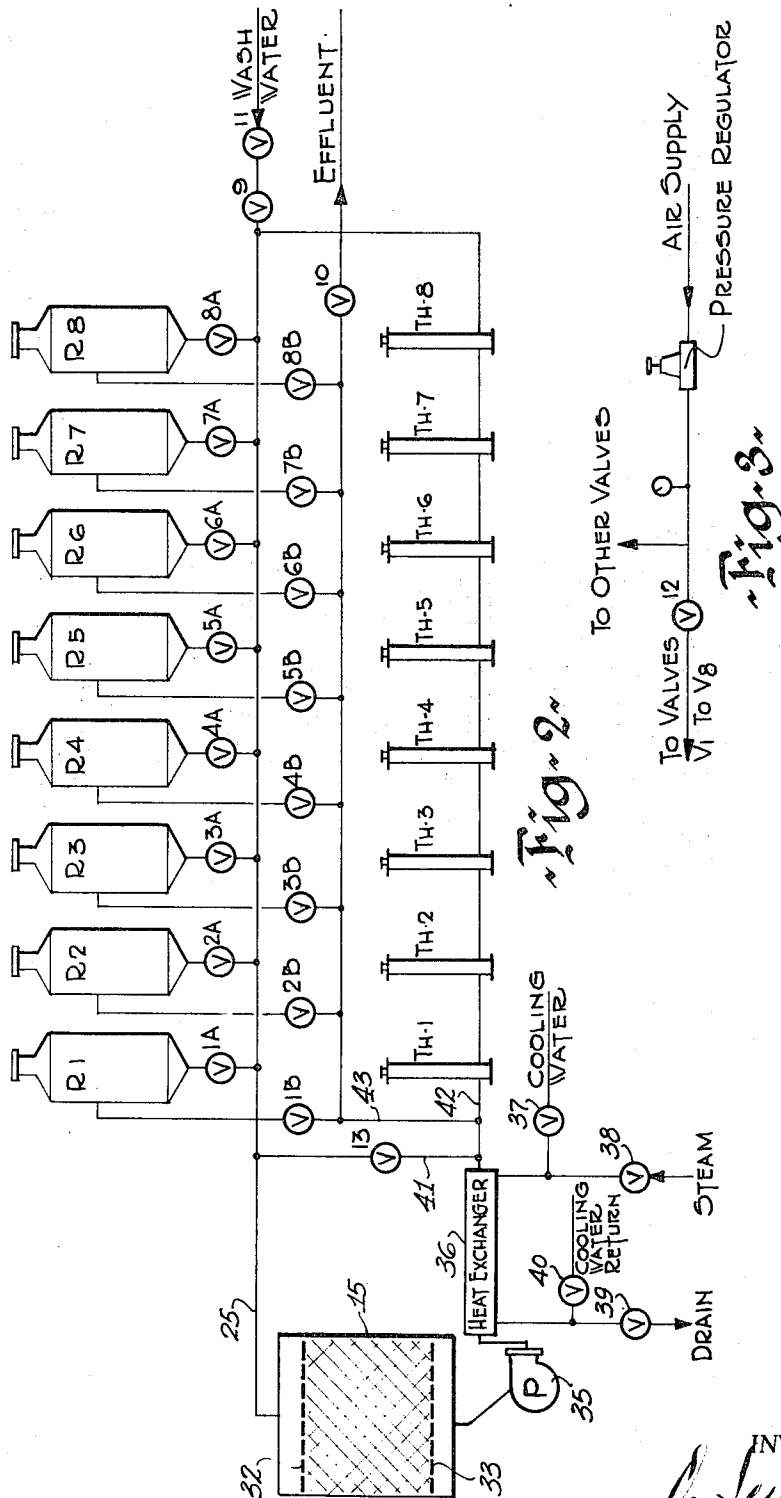
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Filed Jan. 27, 1969

4 Sheets-Sheet 2



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Filed Jan. 27, 1969

4 Sheets-Sheet 3

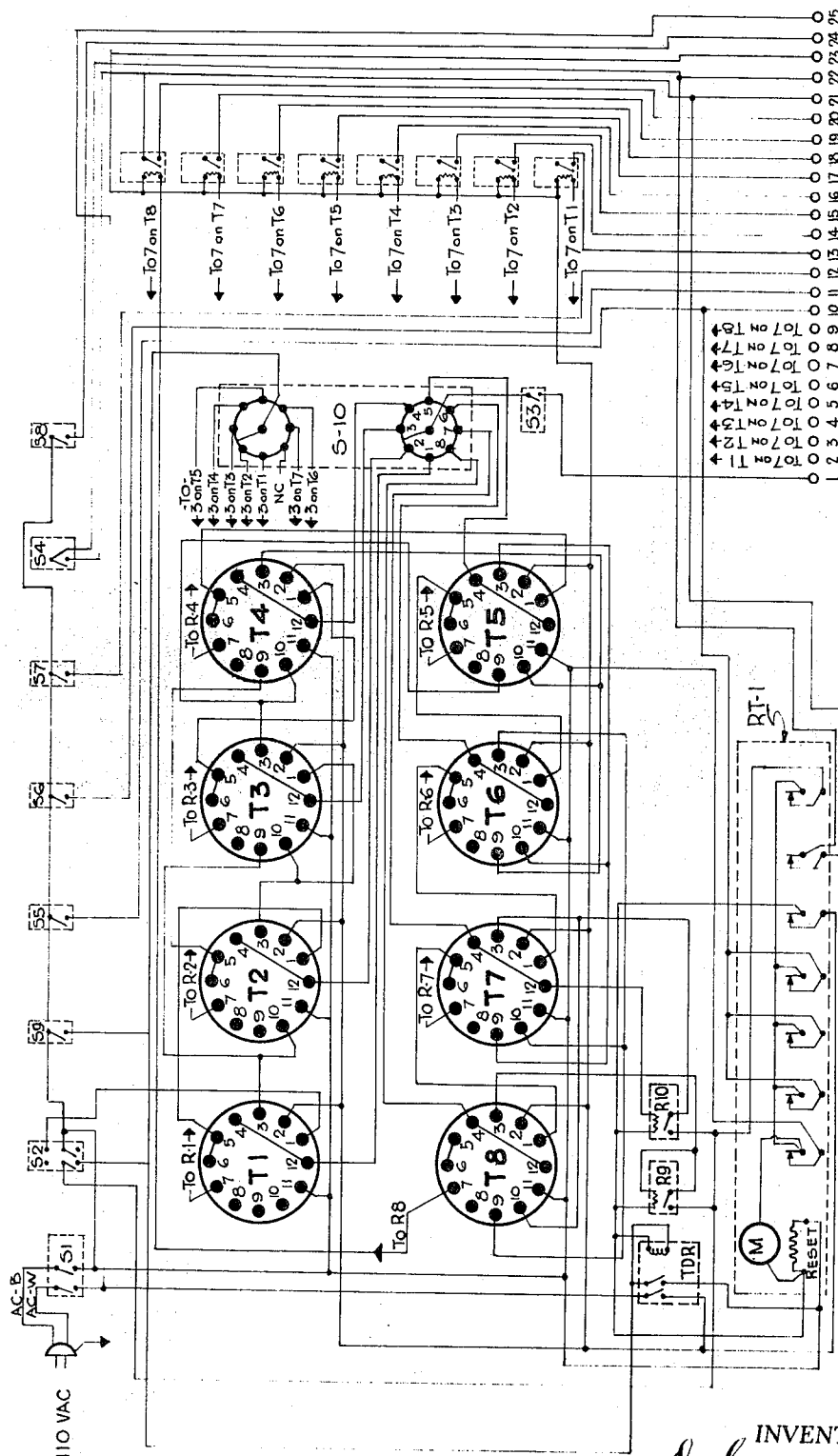


Fig. 6

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Nov. 17, 1970
LABORATORY A.
Filed Jan. 27, 1969

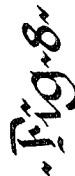
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TREATMENTS OF CELLULOSICS	

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LABORATORY APPARATUS FOR EXPERIMENTAL WORK IN PULPING, BLEACHING
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4 Sheets-Sheet 4



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3,540,982

**LABORATORY APPARATUS FOR EXPERIMENTAL
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Filed Jan. 27, 1969, Ser. No. 793,941

Int. Cl. D21

U.S. Cl. 162—253

6 Claims

ABSTRACT OF THE DISCLOSURE

The invention relates to a laboratory device suitable for conducting heterogeneous reactions on filterable solids and is particularly useful in situations where a sequence of reactions is desired with various reagents under different conditions of temperature and time. A particular field of usefulness is in experimental work in studies related to fields such as pulping, bleaching and chemical treatments of cellulose and other textiles.

The conventional method of conducting physical and chemical treatments on solids as practiced in the laboratory involves considerable manual manipulation. Typically, the procedure involves all or some of the following steps: (1) mixing of the solid with the required treating liquid by kneading either manually or with a mechanical stirring device, (2) enclosing the mixture in a suitable container for immersion in a temperature controlled bath for the requisite time, (3) manual transfer of the material after treatment to a filtering device (such as a Buchner funnel) for removal of the spent liquor, (4) manual washing of the solid with an appropriate solvent, and (5) repetition of steps (1) to (4) as required for additional treatments according to the desired sequence. The number of repetitions of treatments varies with the kind of work being done. In the bleaching of woodpulp, for example, the sequences range from one to eight stages in normal practice.

The laboratory work required in experimentation and testing related to the above is expensive and time consuming since it is essentially all done manually at the present time.

The present invention seeks to provide an experimentation apparatus suitable for conducting heterogeneous reactions, solvent treatments, extractive processes and similar operations on filterable solids. It proposes to carry out such work automatically with prior programming of any desired sequence of operations.

The invention thus resides in an apparatus for automatically conducting sequences of treatments on solids. Important features include:

- (1) an enclosed vessel where the reaction or treatment is effected,
- (2) a suitable filter within the vessel to retain the solid material therein,
- (3) a suitable pump to withdraw liquid from the vessel and to recirculate it to the vessel,
- (4) a multiplicity of reservoirs which can be connected into the recirculation line so as to introduce different chemicals as required,
- (5) a temperature regulating system, such as a heat exchanger in the recirculation line with associated equipment,
- (6) suitable connections to provide introduction of washing fluids and to discard spent liquor, and
- (7) a control system to arrange the desired sequencing of treatments as well as the required conditions for each,

2

The advantage of the equipment is that it eliminates the manual handling normally required in this kind of experimental work without sacrificing versatility needed to cope with different treatments and varying conditions.

The invention therefore comprises a laboratory apparatus which includes a reaction vessel having a reaction chamber, a filter plate dividing the chamber into a major portion and a minor portion, and a main circulation line having an inlet in the major portion and an outlet in the minor portion. A pump in the line provides circulation. A plurality of reservoirs for various chemicals is provided, the main circulation line having a plurality of branch lines each leading through one of the reservoirs and each having valve means for selective introduction into the main line. Temperature control means are provided to adjust the temperature of the materials supplied to the reaction vessel. An electrical programming device actuates any selected number of the valve means in any desired sequence.

The reaction vessel constitutes an important feature of the invention and preferably comprises a tubular glass member having end walls of resilient and chemical resistant material. An inlet connection is provided in one end wall and an outlet connection in the other. A perforated filter plate in the member is located in spaced adjacent relation to the outlet wall. Means for clamping the end walls in closed position on the member comprises a rigid plate engaging the outer surface of each end wall and retaining rods releasably clamping the plates together.

The invention will be described with reference to the accompanying drawings, in which

FIG. 1 is a front elevation of one form of apparatus in accordance with the invention,

FIG. 2 is a schematic diagram of the apparatus, FIG. 3 is a diagram of valve-operating air supply means,

FIG. 4 is a side elevation, partly in section, of the reactor vessel,

FIG. 5 is a plan view, partly in section, of the reactor vessel,

FIG. 6 is a wiring diagram of the programming device, FIG. 7 is a wiring diagram of the reactor system, and FIG. 8 is an enlarged view of the programmer.

As shown in FIG. 1, the apparatus may be assembled as a single, compact, substantially self-contained unit in a supporting frame 10 having legs 11 provided with casters 12 for ease of movement.

A housing 13 carried by the frame supports a plurality of reservoirs identified as R-1 to R-8, inclusive, and a reaction vessel 15. The housing also contains a plurality of thermoregulators identified as TH-1 to TH-8, inclusive, various operative connections, and a control panel 17. Also supported in the frame is a programmer 18 and a temperature recorder 19.

Reaction vessel 15 (FIGS. 4 and 5) preferably comprises a tubular member 20 which, as shown, is a standard section of industrial glass pipe with flat, ground end faces. A top end wall 21 and a bottom end wall 22 are formed of resilient and chemical-resistant material such as that sold under the trade name "Teflon" by E. I. du Pont de Nemours & Co.

Top wall 21 is fixed to an overlying plate 23, which may be metal, and is provided with an inlet connection 24 to which is connected a main circulation line 25 comprising chemical-resistant tubing such as that sold under the trade name "Nalgon" by Nalge Co.

Bottom wall 22 is provided with an outlet 26 to which is connected tubing 27.

Means for removably clamping the end walls 21 and 22 in closed position on the member 20 comprises a metal plate 28 on which wall 22 is seated, a pair of threaded

rods or posts 29 mounted on plate 28, ears 30 on plate 23 through which the posts extend, and nuts 31.

A perforated flow distributor plate 32 (preferably of Teflon) is carried by end wall 21 in spaced relation thereto.

A similar perforated filter plate 33 (also preferably of Teflon) is supported slightly above end plate 22 by ribs 34. It serves as a retainer filter for the solids in the material under treatment and permits liquid to flow to the outlet connection.

The described vessel assembly is of simple nature and requires only two retaining nuts to place it in closed operative condition. There are no restricted openings and the assembly is subject to ready and rapid dismantling for filling, emptying and cleaning.

Referring to FIG. 2, main line 25 is provided adjacent the outlet from vessel 15 with a circulation pump 35 and a heat exchanger 36. The heat exchanger has a valved connection 37 for cooling water, a valved connection 38 for steam, a valved drain 39 and a valved cooling water return line 40.

After leaving the heat exchanger, the flow is divided into three streams. The first is returned directly to vessel 15 through branch line 41 and valve V-13 for mixing by recirculation. The second flows through branch line 42 into contact with thermoregulators TH-1 to TH-8. The appropriate thermoregulator corresponding to the treatment stage being conducted is connected automatically to the electrical control that triggers the appropriate supply of coolant or heating fluid to the heat exchanger in order to achieve the desired temperature control. The third stream flows into a manifold 43 from which it may be led into any one of the reservoirs R-1, etc., or any selected number of such reservoirs in sequence, through valves V-1B, V-2B, etc. Flow from the reservoirs employed back to the main line is provided through valves V-1A, V-2A, etc.

Wash liquid may be flowed through the reactor vessel by means of valves V-9 and V-11 with release of effluent through valve V-10.

The valves employed are preferably of the air pressure actuating type. As shown in FIG. 3, air supply line 44 is provided with a pressure regulator 45, gauge 46, valved line 47 leading to valves V-1A and V-1B, etc., and line 48 leading to the other valves.

FIG. 4 illustrates a typical programmer wiring diagram for the apparatus. A plurality of timers T-1 to T-8 (one for each of the reservoir treatments) are indicated. The timers are of conventional type such as those sold under the trade name "Eagle Cycl-Flex." R-1 to R-8 are relays for appropriate thermocouple selection. A reset timer or wash cycle controller is indicated at RT-1. R-9 and R-10 are isolation relays for reset pulse to RT-1. TDR is a time delay relay for reset pulse to RT-1. The various switches are identified as follows:

- S-1—Main power switch
- S-2—Timer reset switch
- S-3—Circulating pump switch
- S-4—Temperature control cut out switch
- S-5—Wash and drain switch
- S-6—Wash water supply switch
- S-7—All reservoir drains switch
- S-8—Cooling water control switch
- S-9—Manual reset of wash cycle timer RT-1
- S-10—Terminal stage selector switch

The numbered connections are made through a cable to the reactor system, a wiring diagram of which is illustrated in FIG. 7.

FIG. 7 shows the connections for the various valves and thermoregulators previously described. The following elements are also included in the diagram:

- R-11—Pump power relay
- R-12—Relay for deactivating temperature regulator during wash cycle

R-13—Cooling heating selector relay

R-14—Temperature control relay

S-11—Cooling mode selector switch

It will be observed that, in the modification illustrated, a separate timer RT-1 is employed for the washing sequence and is automatically reset after each stage. It deactivates the stage timers during the wash and, after completing its task, starts the next stage and turns itself off.

After all programmed stages have been completed, the apparatus shuts down and remains this way until reset manually. Spent liquor may be retained from each stage in the corresponding reservoir for analysis such as residual chemical which is frequently of interest. A strip chart record 19 of temperature throughout the experiment is also available as a check on time and temperature of all steps.

An important feature is the arrangement to avoid disrupting the experiment by momentary power failure. All valves close and the programming system remains in the condition that existed at failure. Thus, the experiment continues from where it left off when power returns. An extended power failure will, of course, alter the time/temperature cycle at the time of failure. This feature also permits the unit to be shut down if it is desired to make some change in the sequence.

In addition to the automatic control system, the manual switches S-1 to S-10 are provided for manual control. These switches are indicated at the top of the console in FIG. 8.

In order to illustrate the type of treatment which may be advantageously conducted with the equipment set forth, a typical experiment involving the multistage pulping of wood chips with chlorine dioxide and caustic treatments will now be described.

The wood chips to be reacted are placed in the vessel 15 which is then filled as desired with water and sealed. Chemicals to be applied in the first stage, such as 10% chlorine dioxide as an aqueous solution, are placed in reservoir R-1. The temperature desired in this stage one is adjusted on thermoregulator TH-1. The duration of the stage is controlled as desired by adjustment of timer T-1. Similarly, the chemicals (such as 4% sodium hydroxide in aqueous solution) for stage two are placed in reservoir R-2, thermoregulator TH-2 adjusted for desired temperature, and timer T-2 adjusted for desired time.

Additional stages, as required, could be similarly programmed.

When the programming is completed, the timers are activated to commence reaction. When stage one starts, valves V-1A and V-1B are opened automatically to introduce the chemicals from reservoir R-1. Steam or coolant is applied to the heat exchanger to control temperature as desired. Valves controlling entry of wash water and discharge of effluent are held closed during the time of reaction.

When stage one is completed, valves V-1A and V-2A close, thus retaining a sample of the reaction liquor for analysis. Wash liquid is introduced by opening valves V-9 and V-11 and effluent is released through valve V-10. The duration of the wash is regulated by arrangement of the timer RT-1.

The washing cycle can readily be varied to suit conditions. For example, one useful arrangement with materials that are difficult to wash clean is to apply wash water to displace the free liquor, and then to recirculate for some time without addition of fresh water to permit diffusion of the chemicals from the solid. Such treatment may be repeated a number of times as desired.

Stage two is then automatically initiated with opening of valves V-2A and V-2B.

Subsequent stages and intermediate washing proceeds similarly up to the arbitrary maximum number provided.

5

What is claimed is:

1. Laboratory apparatus comprising a vessel having a reaction chamber therein, a filter plate in said vessel dividing said chamber into a major portion and a minor portion, a main circulation line having an inlet in said major chamber portion and an outlet in said minor chamber portion, a pump in said line, a plurality of reservoirs for reception of various chemicals, said main circulation line having a plurality of branch lines each leading through one of said reservoirs, valve means for connecting each of said branch lines into said main line, temperature control means in said main line to adjust the temperature of materials flowing therethrough, means for actuating each of said valve means, and an electrical programming device for actuating a selected number of said valve means in a selected sequence.

2. Laboratory apparatus as defined in claim 1, said circulation line having valve means for admitting wash liquid thereinto and valve means for releasing effluent therefrom, said programming device including means for selective sequential actuation of said wash liquid admitting means and said effluent releasing means.

3. Laboratory apparatus as defined in claim 2, said programming device including timers for regulating the duration of time when each of said valve means is in open position.

4. Laboratory apparatus as defined in claim 1, said temperature control means comprising a heat exchanger in said main line, and a plurality of thermoregulators, said main circulation line having a branch line communicating with all of said thermoregulators, said programming device having means for activating a selected one of said

6

thermoregulators in response to incorporation of a selected one of said branch lines in said main line to adjust the temperature output of said heat exchanger.

5. A chemical reaction vessel comprising a tubular glass member, end walls of resilient and chemical resistant material closing said member, a first one of said walls having an inlet connection and a second one of said walls having an outlet connection, a perforated filter plate in said member and extending transversely thereof in spaced adjacent relation to said second end wall, and means for clamping said end walls in closed position on said member comprising a rigid plate engaging the outer surface of each said end wall and retaining rods releasably clamping said plates together.

6. A chemical reaction vessel as defined in claim 5, including a perforated distribution plate in said member and extending transversely thereof in spaced adjacent relation to said first end wall.

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U.S. Cl. X.R.

23—259; 73—63; 162—49, 198, 263