Jan. 13, 1959 W. W. ALLEN 2,868,626

SEMI-MICRO EVAPORATION APPARATUS

Filed June 18, 1956 3 Sheets-Sheet 1

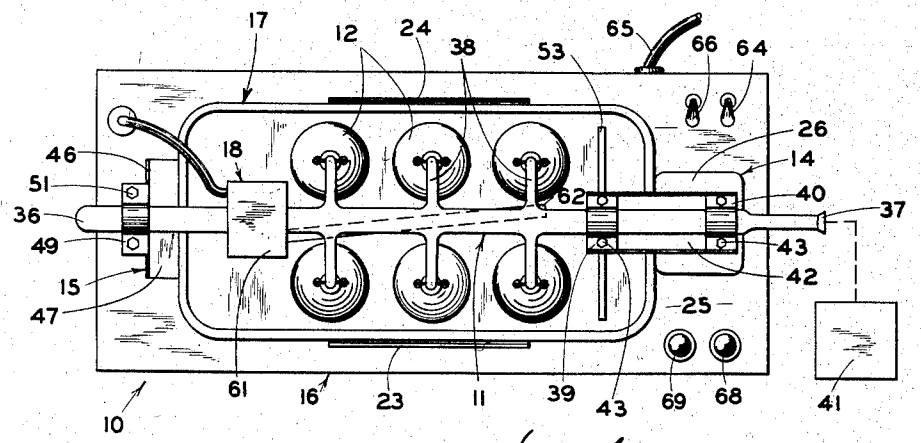


fig. 1

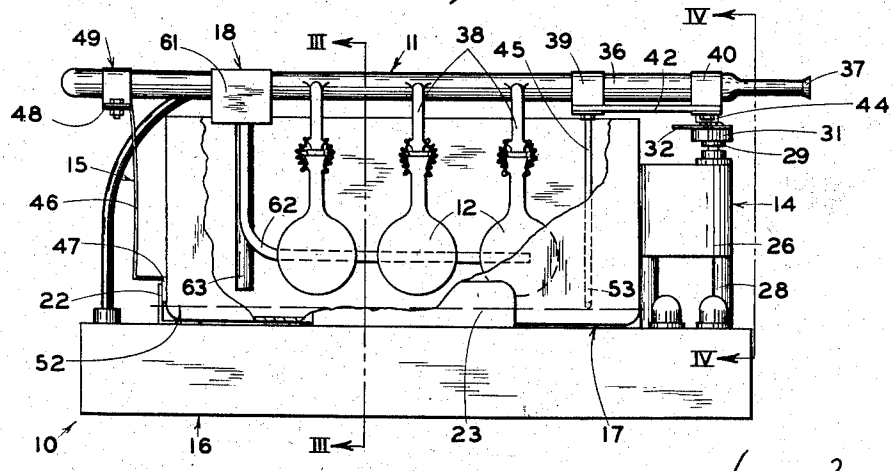


fig. 2

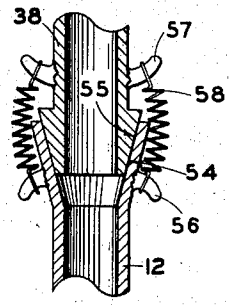


fig. 7

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SEMI-MICRO EVAPORATION APPARATUS

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3 Sheets-Sheet 2

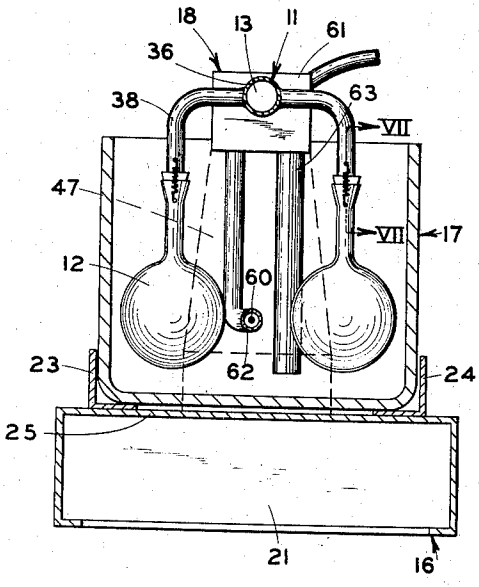


fig. 3

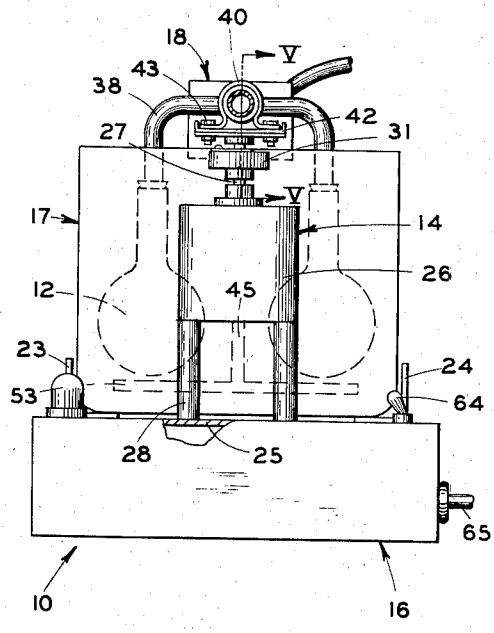


fig. 4

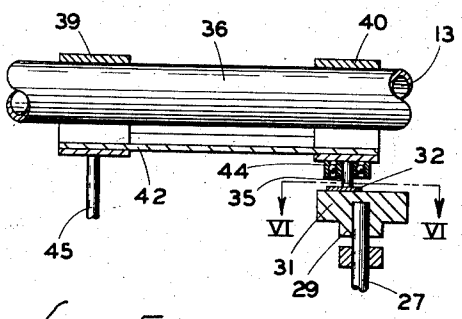


fig. 5

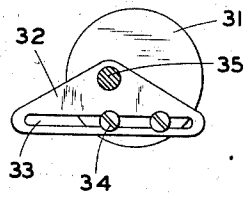


fig. 6

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3 Sheets-Sheet 3

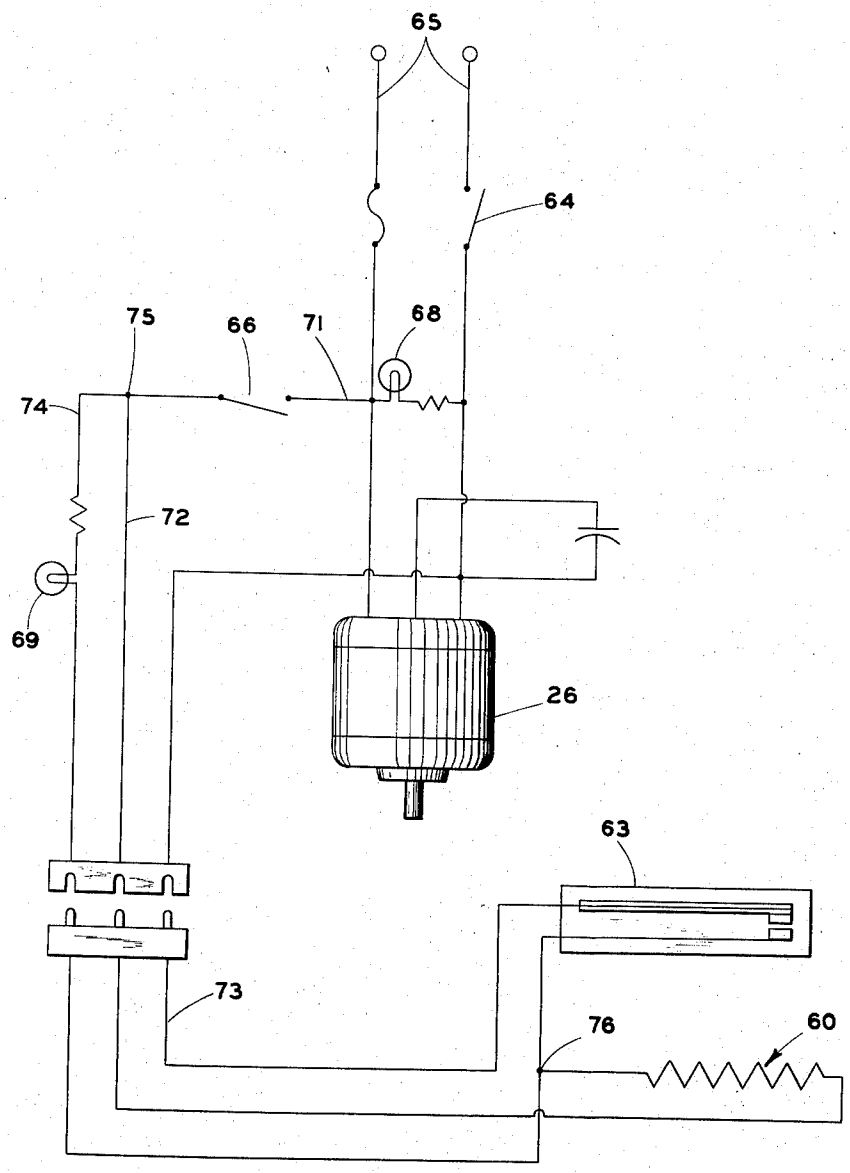


fig. 8

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2,868,626

SEMI-MICRO EVAPORATION APPARATUS

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Application June 18, 1956, Serial No. 592,127

7 Claims. (Cl. 23—259)

This invention relates in general to equipment for simultaneously and continuously agitating a plurality of containers, controlling their temperatures, and removing a gas or vapor from within each container. More particularly, said invention relates to equipment operable with a plurality of containers, each containing a solution, for separating the solvent from the solute in each container and for removing such solvent from said containers.

In many types of laboratory work, it becomes necessary to evaporate solvents for the purpose of recovering materials dissolved therein. In various instances of analytical work, this becomes a particular problem where it is desired to handle a number of samples simultaneously, to draw the evaporated solvent into exhausting means and to retain the residual solute as a deposit in the container. Further, in order to carry out effective and rapid evaporation, it is often necessary to heat the solutions within the containers, but to do so at a controlled rate in order to avoid deleterious effects on the solutes. Various of these functions have been performed by the equipment available in the prior art, but, insofar as I am aware, none of the previously available equipment has been able to perform all of these functions at one time or to do so efficiently and effectively.

It is also evident that, while the foregoing discussion has dealt entirely with the withdrawal of a solvent from a plurality of solutions or solution samples, there are many other instances in laboratory work where it is desired to agitate simultaneously a plurality of containers, control the temperatures of said containers, and withdraw a gas or vapor from within each of the containers, said gas or vapor being withdrawn under such conditions that its escape into the laboratory, or other space occupied by personnel, is effectively prevented.

Therefore, a principal object of the invention has been to provide apparatus which will simultaneously agitate solutions or solution samples contained within a plurality of containers and draw a gas or vapor from within each of said containers through a closed system to a point of reception or discharge.

A further object of the invention has been to provide a device, as aforesaid, which will effectively control the temperature of the several solutions or solution samples.

A further object of the invention has been to provide a device, as aforesaid, which can be readily assembled and disassembled for loading, use and unloading.

A further object of the invention has been to provide a device, as aforesaid, which will have a minimum of moving parts.

A further object of the invention has been to provide a device, as aforesaid, wherein the agitation function is carried out from only a single connection, and wherein the withdrawal of gas or vapor is carried out from only a single connection.

A further object of the invention has been to provide a device, as aforesaid, which is of sufficiently simple construction that it will be economical to fabricate and will have low maintenance costs.

A further object of the invention has been to provide a device, as aforesaid, which can be readily modified for use with containers of various sizes and shapes.

A further object of the invention has been to provide a device, as aforesaid, particularly adapted for the evaporation of sample solutions, in which the several samples can be heated to a carefully controlled temperature level during the evaporation process in order to avoid breakdown or other deleterious effects with respect to the solutes being obtained.

A further object of the invention has been to provide a device, as aforesaid, in which all of the operating parts are readily accessible for adjustment and/or repair, if and when such becomes necessary.

Other objects and purposes of this invention will become apparent to persons familiar with this type of equipment upon reading the following specification and examining the accompanying drawings, in which:

Figure 1 is a top plan view of an apparatus characterizing the invention.

Figure 2 is a broken, side elevation view of said apparatus.

Figure 3 is a sectional view taken along the lines III—III of Figure 2.

Figure 4 is a sectional view taken along the line IV—IV of Figure 2.

Figure 5 is a sectional view taken along the line V—V of Figure 4.

Figure 6 is a sectional view taken along the line VI—VI of Figure 5.

Figure 7 is a sectional view taken along the line VII—VII of Figure 3.

Figure 8 is a diagram of a circuit which may be used with said apparatus.

For purposes of convenience in description, the terms "upper," "lower," "right," "left," "front" and "rear," as used herein, will have reference to the apparatus and parts associated therewith as appearing in Figure 2. The terms, "inner," "outer," and derivatives thereof, will have reference to the geometric center of said apparatus and parts associated therewith. This terminology is for reference purposes only, and is not limiting.

General description

In order to meet the objects and purposes set forth above, as well as others related thereto, I have provided an apparatus **10** (Figures 1 and 2) comprised of a housing **11**, here elongated, upon which a plurality of containers **12** are removably supported, and having a plenum chamber or manifold **13** (Figure 3) with which said containers communicate. On end of said housing **11** is removably supported upon an actuating mechanism **14**, which is arranged to impart a predetermined amount and pattern of movement to said housing. The other end of said housing **11** is mounted upon a support mechanism **15**, which merely supports said cooperable other end and permits it to follow the motion imparted by the actuating mechanism **14**. The actuating mechanism **14** and support mechanism **15** are both mounted upon a common base member **16**. Said containers **12** are subjected to a source of heat exchange, as by insertion into a liquid bath contained within a tank **17**, which tank is also supported upon the base member **16**, between said actuating mechanism **14** and support mechanism **15**. A controllable heat exchange device **18**, which may be conveniently mounted upon the housing **11**, is provided for maintaining the liquid within the tank **17** at a desired temperature.

Detailed construction

As shown in Figures 1, 2 and 3, the base member 16 is preferably fabricated from a durable sheet material, such as steel, and is shaped to define a compartment 21, in which is housed the major part of the wiring by which the device is operated, as hereinafter described in detail. A left end bracket 22, a front bracket 23, and a rear bracket 24, are mounted upon the upper surface of the base member 16, which is provided by the upper wall 25 of the compartment 21, for engaging the corresponding walls of the tank 17.

The actuating mechanism 14 (Figures 2 and 4) includes a motor 26 having a vertical, upwardly extending shaft 27. Said motor is supported upon the upper ends of the support posts 28, the lower ends of which are secured to said upper wall 25 near the rightward end of the base member 16. The housing or frame of the motor 26 is preferably positioned to engage the rightward end of the tank 17 to substantially prevent endwise movement of the tank 17 with respect to the base member 16. Thus, by the brackets 22, 23 and 24, together with said motor 26, the tank 17 is held against all lateral movement with respect to the base member 16, but it can be readily removed therefrom merely by lifting upwardly.

The hub 29, which is removably secured to the upper end of the motor shaft 27, is provided with a radially extending, annular flange 31 at its upper end (Figures 5 and 6). An adjustment plate 32 (Figure 6), having an elongated slot 33, is adjustably supported upon the upper surface of the flange 31 by means of a pair of machine screws 34, which slidably extend through said slot 33 for threaded engagement with said flange 31. The center line distance between said screws 34 is materially less than the lengthwise extent of the slot 33, thereby permitting substantial adjustment of the position of the plate 32 with respect to the flange 31 in a direction parallel with the lengthwise extent of the slot 33 and transverse of the axis of the motor shaft 27. A stub shaft 35 is secured to, and extends upwardly from, the adjustment plate 32, preferably on one side of the slot 33 and parallel with said axis of said motor shaft 27. Said stub shaft 35 is preferably positioned on said plate 32 so that it can be moved with the plate 32, upon proper adjustment of the screws 34, between conditions of concentricity and substantial eccentricity with respect to the motor shaft 27, thereby constituting an adjustable crank.

The housing 11 (Figures 1 and 2) is, in this particular embodiment, a relatively straight, glass tube 36, having an opening at one end, here the rightward end 37, thereof, and the other end being closed. Said tube 36 is provided with a plurality of integral, laterally and downwardly extending pipes 38, which are preferably clustered near the center portion of the tube 36 and arranged for removable engagement with the containers 12 in a manner discussed in detail hereinafter. The open end 37 of the tube 36 may be connected, in a conventional manner, to a conventional evacuating device, such as the vacuum pump 41 indicated schematically in Figure 1.

The tube 36 (Figures 1, 2 and 5) is embraced between its open end 37 and said pipes 38 by a pair of spaced clamps 39 and 40, which clamps are mounted upon an elongated, relatively flat connector element 42 by means of the machine bolts 43. A bearing 44 (Figure 5) is mounted upon the lower surface of the connector element 42 near the right the right end thereof, as by means of the machine bolts 43 which secure the clamp 40 to said element. The bearing 44 is axially perpendicular to the connector element 42 and arranged for snug, but removable, reception of the stub shaft 35.

The upper end of a T-shaped support post 45 is secured, with its crossbar 53 downwardly, by any convenient devices, such as the machine bolts 43 which engage the clamp 39, to the lower surface of the connector element 42 near the left end thereof. Thus, when said tank 17 and housing 11 are in position upon the base member 16, the post 45 will be disposed within, and spaced from the walls of, the right end of the tank 17. Said support post 45 is utilized in cooperation with the support mechanism 15 to support the housing 11 when removed from the tank 17 and the base member 16 in a manner discussed in detail hereinafter.

The support mechanism 15 (Figures 1, 2 and 3) is comprised of a relatively flat, substantially vertical plate 46, which is fabricated from a flexible material, such as sheet metal. Said plate 46 is preferably disposed, as in this particular embodiment, perpendicular to the lengthwise extent of the housing 11 and the upper wall 25 of the compartment 21, for reasons appearing hereinafter. The lower end 47 of the plate 46 is offset toward the actuating mechanism 14 and vertically slidably disposable between the left end bracket 22 and the adjacent end of the tank 17 when said tank is in operating position upon the base member 16. The upper end of the plate 46 is provided with an integral, leftwardly extending flange 48, upon which the leftward end of the tube 36 is secured, by means of the clamp 49 and machine bolts 51.

Thus, the housing 11 will be normally held firmly in place with respect to the base member 16 or it can, at the will of the operator, be removed from its position of support upon the base member 16 merely by lifting said housing upwardly away from said member. Such lifting will disengage the lower end 47 of the plate 46 from between the bracket 22 and the tank 17, and will withdraw the bearing 44 from the stub shaft 35. The housing 11 can then be supported upon any horizontal surface, such as a table, said surface being engaged by the lower end 47 of the plate 46 and the crossbar 53 of the support post 45, as indicated in broken lines at 52 in Figure 2.

The outer ends of the pipes 38 (Figure 3) are provided with tapered, ground surfaces 54 (Figure 7), engageable with cooperating ground surfaces 55 on the containers 12. Said containers 12 and pipes 38 are both provided near their respectively engaging ends with a pair of diametrically disposed, integral hooks 56 and 57. Resilient means, such as springs 58, are used to connect the corresponding hooks 56 and 57 for holding the containers 12 in engagement with the pipes 38.

The heating device 18 includes a head 61, which is removably supported upon the tube 36 between the clamp 49 and the pipes 38, and preferably disposed above the tank 17 when said tank is in operating position (Figures 1 and 2) upon the base member 16. A heating element, indicated by the resistance 60 in Figure 8, of an appropriate, known type, is hermetically sealed into the lower end of a bent metal tube 62, referred to as the heater tube. The lower end of said tube is located adjacent to the lower ends of the containers 12, which containers are arranged in two substantially parallel rows, in this particular embodiment, on opposite sides of, and below, the tube 36. A temperature control 63, normally a thermostat, extends downwardly from the heating head 61 for controlling automatically the operation of the heating element 60.

As shown in Figures 1 and 8, the motor 26 is controllably connected to a source of electrical potential through the power leads 65 and a master switch 64. A "switch-on" light 68 is provided, as shown, to indicate the electrical energizing of the device. A conductor 71 from one side of the power source is connected through a heater switch 66 to a conductor 72, which is connected through a conventional plug connection, if desired, to the heater element 60 and thence through the thermostat 63 and a conductor 73 back to the opposite side of the power source. Thus, upon closing of both the master switch 64 and the heater switch 66, operation

of the heating element **60** is effected under the control of the thermostat **63**. A further pilot light **69** is connected by the conductor **74** from a junction point **75** to a junction point **76** on the heater side of the thermostat **63**. Thus, the pilot light **69** will indicate whenever the thermostat is closed and act as a warning to the operator if the thermostat stays in closed position for longer than a reasonable length of time.

Operation

Before commencing the operation of the apparatus **10**, it will normally be necessary to remove the housing **11**, with its associated containers **12**, from its position upon the base member **16**, in order to place in said containers **12** the solutions or solution samples to be evaporated. Because of the vertically sliding engagement of the support mechanism **15** with the end bracket **22**, and of the bearing **44** with the stub shaft **35**, said removal of the housing **11** can be effected easily and quickly simply by lifting the housing **11** upwardly away from the base member **16**. If the containers **12** are already attached to the housing **11**, they are easily and quickly removed by releasing one end of the springs **58** from the hooks **56** or **57** and drawing the containers **12** away from their associated pipes **38**. The containers **12**, having been removed from said pipes **38**, are then filled with the solutions or solution samples to be evaporated, after which they are returned to their position of engagement with the pipes **38** and secured with respect thereto by the springs **58**. During this filling of the containers **12**, the housing **11** will preferably be supported by the support mechanism **15** and the support post **45** on a convenient surface, as a table, as stated hereinabove.

The tank **17** is filled to a convenient level with a suitable liquid, such as water or oil, which can be heated by the element **60** in the tube **62** for the purpose of heating the contents of the containers **12**. The adjustment plate **32** is adjusted, as desired or required, to place the stub shaft **35** thereon in the desired position of eccentricity with respect to the motor shaft **27**. The complete housing assembly, including the housing **11**, the containers **12** attached thereto, the support mechanism **15**, the connector element **42**, and support post **45**, is then returned to its position, as shown in Figures 1 and 2, so that the containers **12**, lower end of the heating tube **62**, the temperature control **63**, and, in this particular embodiment, the support post **45**, are all disposed within the tank **17**. A substantial portion of the containers **12**, the heater tube **62** and the temperature control **63** will be immersed in the liquid within the tank **17**. The plate **46** will be re-engaged with the end bracket **22** and the bearing **44** will again receive the stub shaft **35**. The vacuum pump **41**, or a similar device, is then connected in a conventional manner to the open end **37** of the tube **36**.

The motor switch **64** and heater switch **66** may then be closed, thereby energizing the circuit shown in Figure 8. This results in imparting a rotary motion to the rightward end of the housing **11**, due to the eccentricity of the stub shaft **35**. The flexible plate **46** will bend leftwardly with each rotation of the motor **26**, in a substantially simple, harmonic motion. Accordingly, all of the elements mounted upon the tube **36** between the support mechanism **15** and the open end **37** of said tube will be rotated in circles of increasing diameter from left to right. Thus, the containers **12** will be given a rotary motion within the tank **17**, which will both agitate the heat controlling liquid within said tank and will cause the solution within said containers to move around the side walls thereof in a well-known manner. As soon as the heater switch **66** is closed, the switch-on light **68** will be illuminated. If the temperature within the tank **17** is below the desired level, the temperature control **63** will be closed, thereby energizing the heating element **60** and illuminating the heater light **69**. When the liquid in the tank **17** has reached the proper temperature, the temperature control **63** will open, thereby deenergizing the heating element **60** and the heater light **69**.

The contents of the containers **12** can be examined periodically, if not visible through the liquid in the tank **17**, by opening the motor switch **64** and then raising the housing **11** away from the base member **16**.

It will be understood that while a given housing **11**, if made of glass, as is usual, will be provided with pipes **38** of fixed sizes and, therefore, capable of use only with containers having mouths of correspondingly fixed sizes, the remainder of the apparatus can be readily used with a variety of different housings corresponding to the housing **11** and, hence, merely by providing a suitable supply of such housings, the apparatus can be used with a variety of different sizes and shapes of containers. Alternatively, said apparatus may be provided with adapter means, not shown, fitting onto the surface **54** of the pipes **38** for receiving containers of other selected sizes and/or shapes.

Although the liquid within the tank has been assumed to be for heating purposes, and the tube **62** has been utilized to house a "heating element," it will be obvious, in view of the foregoing, that if desired, as where an exothermic reaction is progressing in the containers simultaneously with the evaporating of the solvent, the heating element **60** may be replaced by a suitable cooling element and the liquid will then function to cool the containers. Further, although the apparatus above described was developed primarily for evaporation purposes, and such has been the use disclosed herein for illustrative purposes, it will be evident from the foregoing that many uses are possible involving simultaneous agitation of material in a plurality of containers, temperature control thereof, and withdrawal of a gas or vapor from within each container.

Thus, although a particular, preferred embodiment of my invention has been disclosed hereinabove for illustrative purposes, it will be understood that variations and modifications thereof, which come within the scope of such disclosure, are fully contemplated unless specifically stated to the contrary in the hereinafter appended claims.

I claim:

1. Apparatus for simultaneously moving, and withdrawing a gas or vapor from, a plurality of sealed, interconnected containers, comprising: a base member; a housing defining an evacuable chamber; means removably connecting said containers to said housing with said chamber communicating with the interior of said containers, said containers being supported on said housing for movement therewith; a source of vacuum and means connecting said source to said chamber; a tank supported on said base member; a heat exchange medium within said tank and means for controlling the temperature thereof; means supporting said housing above said tank with said containers being positioned within said tank, said support means including means mounted on said base member for permitting movement of said housing and said containers with respect to said tank through a closed path about an axis substantially perpendicular to said housing; actuating means mounted on said base member and secured to said housing adjacent one end thereof for effecting such movement of said housing and said containers with respect to said tank.

2. Apparatus for simultaneously moving, and withdrawing a gas or vapor from a plurality of sealed, interconnected containers, comprising: a base member; a motor secured to said base member; actuating mechanism secured to the motor shaft; a tank removably positioned on said base member; a heat exchange liquid within said tank and means for controlling the temperature thereof; an elongated housing disposed above said base member, said housing having an evacuable chamber therewithin; means removably connecting said containers to said housing with said chamber communicating with the interior

of said containers, said containers being immersed in said liquid and being supported on said housing for movement therewith; a vacuum source and removable means connecting said source to said chamber; support means secured to said housing adjacent the respective ends thereof, one of said support means being removably connected to said base member and permitting said housing and said containers to move with respect to said tank through a closed path about an axis substantially perpendicular to said housing, the other of said support means being removably connected to said actuating means whereby upon operation of said motor said actuating means causes said other support means to move said housing and containers with respect to said tank through said closed path.

3. An apparatus according to claim 2 wherein said means for controlling the temperature of said liquid includes a heating head secured to said housing, a heating element extending from said heating head into said liquid, and temperature responsive means including a switch extending from said heating head into said liquid.

4. An apparatus for supporting containers which includes: an elongated tubular housing defining a chamber and having a plurality of pipes extending therefrom, each of said pipes having a frusto-conical outer end and each of said pipes further having a plurality of hooks thereon adjacent said outer end; said containers having frusto-conical openings for receiving the ends of said pipes; a plurality of hooks on each of said containers adjacent said openings; and resilient means connecting the hooks on said pipes to corresponding hooks on said containers for resiliently urging the ends of said pipes into the openings of said containers.

5. Apparatus for simultaneously moving, and withdrawing a gas or vapor from, a plurality of sealed, interconnected containers, comprising: a base member; an elongated tubular housing defining an evacuable chamber and having a plurality of pipes extending therefrom, each of said pipes having a frusto-conical outer end and having a plurality of hooks thereon adjacent said outer ends; said containers having frusto-conical openings for receiving the ends of said pipes; a plurality of hooks on each of said containers adjacent said openings; resilient means connecting the hooks on said pipes to corresponding hooks on said containers for resiliently urging the ends of said pipes into the openings in said containers; a source of vacuum and means connecting said source to said chamber; a tank supported on said base member; a heat exchange liquid within said tank and means for controlling the temperature thereof; means supporting said housing above said tank with said containers being positioned within said tank, said support means including means mounted on said base member for permitting movement of said housing and said containers with respect to said tank through a closed path about an axis substantially perpendicular to said housing; actuating means mounted on said base member and secured to said housing adjacent one end thereof for effecting movement of said housing and said containers.

6. Apparatus for simultaneously moving, and withdrawing a gas or vapor from, a plurality of sealed, interconnected containers, comprising: a base member; a housing defining an evacuable chamber; means removably connecting said containers to said housing with said chamber communicating with the interior of said containers, said containers being supported on said housing for movement therewith; a source of vacuum and means connecting said source to said chamber; a tank supported on said base member; a heat exchange liquid within said tank and means for controlling the temperature thereof; means supporting said housing above said tank with said containers being positioned within said tank, said support means including means mounted on said base member for permitting movement of said housing and said containers with respect to said tank through a closed path about an axis substantially perpendicular to said housing; a motor mounted on said base member with its shaft extending vertically; a vertically extending pin; a plate connected to said pin, said plate having an elongated slot therein and fastening means secured to said shaft and extending through said slot for securing said plate and pin to said shaft; a collar on said housing adjacent one end thereof, said collar having an opening for receiving said pin whereby said housing may be moved through said path.

7. An apparatus according to claim 6 wherein said support means includes an upwardly extending flexible plate secured to said housing adjacent the other end thereof, said plate being removably connected to said base member.

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