United States Patent [19]

Sutherland et al.

[11] **3,795,986**

[45] **Mar. 12, 1974**

[54] **MODULAR COMPARTMENT SUBLIMATOR**

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[73] Assignee: **Cenco Medical/Health Supply Corporation,** Chicago, Ill.

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[21] Appl. No.: **207,507**

[52] **U.S. Cl.** **34/92,** 34/5

[51] **Int. Cl.** **F26b 13/36,** F26b 5/06

[58] **Field of Search** .... 34/5, 92; 292/338, 267, 292/278, DIG. 4

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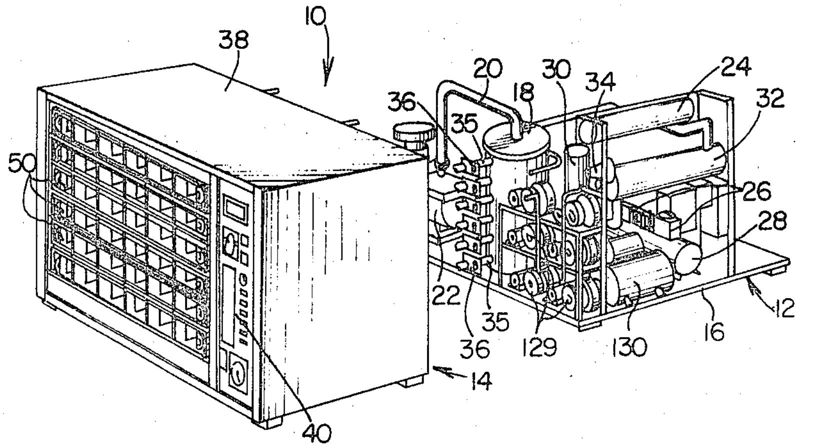
Primary Examiner—William F. O'Dea
Assistant Examiner—William C. Anderson
Attorney, Agent, or Firm—Gomer W. Walters

[57] **ABSTRACT**

An apparatus particularly adapted for freeze drying various materials and for stoppering, under vacuum, receptacles containing these materials after freeze drying having a plurality of individual drying chamber modules, each chamber forming a self-supporting drying module capable of receiving a number of receptacles upon one wall of the chamber also which serves as a shelf for supporting them. Each chamber is independently connected to a vacuum source, a condenser and heat transfer means mounted on an adjacent but separate equipment unit in such a manner that thermal transfer and the duration of the freeze drying cycle may be varied independently with respect to each individual module to permit freeze drying of diverse materials and allow different drying rates. A latching means and a door support means mounted adjacent the chamber door permit doors of adjacent modules to be opened without obstruction and act to maintain each door in an open position. Stoppering means having upper and lower plates sandwiching an expansible means may be removably mounted within the separate drying modules and independently activated by operation of fluid pressure means mounted on the equipment unit to pressurize the expansible means and urge the plates apart to stopper the receptacles. Stoppering control means require the force exerted by the stoppering means on the receptacles to remain constant despite variation in height of the receptacle and permit discontinuation of the stoppering operation at any time.

25 Claims, 19 Drawing Figures



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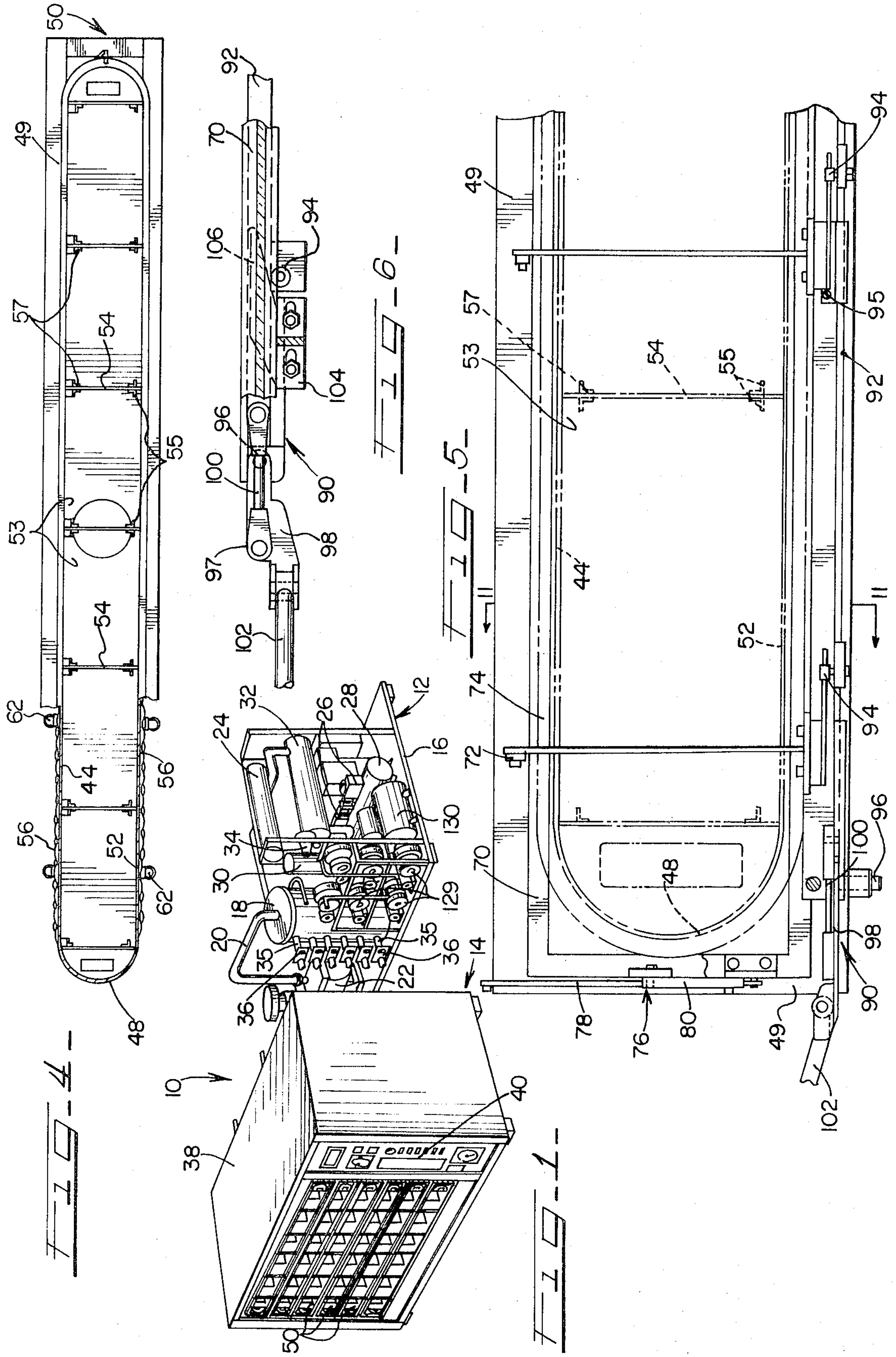

FIG. 1

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FIG. 5

FIG. 6

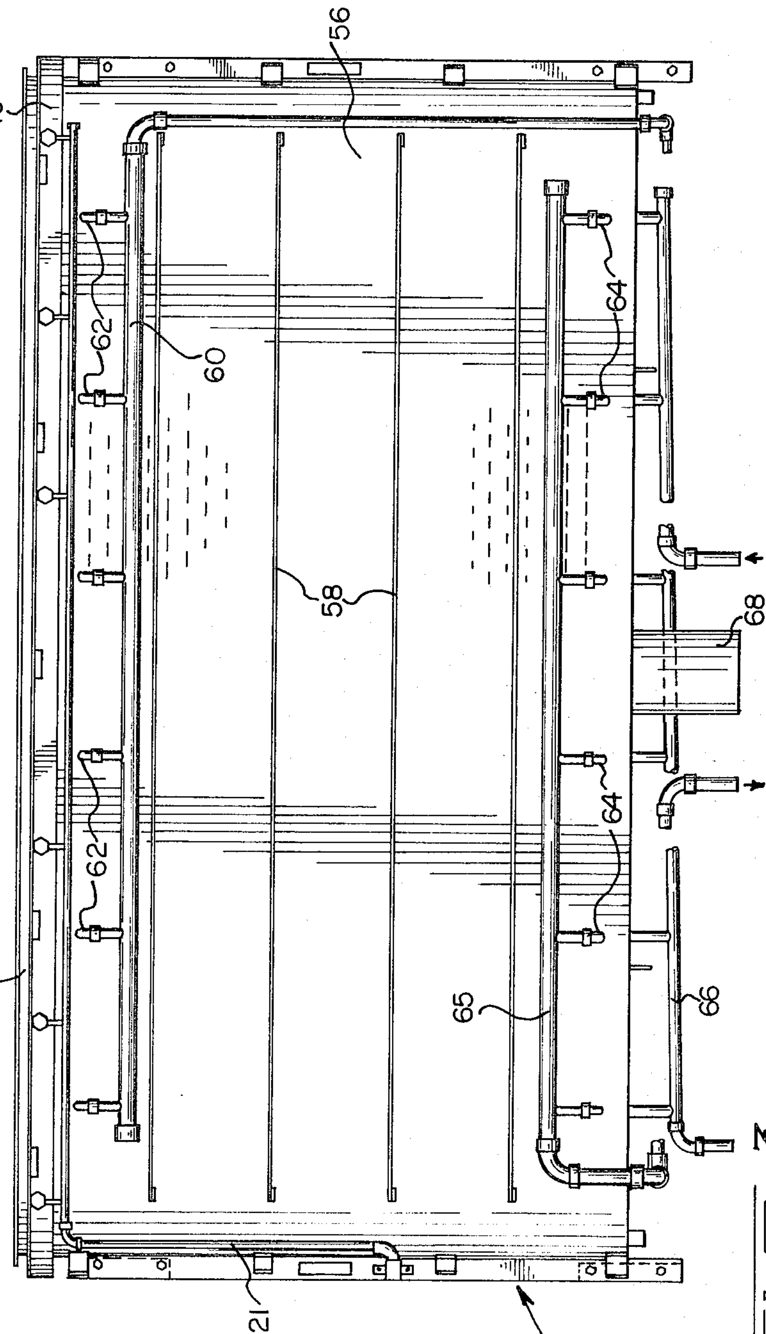

FIG-2

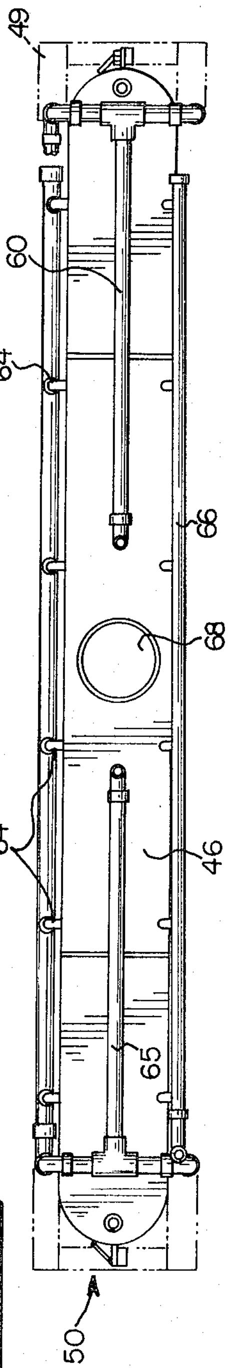

FIG-3

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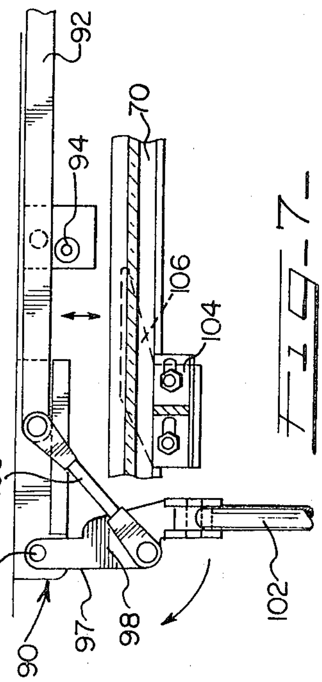

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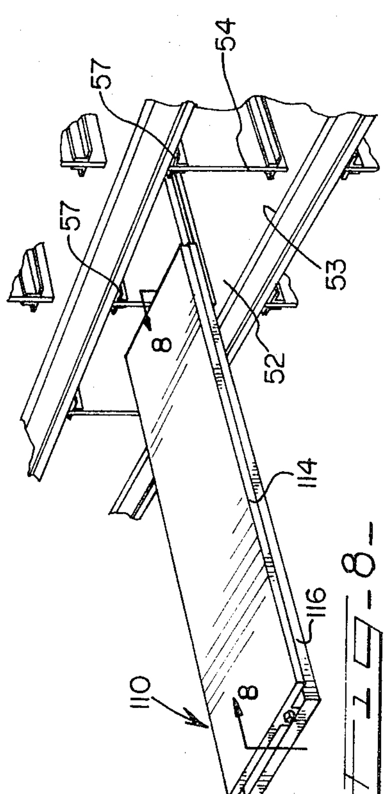

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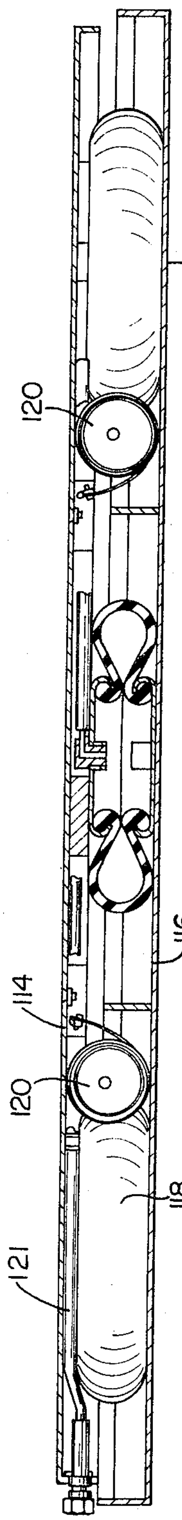

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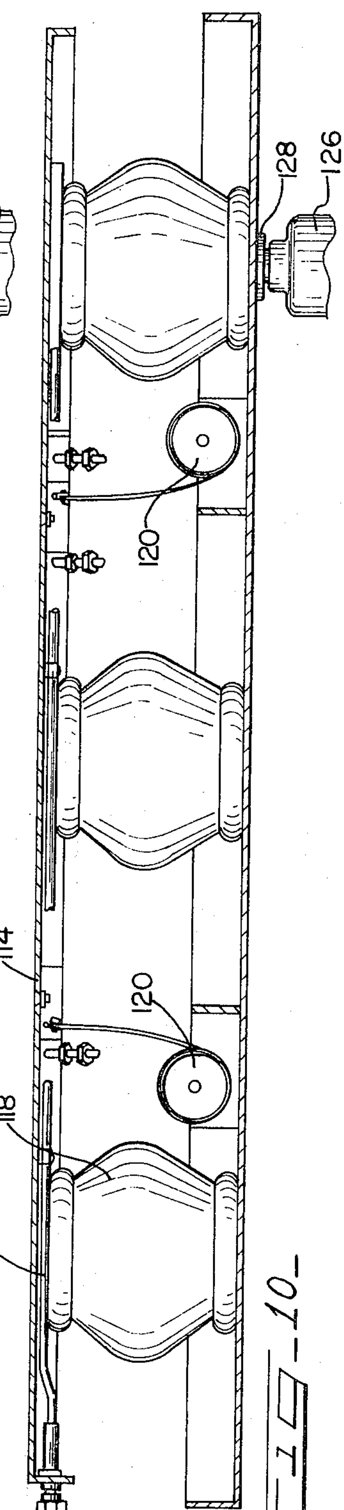
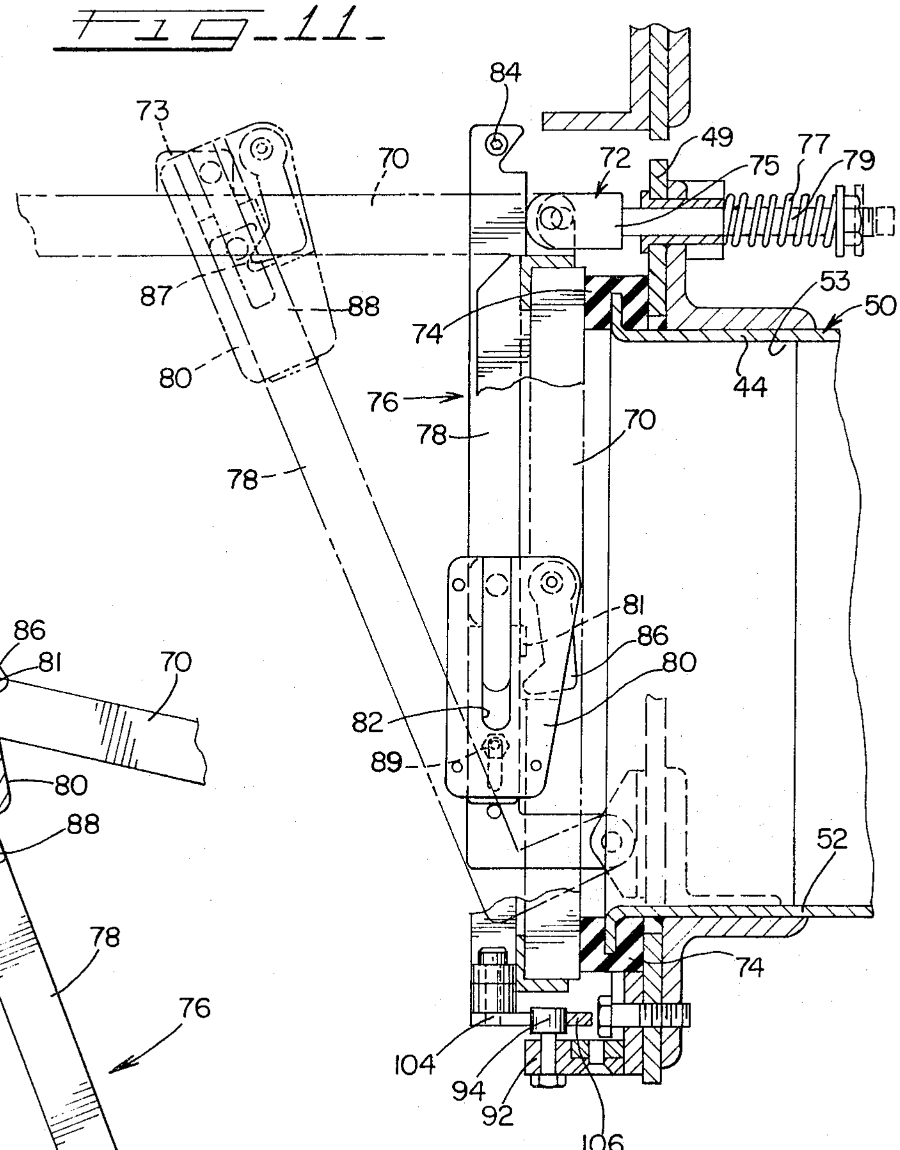


FIG-10

FIG. 11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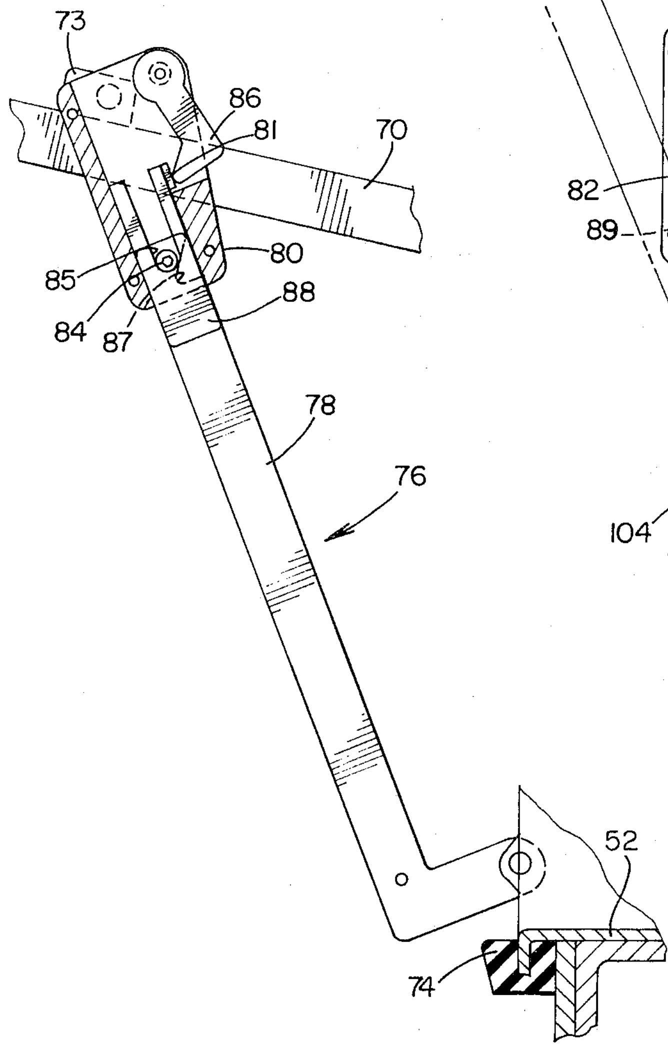
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FIG. 12

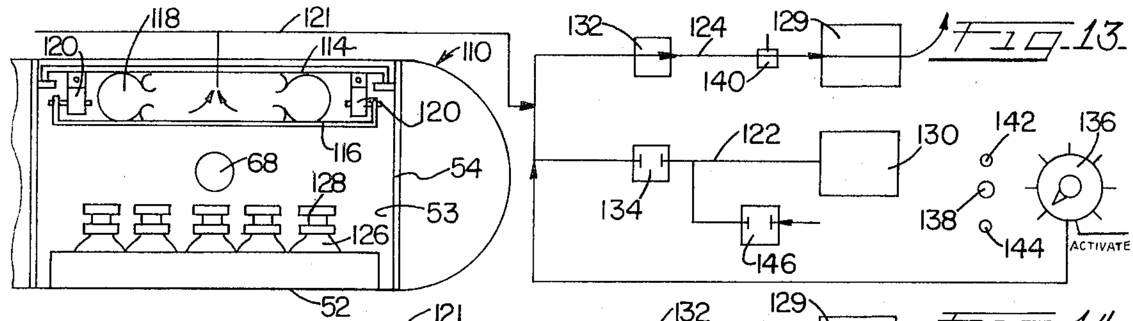

Fig. 13.

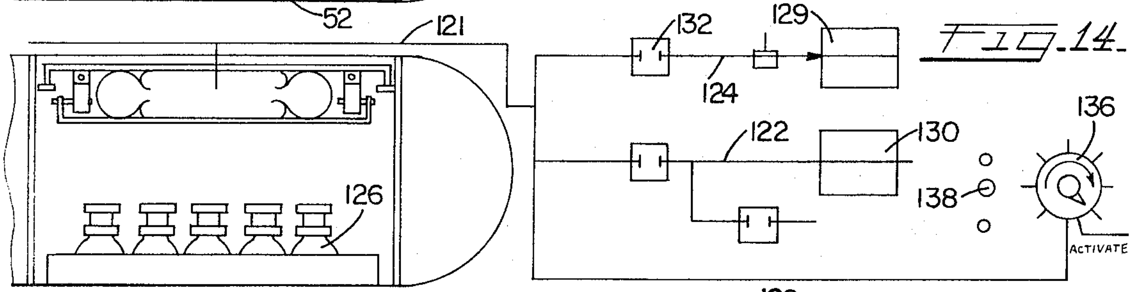

Fig. 14.

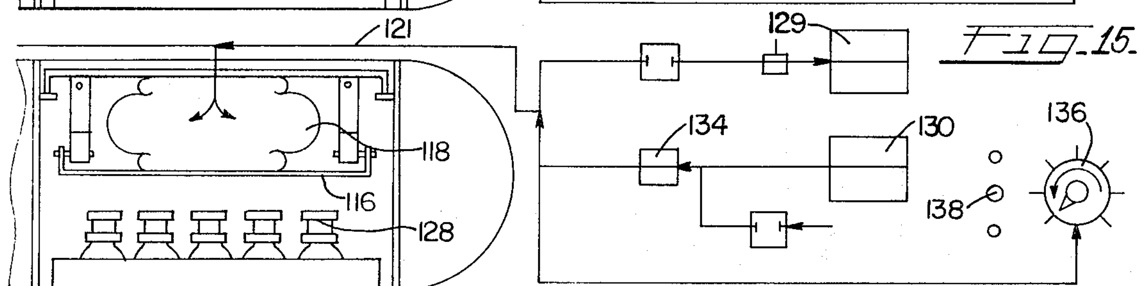

Fig. 15.

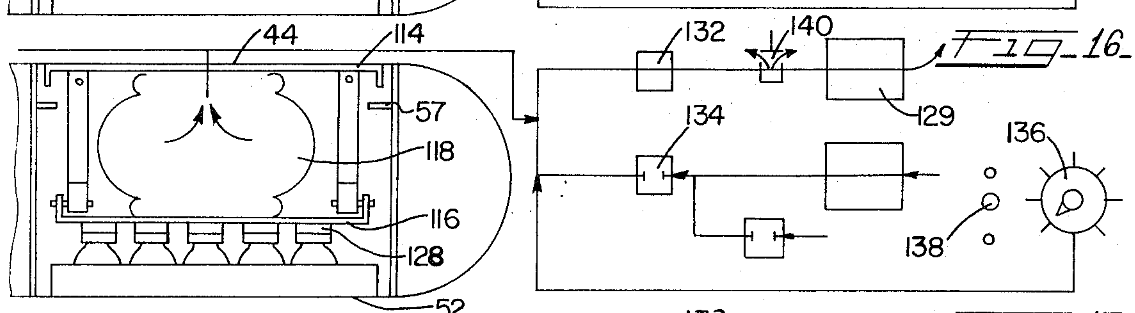

Fig. 16.

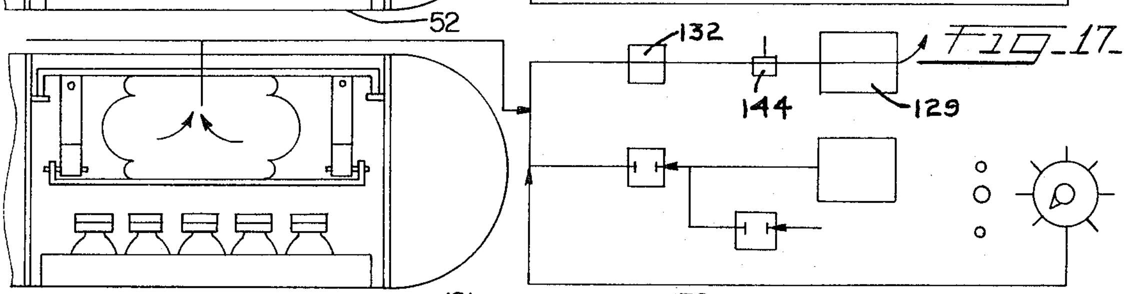

Fig. 17.

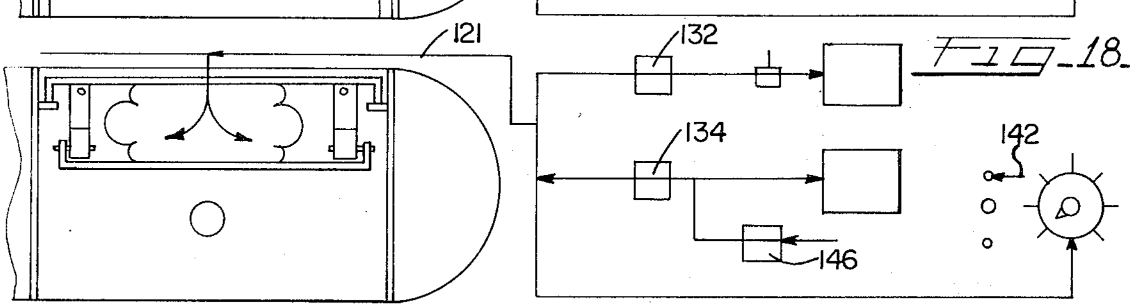

Fig. 18.

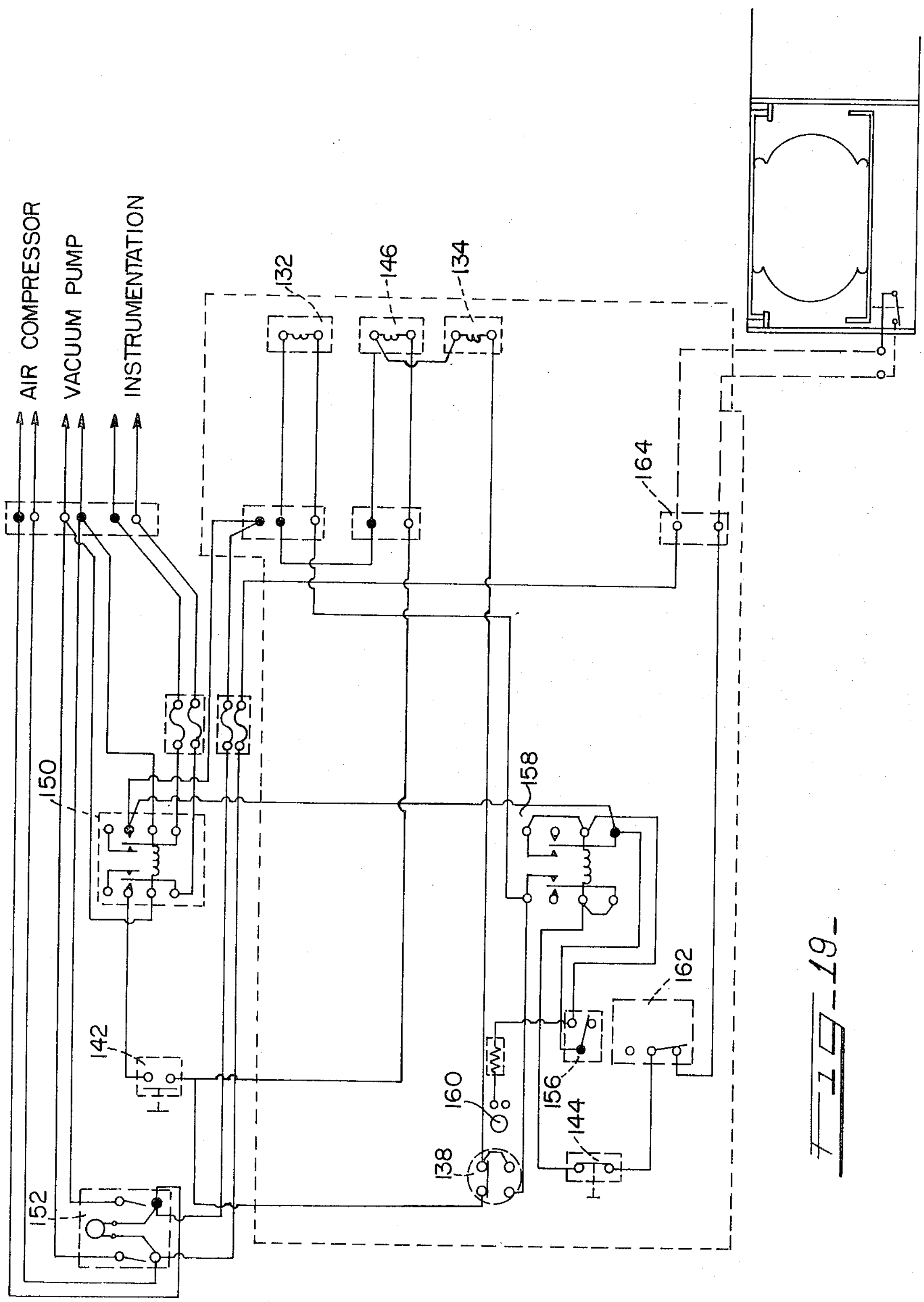
AIR COMPRESSOR
VACUUM PUMP
INSTRUMENTATION
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FIG. 19

MODULAR COMPARTMENT SUBLIMATOR

BACKGROUND OF THE INVENTION

This invention relates to a sublimator for freeze drying materials, and, in particular, to an improved sublimator which permits freeze drying of various materials independently through the use of separate drying chamber modules and provides for stoppering of receptacles containing these materials, under vacuum, by a removable stoppering means.

The method of drying biologicals and other materials by sublimation of the ice vacuo to produce high-quality products for human and animal, consumer, medicinal and like uses has been known for over 50 years, since Shackell applied vacuum pumps to his experiments to accelerate the process. It was not until shortly before World War II, however, that the true potential of laboratory freeze dryers and the process of sublimation were made known, since, during that War, much attention was given to the development of equipment and techniques for the purposes of supplying dried blood plasma and penicillin to the Armed Forces. Since that time, the freeze drying process has come to be accepted as one of the most perfect methods of preserving biological material. This has led to increased interest by food processers as well as by pharmaceutical manufacturers in the possible uses of freeze drying. In the past, with emphasis on this technique as a research tool, the greatest concern has been with perfect preservation of the material being dried. Now, however, with the increasing major industrial uses of the process, equipment cost, flexibility, and efficiency and speed, in addition to quality results, have become important considerations.

The technique of freeze drying, which has become wel known, is similar to ordinary vacuum distillation except that the material to be dried is first frozen and then subjected to a high vacuum and controlled heat input. Under these conditions, the water content of the material is removed selectively by sublimation, that is, ice goes directly to water vapor, bypassing the liquid phase. Since the solids are locked in an ice matrix during the entire dehydration, they cannot interreact. The high vacuum also prevents oxidation from occurring.

There are three components required for any freeze drying application: a vacuum-type chamber to contain the product; a condenser to trap sublimating water vapor; and a two-stage, oil-sealed, high-vacuum pump. In addition, means of transferring heat to the product and providing refrigeration within the condenser is essential. When bulk drying is not being carried on, some means of stoppering the receptacles containing the small samples of dried material under vacuum is also desirable.

The above components have usually been assembled and sold as units called freeze dryers or sublimators. One common and popular type of freeze dryer is the chamber type, as manufactured by The Virtis Company of Gardiner, N.Y., and includes a vacuum-tight, cylindrical or rectangular chamber, containing a number of horizontal shelves disposed in a vertical stacked arrangement. The condenser is usually positioned adjacent the sides of the shelves, but may be located near the support equipment. The vacuum pump and heat transfer means, including a central heater and one or more refrigerant compressors for the condenser, are usually located in close relationship to, but outside the chamber itself. A control means permitting control of the variables in the sublimation process completes the package, which is then surrounded by sheet metal and sold as a unit.

Attempts to use present freeze drying equipment in new fields such as pharmaceuticals, however, have served to highlight several disadvantages in such equipment. Among the major disadvantages of present chamber-type freeze dryers are their inflexibility and lack of economy. The use of a single chamber having several shelves does not permit sublimation of diverse materials or products requiring different length drying times. The single large chamber must be operated to full capacity in order to operate economically even though in certain uses, such as medical and pharmaceutical research, small batch runs may be desirable. Also, the use of a series of stacked shelves within the drying chamber is not an efficient use of chamber space since it wastes all drying space outside the rectangular volumetric area. Finally, the method of heat transfer in the chamber-type freeze dryer is not as efficient as is desirable since the transfer fluid is circulated within coils attached to the underside of the shelving, thus conducting heat unevenly only to certain points on the surface of the shelving. In addition, in bulk drying, the top of the tray receives little heat by radiation from above. The heat transfer fluid itself is cycled on and off, causing undesirable hot spots or temperature lags.

An improved concept in the construction of freeze dryers or sublimators has been advanced in an article by Taylor N. Thompson, Vice President of The Virtis Company, Gardiner, N. Y., which was presented at a meeting of the Parental Drug Association in New York City on Oct. 30, 1963. Mr. Thompson suggested use of modular rectangular chambers connected to central refrigeration, vacuum and heating systems as an alternative to present large single chambers. In a modular design, smaller drying chambers are connected in series and each module has its own refrigeration, vacuum and heating system with a means for vacuum equalization and common heating available. The modular concept would permit drying of one large batch with only one assay required since all modules may be operated with a common vacuum and common heating or, in the alternative, flexibly allow a small run to be processed simultaneously and independently, eliminating the uneconomical procedure of drying a small batch in a large chamber. Furthermore, in the event of failure of one module, only a fraction of production is affected by down time for maintenance. Finally, one or more chamber modules may be purchased to meet present requirements and additional chambers may be added as future requirements demand. The modular-type design would also permit acceleration of drying rates with no deterioration in quality, characteristics important in both the food processing and the pharmaceutical industries. Despite these theoretical advantages, however, the most advanced type of modular designs still have had, up to this time, inherent disadvantages and have therefore seen little or no commercial use.

One of the major disadvantages of all present freeze drying equipment lies in the construction of the drying chamber itself. If the drying chamber is of rectangular construction, it requires an inordinate degree of reinforcement about its perimeter because of the low strength characteristics of the rectangular shape. A rectangular shape also presents problems in gasketing to insure tight seals because of the difficulty in securing and sealing gasketing around rectangular corners.

The cylindrical chamber too, has disadvantages despite providing a shape which is easier to fix gasketing to, and despite the fact that the cylinder requires less reinforcement because its inherent strength. In either cylindrical or the rectangular shape, the chamber must necessarily contain shelves in a rectangular configuration to support specimens. Thus, even in a cylindrical chamber, the maximum productive space is not being used. Moreover, this non-productive space is not only useless, but, in fact, increases the drying time required, since the vacuum pump must evacuate all space within the chamber equally and is therefore working to evacuate space not even devoted to the shelving of specimens.

Finally, the use, in present freeze dryers, of the stacked shelf arrangements to support the material to be dried, interferes, whatever the shape of the chamber, with easy cleaning and essential sterilization and provides corners for the collection of contaminants.

A second major disadvantage of present freeze drying units is their inability to satisfactorally transfer heat or cold as is necessary during the freeze drying process. Normally, in present units, heat transfer is accomplished by circulating fluid through coils attached to the underside of the shelves supporting the samples. The coils function to transfer the heat of the fluid by conduction through the shelf to the receptacles supported on the shelf. There is little heat conducted quickly by radiation and the coils produce uneven heating of the shelves. This uneven heating and lack of effective radiant heating is extremely undesirable in attempting to produce the quality product necessary in pharmaceutical operations. Another disadvantage of the present methods of heat transfer is presented by the on and off cycling of the heat transfer fluid which increases the process time and increases mechanical wear problems of the heat transfer fluid pumps.

A disadvantage related to the construction and shape of previous drying chambers is the thickness required in the chamber door. This door is usually made of Lucite and must necessarily be made of sufficient thickness across the diameter or longest span of the chamber to prevent its inward failure during maximum vacuum in the interior of the chamber. As a result of the large spans of single chamber, this door must be quite thick and therefore visibility of the specimens during sublimation is usually poor. This is undesirable since constant observation of new samples or in research operations is often critical.

A final disadvantage related to the structure of previous freeze drying units of the single chamber type is that only one type of sample can be freeze dried at a time because all the shelves are controlled by the same heating and cooling system and subject to the same vacuum. Thus, the freeze drying period cannot be varied and independent freeze drying of different products cannot take place. Even in the improved concept of chamber modules, a plurality of heating and vacuum means are required for independent drying.

Though it is often desirable in a freeze drying process to stopper and seal the receptacles or vials containing the freeze dried material under vacuum, the apparatus used in accomplishing this stoppering also has major drawbacks. Previous apparatus are represented by U.S. Pat. No. 3,022,619 to R. D. Strong et al., in which the receptacles are placed on a rigid supporting plate beneath a platen, which, when desired, is urged downward along guide rods to stopper and receptacles. The plates are normally urged apart by a spring surrounding the guide rods. Since this stoppering apparatus is not easily removable from the chamber, bulk drying and stoppering of smaller samples cannot take place within the chamber at the same time as is often desirable. In addition, most stoppering equipment, like that in Strong, is bulky and mechanically complex which makes it prone to unexpected breakdowns. Present stoppering devices have no built-in safety features by which automatic or independent control can be exercised over the amount of pressure applied to the receptacles and the shelf on which they are supported. Such lack of control could easily lead to breakage of the receptacles or deflection and deformation of the shelf.

DESCRIPTION OF THE INVENTION

This invention provides a novel apparatus which allows great flexibility in the freeze drying of various materials and overcomes the major disadvantages which limit the usefulness and the quality achieved by presently available freeze drying equipment. The novel apparatus of this invention overcomes these disadvantages by use of an elongated, low profile drying chamber module having cylindrically-shaped ends in which the bottom wall of the drying chamber also serves as the shelf supporting the specimens to be dried. Any number of individual drying chamber modules of this configuration may be stacked one above the other in a unit and connected independently through valve means to a condenser in communication with a vacuum pump mounted on a separate equipment unit. In this manner, a vacuum may be applied to each drying chamber independently over any individually desirable length of time.

The chamber itself is formed with generally rectangular plates, top and bottom, held in spaced relationship by vertical supports between them and joined at their corresponding ends by cylindrically-shaped members. The vertical supports resist atmospheric pressure across the chamber span and the cylindrically-shaped ends provide inherent strength at that location. The outside surface of each top and bottom rectangular plate is covered by a sheet of stainless steel continuously welded around its perimeter to the rectangular plate, and spot welded at various points across its inside surface. This forms a hollow quilted envelope through which heat transfer fluid is circulated. The heat transfer fluid is pumped from a heat exchanger mounted on the adjacent equipment unit. By this means, the drying chamber is heated on its top and bottom surfaces over approximately 95 percent of these surfaces to provide an even heat transfer. In addition, the heat transfer fluid is continuously circulated and heat is proportionally controlled, by merely applying heat to the fluid in proportion to the need, to eliminate hot spots. Thermocouples are used to sense the temperature in the heat transfer system and vary it by either refrigerating or heating the fluid in the system. A solenoid valve may be placed in the conduit lines between the heat transfer system and each module to permit independent temperature control of each module.

A Lucite door is hingedly attached to the front of each drying module so that it may be swung over the chamber opening and tightly closed by a latching means to hermetically seal the interior of the chamber from the atmosphere. The unique latching means of this invention has a handle with an attached locking bar laterally movable with respct to the front of the chamber. The handle is pivotal between horizontal and vertical positions and may be rotated in its horizontal position to move the locking bar to lock and unlock the chamber door. In any position, the latching means handle may be pivoted upwardly adjacent the chamber itself to permit the opening of the chamber door immediately below it. The door itself is maintainable in an open position by a novel self-operating sliding catch means which moves upwardly on a support arm as the door is swung open and catches and locks the door in an open position. The door is also mounted on unique reciprocating hinges which act in conjunction with the latching means to self-locate the door, thereby providing a uniform, air-tight seal around the entire periphery of the door.

The stoppering means of this invention is unique in design as well as operation having parallel plates connected by negator springs, and sandwiching between them a number of commercially available expansible bellows inflatable by fluid pressure means connected thereto and mounted on the equipment unit. As these bellows expand, they act to urge the plates vertically apart. The upper plate moves adjacent the upper wall of the chamber and the lower plate presses upon the receptacles to be stoppered. The entire unit may be easily slip-fitted over angle bracket hangers mounted on adjacent vertical supports in each drying module and requires no guide means or other attachment to the chamber itself. The stoppering means is operable under vacuum to stopper receptacles of any height supported by the lower wall of the drying chamber.

The control of the stoppering means provides a safe and sure method of automatically stoppering receptacles after freeze drying. It provides an automatic safety factor requiring resetting to the lowest possible pressure before each operation of the stoppering means. In addition, the control system provides for exertion of a constant maximum force for any height receptacle to effectively stopper the receptacles containing the freeze dried materials without danger of breaking the receptacles or deflecting the supporting wall of the chamber. The control system provides for automatic release of air pressure after stoppering and evacuation of the bellows to return the lower plate to an upper position within the chamber.

One of the major advantages provided by the apparatus of this invention is the great freeze drying flexibility made possible by the use of the modular system. This modular concept allows the operator to vary drying capacity as desired. For example, in small batch runs, one module may be sufficient to provide the drying volume necessary whereas in bulk drying, several modules may be needed. Modules may be added in a stack-type arrangement as drying capacity requirements increase.

A second major advantage is that each drying module may be operated independently from the others, thereby allowing drying of different products or the use of different drying times.

A third advantage of this invention is that the novel elongated cylindrical design of the drying module itself offers a clean unobstructed shape which provides inherent strength at the ends, allows easy cleaning and sterilization since it eliminates shelves, coils and location of the condenser within the chamber itself, and provides shelf capacity similar to the conventional chambers. The elimination of shelves and condensers from the chamber and the use of the chamber wall as a shelf not only maximizes shelf capacity, but also eliminates useless and wasted chamber volume thereby decreasing required drying time and vacuum pump down time.

The elimination of shelves within the drying chamber is also particularly significant since the quilted plate envelopes circulate heat transfer fluid over 95 percent of the outside chamber surfaces. This large wetted surface allows direct, even heat transfer by conduction from below and by radiation from above which eliminates the lack of uniformity in heating and cooling found in present sublimators and also provides for more rapid heat transfer which, in turn, reduces the drying time required.

Heat transfer fluid may be circulated continuously to eliminate hot spots since proportional heater control assures uniform and gradual temperature change. Also, the temperature in each drying chamber may be independently varied by solenoid valves which admit fluid to the transfer envelopes as necessary to maintain the desired temperature in each chamber.

A further advantage related to the chamber construction itself is that the thickness of the chamber door may be drastically decreased because of the low profile shape of the chamber so that visibility is greatly increased over the apparatus of the prior art. In addition, the door of this invention is self-locating and operated by a unique latching mechanism which latches and seals the entire door in one motion and does not obstruct operation of adjacent chamber doors.

The gasketing problem often encountered in rectangular chambers of the prior art is eliminated by the elongated cylindrical design of this invention because the rounded corners make possible the tight and firm adhesion of the gasketing material.

A further advantage over the prior art lies in the unique stoppering assembly which forms a necessary part of this invention in research and small batch drying. The stoppering assembly is a vast improvement over the prior art since its slip-fit design allows easy removability from each chamber to permit the chamber to be completely cleaned and sterilized and to allow bulk drying and stoppering in the same chamber at the same time. The construction of this stoppering assembly also eliminates the bulky mechanical mechanisms previously used in stoppering.

The novel stoppering apparatus and the method of controlling this apparatus insures exertion of a constant, maximum force upon the receptacles to be stoppered which will not result in the breakage of these receptacles of deflection of the stoppering plate or shelf. The automatic safety factor built into the control means requires manual setting and recognition of the correct stoppering height, and provides for cancellation of the stoppering operation while in progress. Such control and safety features are not available on any stoppering mechanism now in use.

Accordingly, it is an object of this invention to provide greater flexibility in freeze drying processes by the use of drying chamber modules.

It is a further object of this invention to provide for the independent operation of each drying chamber module to permit different drying times in different drying chambers and/or the drying of diverse products.

It is a still further object of this invention to provide for variation of the length of the drying cycle in different drying chamber modules by independent valving of each chamber to a vacuum system mounted on a separate equipment unit.

It is another object of this invention to provide a drying chamber module having a wall which acts as a shelf to support the material to be freeze dried thereby maximizing usuable drying space and minimizing non-productive space.

It is also an object of this invention to reduce vacuum pump down time and reduce freeze drying time by eliminating non-productive drying chamber space.

It is still another object of this invention to provide a drying chamber module of such design that necessary reinforcement of the chamber walls is substantially reduced.

It is still a further object of this invention to provide a sublimator apparatus having an improved heat transfer system which includes continuous circulation of heat exchange fluid.

It is also another object of this invention to provide a sublimator apparatus providing more uniform heating of the material to be freeze dried, elimination of hot spots, and more rapid heat transfer.

It is yet a further object of this invention to provide a sublimator apparatus having means to individually control the flow of heat transfer fluid to each chamber.

It is one more object of this invention to provide a sublimator apparatus which improves visibility of specimens during freeze drying.

It is also a further object of this invention to provide a sublimator apparatus having a latching means for opening and closing the chamber door which is easily operable by one person to effectively seal the door, but which may be moved out of the way to allow operation of adjacent doors.

It is still one more object of this invention to provide a sublimator apparatus having an automatically operating catch means to maintain the chamber door in open position and hinge means to self-locate the door with respect to the chamber.

It is another object of this invention to provide a drying chamber module of such design that gasketing may be tightly sealed thereto.

It is a further object of this invention to provide a sublimator apparatus having a stoppering means which may be removably slip-fit into the drying chamber modules.

It is also a further object of this invention to provide a sublimator apparatus having a lightweight, slipfit stoppering means which requires no guide means or a bulky mechanical connection within the drying chamber.

It is also an object of this invention to provide a sublimator apparatus having a stoppering means operated by a controlled process which insures stoppering of receptacles containing freeze dried materials automatically and safely.

It is still another object of this invention to provide a sublimator apparatus including a stoppering means having a stoppering control means which regulates the amount of force used in stoppering to eliminate breakage of receptacles and deformation of stoppering plates and the supporting shelf and which provides for cancellation of the stoppering process at the discretion of the operator.

These and other important objects of this invention will become apparent from the following description taken in conjunction with the drawings illustrating preferred embodiments wherein:

FIG. **1** is a perspective view of the modular compartment sublimator of this invention showing the chamber unit and the adjacent support equipment unit;

FIG. **2** is a top plan view of a single drying chamber module located within the chamber unit shown in FIG. **1**;

FIG. **3** is a rear elevational view of the drying chamber module shown in FIG. **2** in which the hidden lines represent bracing for supporting the door of the chamber unit;

FIG. **4** is a front elevational view of the drying chamber module shown in FIG. **2** having the bracing for supporting the door cut away at one end to show the quilted envelopes through which the heat transfer fluid is circulated;

FIG. **5** is an enlarged front elevational view of one end of a drying chamber module showing the chamber door and the latching means and catch means adjacent thereto;

FIG. **6** is a fragmentary plan view of the latching means shown in FIG. **5** in locked position;

FIG. **7** is a plan view of the latching means shown in FIG. **5** in open position with the chamber door ajar;

FIG. **8** is a perspective view of the stoppering means shown in slip-fit relationship to a drying chamber module according to this invention;

FIG. **9** is a cross-sectional elevational view of the stoppering means, taken generally along line **8—8** in FIG. **8**, shown in its unexpanded position with respect to a receptacle to be stoppered;

FIG. **10** is a cross-sectional elevational view of the stoppering means shown in FIG. **9** in extended position to stopper the receptacle shown;

FIG. **11** is a cross-sectional elevational view of the chamber module and door taken generally along line **11—11** in FIG. **5** and showing in broken lines the door supported in open position;

FIG. **12** is a fragmentary elevational view of the door and catch means shown in FIG. **11** with the door raised slightly above the horizontal;

FIGS. **13–18** inclusive are schematic diagrams of the position and operation of the stoppering means of this invention shown from during freeze-drying, through actual stoppering until after vacuum break; and

FIG. **19** is a schematic diagram of the electrical control circuit operating the stoppering means of this invention.

Referring now to the drawings, and more particularly to FIG. **1**, the modular compartment sublimator of this invention is shown at **10**. This apparatus includes a support equipment unit **12** and a chamber unit **14**. The support equipment unit **12** has a frame **16** on which are mounted various items of equipment necessary to carry out the freeze drying process.

Such equipment includes a low temperature condenser **18**, vacuum pump conduit means **20**, a vacuum pump **22**, heat transfer fluid expansion tank **24**, refrigeration compressors **26**, refrigeration condenser **28**, heat transfer fluid heater **30**, heat transfer fluid cooler **32**, heat transfer fluid pump **34**, condenser conduit means **35**, vacuum cut-out valve **36** disposed on each conduit means 35, fluid pressure means 130 and secondary vacuum pump means 129.

Though some of this equipment is necessary to any freeze drying or sublimation operation, their arrangement with respect to one another on the support equipment unit 12 and the total separation from the chamber 14, is unique in the field of freeze drying. Moreover, as shall be more clearly pointed out below, many of these conventional pieces function in a novel manner with respect to the chamber unit 14.

The chamber unit 14 includes a chamber cabinet 38 which surrounds a chamber module support structure which is not shown and an instrument recording and control panel 40. The chamber unit 14 includes a plurality of individual drying chamber modules 50 in vertical stacked relationship supported on this structure.

Referring now to FIGS. 2–4, each drying chamber module 50 has a top plate 44 forming an upper wall and a bottom plate 52 forming a lower wall and specimen supporting shelf. Top plate 44 and bottom plate 52 are disposed substantially parallel to one another and held in spaced relationship by internal vertical supports 54 which serve to divide the interior of the module 50 into compartments 53 of substantially equal dimension. The supports 54 are purposely shorter than plates 44 and 52 to allow pressure equalization between compartments 53. The corresponding ends of the top plate 44 and bottom plate and shelf 52 are joined by cylindrically-shaped ends 48. One end of the module 50 is closed by vertical back plate 46 which is fixedly joined to top plate 44, bottom plate 52 and cylindrical ends 48 about their periphery. The shell construction forms the elongated hollow chamber module 50 of generally low profile shape which is adapted for receiving samples of materials to be freeze dried within each of its compartments 53 and has sufficient strength by virtue of vertical supports 54 and cylindrical ends 48 to be evacuated during the freeze drying process without failure or deformation.

Sample tray guide tracks 55 are disposed on opposing faces of the vertical supports 54 at the lower end of these supports to guide the sample trays as they are inserted into compartments 53 and maintain them in relatively fixed position. Near the upper end of the vertical supports 54 similarly shaped stoppering means support brackets 57 are disposed on opposing faces of each vertical support 54 to support a means used for stoppering the receptacles containing the materials to be freeze dried which will be discussed in greater detail below.

As can be seen clearly in FIG. 2, the outside surface of top plate 44 of chamber module 50 includes transverse vertical bracing 58 which not only reinforces the long span of the chamber, but also acts to support the insulation which is placed around the chamber 50 in the final manufacturing process of the chamber unit 14. Below this bracing 58 and attached directly to the outside surface of the top plate and upper wall 44 of the module 50 is a TEMP-PLATE heat transfer fluid circulation means or envelope 56. This envelope 56 has a quilted surface and is formed by a process developed by the Paul Mueller Company of St. Louis, Mo. In this process, sheets of metal of approximately the same size as the plates 44 and 52 of the module 50 are attached to the outside surfaces of these plates by welding around their perimeter. The sheets are then spot welded to plates 44 and 52 at various points across their surface. After this is done, a gas such as nitrogen is urged between the sheets and the plates under pressure. This acts to inflate or pop the sheets at the points not spot welded to the plates 44 and 52, creating the quilted envelope 56 which permits circulation of heat transfer fluid over at least 95 percent of the surface of plates 44 and 52 since the unattached areas of the thermoplate envelope 56 are significantly greater than the spot welded areas.

The heat transfer fluid is pumped from transfer fluid heater 30 on equipment unit 12 through transfer fluid inlet conduit 60, through inlet elbows 62 and into and through the interior of quilted envelope 56. The heat transfer fluid flows front to back and exits through fluid outlet elbows 64, to fluid outlet conduit 65 which discharges it to cooler 32 on equipment unit 12. The fluid is then moved through pump 34 and back to heater 30 to complete the transfer circuit. An expansion tank 24 to provide for expansion of heated fluid is also mounted on equipment 12 in parallel to the transfer circuit. A liquid level control shuts down the transfer system if necessary. There is also provided on the module 50 a heat transfer fluid drain and clean out 66.

At the rear of the chamber module 50, as can be seen in FIGS. 2 and 3, there is a chamber vacuum outlet port and flange 68 which is connected to condenser conduit 35 for communication with the condenser 18. The air and water vapor in the chamber is evacuated through this port 68 during the freeze drying operation.

The front of chamber module 50 is shown in FIG. 5. This figure shows the module door support frame 49 which is attached to the chamber unit support frame which acts to support each module within the frame. The front opening of each chamber is covered by a chamber door 70 usually fabricated of a Lucite material to permit visibility during the freeze drying operation. A gasket 74 is fixed and sealed about the periphery of the front opening of the module 50 so that when the chamber door 70 is brought securely adjacent to it, an air-tight seal will be formed.

The door 70 is pivotally mounted with respect to the front opening of the chamber 50 on hinges 72 which are slidably mounted on the module door support frame 49. Hinges 72, as will be explained in greater detail below, are spring biased so that the pivot point of the door may be moved inwardly and outwardly with respect to door support frame 49, depending upon whether the door is in open or closed position. This movability of the pivot point enables the door to be self-located with respect to the front opening of the chamber module 50.

At opposite ends of each chamber door 70 is disposed a door support means 76. This support means 76 includes an L-shaped support arm 78 which is pivotally attached to the door support frame 49 at the inward end of the foot portion of the "L" so that the upwardly extending unattached leg portion of the L may be moved inward and outward with respect to the front opening of the chamber 50.

A catch means 80 is slidably disposed on the upwardly extending leg portion of the support arm 78 and movable toward the upper end of this arm as the chamber door 70 is opened. Catch means 80 is formed by two substantially parallel plates, one disposed on either side of support arm 78, and attached to one another, a U-shaped longitudinally extending slot 82 being formed in one plate to receive an upper stop pin 84 disposed on the upper end of the vertical leg of the support arm **78.** Catch means **80** also has a swinging pawl **86** mounted thereon, which serves to engage a notch **87** formed at the upper end of the vertical leg of the support arm **78.** The catch means **80** also encloses a friction slide **88** adjacent one surface of the support arm **78** which slides freely with respect to the support arm **78** and is frictionally movable relative to the catch means **80** depending upon the adjustment of a slide friction retainer nut **89.** This friction slide **88** has a longitudinally U-shaped slot **85** formed in it which generally corresponds to the shape of the slot **82** formed in the catch means **80.** The slide friction retainer nut **89** rides in a second slot formed in catch means **80** below the U-shaped slot **82.** The catch means **80** itself is mounted on a bracket **73** disposed on one edge of the chamber door **70** so that when the door is moved upwardly to an open position, the catch means **80** moves with it, upper stop pin **84** enteringc the U-shaped slot **82,** until the pawl **86** engages the notch **87.** The door is thereby supported on the support arm **78** in a horizontal, open position.

A latching means **90** is also disposed adjacent the door **70** at the front opening of the chamber **50** to provide an effective means for closing and sealing the door so that the chamber **50** is rendered air tight, as is essential, during the freeze drying process. The latching means **90** includes a locking bar **92** having a plurality of locking pins or rollers **94** spaced along its length. This bar **92** is supported by spaced flanges or brackets **95** mounted on the door support frame **49.** These brackets **94** allow the bar **92** to freely slide transversely to the front opening of the chamber **50.** The latching means **90** has a handle portion **97** which is connected to the locking bar **92** at one end thereof, and acts to move the locking bar **92** with respect to the chamber door **70.** This handle portion **97** includes a pivot arm **98** which is rotatable in a horizontal plane about a point **96** fixed to the door support frame **49,** and an adjustable connecting arm **100** having one end attached to the pivot arm **98** and the opposite end attached to the locking bar **92,** thereby linking the two. A handle **102** is also mounted on the pivot arm **98** at the end opposite the point **96** where the pivot arm **98** is fixed to the chamber unit and which itself is pivotal in a vertical plane about the point of its mounting.

Thus, the handle portion **97** of the latching means **90** is movable in a horizontal plane about pivot point **96** such that it moves the locking bar **92** transversely to the chamber door **70.** The bottom edge of the door **70** has mounted thereon, a plurality of latching plates **104** which have a finger portion **106** extending substantially parallel to the edge of the door along their length. This finger portion **106** has an outwardly facing edge which forms an acute angle with the edge of the door. The positioning of these latching plates **104** corresponds generally to the location of the locking pins or rollers **94** mounted on the locking bar **92** so that the rollers **94** will engage the latching plate **104** when the door is in a down or vertical position to be closed. As locking bar **92** is moved transversely to the door **70,** rollers **94** are moved along the increasing diameter of the finger **106** of the fixed latching plate **104** to push the door **70** inwardly against the gasket **74** around the periphery of the front opening of the chamber **50** thereby closing and sealing the door.

The stoppering means **110** of this invention, which is shown in more detail in FIGS. **8–10,** functions to close or stopper receptacles containing materials which have been previously freeze dried, under vacuum, at the termination of the freeze drying process, in order to insure specimen preservation. As can be seen in FIG. **8** the stoppering means **110** may be removably mounted within each compartment **53** of each drying chamber module **50.** Thus, the stoppering means **110** may be easily removed for bulk drying processes, where stoppering of vials is not required.

As shown in FIG. **9** the stoppering means **110** includes an upper plate **114** having downwardly extending flange about its periphery and serving to support the stoppering means **110** on the stoppering means support brackets **57.** A lower plate or stoppering platen **116** is disposed substantially parallel to upper plate **114** and actually comes in contact with the stoppers **128** disposed in the receptacles or vials **126** containing the freeze dried material. The lower plate **116** is connected to the upper plate **114** by return or negator springs **120** which urge the lower plate toward the top plate. Rubber bellows **118,** such as Firestone Airrides, are sandwiched between the top plate **114** and the lower plate **116** and fastened at their opposite ends to the interior surface of each plate. These bellows **118** are formed by rigid top and bottom plates which are firmly and air tightly crimped to a continuous side wall of rubberized material. When compressed air is admitted to the interior of the bellows **118,** they inflate in a manner similar to an automobile tire and thereby expand, overcoming the force of the negator spring **120** and forcing plate **114** and plate **116** apart in a vertical direction as shown in FIG. **10.** Such expansion continues until lower plate **116** contacts split stopper **128,** upper plate **114** contacts wall **44** and stopper **128** is forced into the receptacle **126.** As will be explained, at stoppering, the interior and exterior forces on the module are substantially in balance.

The functional interrelationship of the features of this invention set forth above will better be understood by the following explanation of the operation of the invention as a whole and the operation of each feature with respect to it.

Once the individual drying chamber module **50** has been fabricated as described above, it is insulated at its top, bottom, and rear by a Freon blown plastic or rubber to prevent heat loss. The individual modules are then placed on the support bracing which forms the frame of the chamber unit **14** and fixed to that bracing by bolting or other suitable means. Each heat transfer conduit **60** and **65** and vacuum port and flange **68** of each individual drying chamber module **50** may then be connected with its corresponding conduit on the equipment unit **12.** There will be one vacuum conduit **35** between each drying chamber module **50** and the condenser **18,** each conduit **35** having a cut-out valve **36** disposed in it. The heat transfer conduits **60** will be connected to a heat transfer conduit manifold (not shown) which is ultimately in communication with the transfer fluid heater **30.** The chamber door **70** is then mounted on the module **50** on hinges **72** along with the corresponding door latching means **90** and door catch means **76** as shown in FIG. **5.**

If the operation to be performed will require stoppering, the stoppering means **110** are slid into the compartments **53** of the desired modules **50** by placing the flanges of the upper support plate 114 above the stoppering means support brackets 57. The freeze drying operation will be controlled, instrumented and recorded by panel 40 shown in FIG. 1.

The materials to be freeze dried are inserted, usually in receptacles or vials 126 on trays into the compartments 53 of drying module 50 as desired. If different types or compositions of materials are to be dried, this invention permits them to be inserted into separate drying chambers 50 and dried independently by varying drying times or drying temperatures. The trays (not shown) containing the receptacles 126 are placed between the sample tray guide tracks 55 which maintain them in a relatively fixed position with respect to the shelf or lower plate 52 of the chamber 50. When the materials are in position, the chamber door is pivoted downwardly on its hingers 72 as shown in FIG. 7 to a position adjacent and covering the front opening of the chamber 50. In this position, shown in FIGS. 7 & 11, the top of the chamber door 70 comes to rest against the gasket 74 which is disposed about the perimeter of the front opening of the chamber 50. The upper portion 75 of hinge 72 has a shaft 79 extending through the door support frame 49 toward the rear of the chamber module 50. The shaft 79 and upper hinge portion 75 are normally urged by a spring means 77 toward the rear of the chamber 50.

When the door is in position for closing, the handle 102 of the latch handle portion 97 of the latching means 90 is grasped by the operator and moved to a horizontal position. In this position, the handle 102 and the pivot arm 98 to which it is connected, act as a lever and may be rotated about the fixed pivot point 96 in a horizontal plane thereby pulling the locking bar 92 which is connected to the pivot arm 98 by connecting arm 100, a predetermined close stroke distance. This distance is determined by adjusting the threaded middle portion of the connecting arm 100 to vary the length of the arm 100 and thereby the distance of the close stroke. As the locking bar 92 is pulled transverse to the front of the chamber module 50, the locking pins or rollers 94 mounted on the locking bar 92 are moved with respect to the latch plates 104 mounted on the lower edge of the door. The rollers 94 move along the fingers 106 of the latch plates 104 and, since the rollers 94 are held in fixed spaced relationship to the front face of the chamber module 50 and the fingers 106 increase diameter in the direction the rollers 94 are moved for closing, the rollers 94 act to urge the bottom of the chamber door 70 toward gasketing 74 at the bottom of the front opening of chamber 50. This action overcomes the oppositely directed force of hinge spring means 77 tending to open the door and causes shaft 79 and the upper hinge portion 75 attached thereto to be moved outwardly as shown in FIG. 11. Thus, when the handle porton 97 of latching means 90 is moved fully to the locking position, the door 70 exerts equal forces at both its top and bottom on the gasketing means 74 and is thereby closed and sealed in a substantially vertical position. In this way, the door 70 is not located with respect to a permanently fixed upper hinge but becomes self-locating with respect to the front opening of chamber module 50 so that a better and more uniform seal around the entire peripheral interface between the front opening of chamber 50 and the door 70 is assured.

Once the chamber has been effectively hermetically sealed from the outside environment, the drying process itself may be begun. If the materials have not as yet been frozen, it will be necessary to cool the heat transfer fluid to an extremely low temperature and circulate it through the envelopes 56 at the top and bottom of each chamber module 50.

After the materials have been frozen, the freeze drying or sublimation process is the same as in conventional freeze dryers. However, the structure of the present invention allows several important differences. First, the chamber is independently valved with respect to the condenser and vacuum means in communication with the condenser to permit separate vacuum operation in each module 50 and to allow a variation in vacuum duration in different chambers and a variation in the length of the freeze drying cycle. Thus, in situations where it might be important to limit the cycle for one particular product, for example, a pharmaceutical, this can be done.

Second, the heat transfer fluid is manifolded in parallel outside and within the module. There is a fluid manifold (not shown) manifolding fluid from the heater 30 to each chamber and six inlet elbows 62 which again manifold the heat transfer fluid within the envelopes 56 forming the walls of each chamber. This accomplishes complete and smooth circulation of heat transfer fluid across the top and bottom surfaces of each chamber 50. In addition, solenoid valves (not shown) may be installed in the heat transfer conduits 60 leading to each particular chamber so that, even though the heat transfer fluid is pumped continuously, the valves 60 only admit fluid sufficient to maintain the temperature which is desired within each chamber 50. Since the chamber temperature, together with the length of the cycle is one of the primary factors in freeze drying, independent variation of the drying temperature in chambers 50 by the operation of solenoid valves will permit the drying of different products within different chambers.

As has been previously stated, it is possible, because of the structure of this invention, to circulate heat transfer fluid continuously though the envelope 56 to permit uniform, smooth heating of the materials during drying. In addition to the chamber construction, the factor making this uniform circulation possible is the proportional heat control of the heat transfer fluid by the heat transfer heater 30 which increases voltage as necessary to meet the demands of the system. A sensor also senses the temperature of fluid entering cooler 32 and controls cooler 32 accordingly.

When the materials have been freeze dried according to the satisfaction or desire of the operator, the stoppering operation may be commenced. When each stoppering means 110 is inserted into a compartment 53 of the particular drying module 50 before freeze drying, it is connected, through a stoppering conduit 121 to a source of fluid pressure such as air compressor 130 mounted on the equipment unit 12. Also mounted on equipment unit 12 for use in the stoppering operation is one secondary vacuum means 129 for all stoppering means 110 in each chamber module 50, vacuum solenoid valves 132, and fluid pressure solenoid valves 134. An electrical control circuit to regulate the stoppering process is operable from the control panel 40. These elements are all represented in the schematic views of the stoppering operation shown in FIGS. 13 to 18.

With reference then to FIGS. **13** to **18,** the method of operating the stoppering means **110** is as follows:

1. The stoppering secondary vacuum means **129,** is switched on with the main vacuum pump 22 to prevent partial inflation of the rubber bellows **118.** The negator springs **120** also assist in preventing movement of the plates **114** and **116;**

2. At the end of the freeze drying cycle, the stoppering system is switched on at the control panel **40,** activating the air compressor **130** and the control circuit. Each chamber **50** has an individual control on the control panel **40.** Each individual control controls all the stoppering means **110** used in that particular chamber **50;**

3. A pressure control knob or first actuator means **136** is then dialed to position "activate" as shown in FIG. **14.** This closes the normally opened vacuum line solenoid valve **132** for that chamber and lights the stoppering push button switch or second actuator means **138;**

4. The pressure control knob **136** is then dialed to the height of the stoppered receptacle **126.** The stoppering push button **138** is pushed opening the normally closed air line solenoid valve **134** to admit compressed air into the bellows **118.** If the stoppering button or second actuator means **138** should be released, the system will hold until the button **138** is pushed once again. If the stoppering button **138** is held down, the stoppering platen or lower plate **116** will descend, with the inflation of the bellows **118** by the application of air pressure, and begin to press the stoppers **128** into the receptacles **126** as shown in FIG. **16.** When the stoppering platen **116** comes in contact with the stoppers **128** and exerts a sufficient force on them, the support plate **114** will be lifted off the brackets **57** until it comes adjacent and in contact with the module upper plate **44.** Therefore, at the point stoppering, the support plate **114** exerts an upward force on the upper plate or wall **44** of the chamber module **50** while the plate **116** exerts a downward force on the receptacles **126** to be stoppered and the lower plate or wall **52** of the module **50** which acts as a shelf supporting them. Thus, the module **50** becomes a structural element of the stoppering system. The external atmospheric pressure on the chamber (14.7 psi.) acts to balance the internal forces exerted by the stoppering assembly (about 15 psi.);

5. At the point of stoppering, the pressure control **162** senses a maximum pressure, as predetermined, for the stoppered receptacle height, and closes the air line solenoid valve **134** and opens the vacuum lines solenoid valve **132**; and,

6. The air, under high pressure in the rubber bellows **118,** then surges into the vacuum line where approximately 80 percent of it is vented to the atmosphere through a large oriface pressure relief valve **140.** The remaining air is evacuated by the vacuum pump **129** which is now in communication with the interior of the rubber bellows **118.**

After completion of the stoppering operation and breaking of the chamber vacuum to remove the freeze dried materials, a vacuum break switch or third actuator means **142** is activated to bring the entire stoppering means **110** to atmospheric pressure, thereby preventing vacuum pump oil migration into the system and eliminating stress on the rubber bellows **118.**

A cancel switch or cancel actuator means **144** may also be included in the stoppering system to enable the operator to manually initiate pressure release and thereby terminate the stoppering operation at any point in the operation. Like any inflatable bellows, the rubber bellows **118** vary as to the ratio of force exerted to air pressure applied to the amount of linear extension of the bellows. To cope with this characteristic in the stoppering unit, a pressure control has been used to vary applied air pressure and develop a constant exerted force of 1,000 lbs. per bellows for stoppering at all heights. The adjustable pressure control switch **136** has, consequently, been calibrated in centimeters of stoppered receptacle height rather than pressure. A millimeter scale and caliper jaw are mounted on the stoppering control panel **40** allowing the operator to measure actual stoppered receptacle height which is transferred directly to the control dial. The initial setting of the control to "activate" drops the pressure set point to the lowest value. The table following illustrates the variation in air pressure required to develop the constant exerted force of 1,000 lbs. per bellows.

VIAL HT. M.M.	40	60	80	100	18120	130
PRESSURE PSIG	95	45	29	22.5	1818	16.5

The pressure control is required to prevent damage to receptacles and to limit stresses in the vacuum chamber. The pressure control switch **136** must always be initially set to the greatest stoppered receptacle height which corresponds to the least pressure before stoppering can be accomplished. Likewise, when the cancel button has been pushed, such reactivation must be made.

Though the use of rubber bellows **118** of the Firestone Airride type is preferred because of the extreme stresses the material is forced to undergo due to pressure and temperature changes, it is possible to substitute a thin bladder which could be inflated by atmospheric pressure alone to urge the stoppering plates apart and accomplish stoppering. Such a naturally inflatable bladder would not necessitate the elaborate safety controls of the present system.

After freeze drying has been accomplished and stoppering of the receptacles if desired, the chamber door **70** may be opened to remove the freeze dried materials from the chamber **50.** To open the chamber **50,** the handle **102** of the latch handle portion **97** of the latching means **90** is pivoted to a horizontal position. The latch handle portion **97** is then moved in a horizontal plane in the opposite direction of locking to move the locking bar **92** across the bar support brackets **95** and move the rollers **94** along the fingers **106** of the latch plates **104** and off of them to release the door **70** at its bottom. As the bottom of the door **70** swings partially ajar, hinge **72** moves toward the rear of the chamber **50** as the upper hinge portion **75** is urged inwardly by spring **77.** The operator may then pivot the door **70** upwardly on the hinge **72,** which at the urging of the spring means **77,** moves adjacent the face of the door support frame **49.** As the door **70** is opened, the catch means **80** moves upwardly on the vertical leg of the support arm **78** thereby moving the support arm outwardly from the front of the chamber **50.** At a position where the door lies in a substantially horizontal plane, the pawl **86** engages the notch **87** formed in the support arm **78.** The operator may then release the door **70** which will be held by support arm **78** in the horizontal position.

When it is desired to move the door to a closed position once again, the door catch means 80 is released by raising door 70 a few degrees above the horizontal. The upper stop pin 84 which rides in the U-shaped slot 82 of the catch means 80 when the door is in horizontal position is thereby urged against the end of the corresponding U-shaped slot 85 formed in the slide friction plate 88. The catch means 80 is moved upwardly with the door relative to the support arm 78 such that the upper stop pin 84 moves slide friction plate 88 downward relative to the catch means 80 and the support arm 78 as allowed by the friction retainer nut 89 riding in an elongated slot in the slide friction plate. As the slide friction plate 88 is moved downward, a shoulder portion 81 on the edge of this plate adjacent the pawl 86 comes in contact with the pawl 86 and, as the plate 88 is pushed progressively downward, moves the pawl 86 out of the notch 87, thereby releasing the door 70 with respect to the support arm 78. When the pawl 86 is disengaged from the notch 87, the door 70 may be dropped to a locking position.

The drying chamber modules 50 of this invention may be formed of any suitable material having high strength, a smooth surface for easy cleaning and sterilization, and resistance to chemical and atmospheric corrosion, such as stainless steel, which is used in the preferred embodiment of this invention to form the complete shell of the drying module.

The latching means and catch means of this invention should also be formed of a corrosion resistant material which is easy to keep clean, is strong and affords a good appearance, such as stainless steel.

The door 70 may be constructed of any suitable material which is strong, durable, able to resist opposing extremes in temperature, is impervious to fluids and affords high visibility through its surface, such as the material sold under the trademark Lucite.

The gasketing means 74 may be formed of any suitable material which is resistant to corrosion, is impervious to fluids, and is resilient, such as rubber. The heat transfer system is fully hermetic Freon 11 or TCE utilizing refrigeration piping practice to insure a leak-free system. The conduit means should be made of materials which are impervious to fluid, corrosion resistant and which may be easily formed in any desired shape. Conventional piping material such as steel or copper may be used and should be insulated with conventional insulation material. The heat transfer pump is a canned pump to provide a leak-free hermetic system.

The condenser is a self contained unit having a vertical cylindrical shape with a plurality of Mueller TEMP-PLATES disposed vertically in a fan shape within it for direct expansion of the refrigerant. The condenser is manufactured from stainless steel or other suitable metals and is insulated with urethane foam. The condenser refrigeration system consists of two compressors sized according to the capacity of the system. The ice which forms in the condenser is usually removed by water for rapid turnaround and protective rinsing of the TEMP-PLATES. The vacuum pump is a two-stage, oil-sealed pump sized according to the number of drying chambers.

The instrumentation used in the control panel should include a proportional heat controller to supply variable voltage to the electric heater to prevent overshoot and fluid temperature variation above and below the point and thereby control the temperature of the heat transfer fluid. It should also include an electric indicator controller for shelf temperature and an electric indicator controller for condenser temperature having an adjustable thermostat. A McLeod gauge, preferably of the tilting type, is used to measure the vacuum in the chamber.

In FIG. 19 there is shown a diagram of the electrical circuitry used to operate and control the stoppering means 110 of this invention. The portion of the circuit shown in dashed lines is that ncessary for control of stoppering in a single chamber and similar portions may be serially connected to accomplish stoppering in other chambers.

The operation of this circuit will be explained by reference to the sequential operation of the stoppering means. When the secondary vacuum pump 129 in communication with the stoppering means 110 in each module 50 is switched on automatically with the main vacuum pump 22, the contacts in the vacuum break relay 150 are opened to de-energize the vacuum break switch and prevent communication of the stoppering means 110 to the atmosphere even if the vacuum break switch 142 is pressed. When it is desired to initiate stoppering, a stoppering system switch 152, which provides electrical power to the stoppering system but does not activate any of the other components of the system is switched on.

Knowing the stoppered height of the receptacles within the particular module in which stoppering is to occur, the operator turns the first actuator means or pressure control knob 136 to a position termed "activate", which closes a single-pull, double-throw, micro-switch 156 from the normally closed pole to the normally open pole which energizes relay 158. Relay 158, which is a form of double-pole double-throw switch, thereby supplies an electrical potential to a stoppering push-button switch or second actuator means 138. If the operator, however, pushes stopper button 138 at this time, nothing would happen except that the relay 158 would click. This safety feature insures that the operator must set the first actuator means 136 to the proper stoppered receptacle height before actually attempting to stopper.

When relay 158 is energized, it also energizes the vacuum solenoid valve 132 for this particular module 50. Valve 132 is normally open to hold the stoppering means 110, under vacuum. However, when it is energized, it closes and thereby cuts off communication between the bellows 118 of the stoppering means and the secondary vacuum pump. The bellows are thereby held under whatever vacuum they have at that time.

The operator then dials pressure control knob 136 to the actual height of the stoppered receptacles 126 and presses stoppering push-button switch 138, which energizes fluid pressure or air line solenoid valve 134. This valve opens the air line 122 in communication with the stoppering means which admits air to the bellows 118 and causes platen 116 to descend toward the receptacles to be stoppered.

The pressure control unit is a commercially available item which includes a piston actuated micro-switch 162. When the pressure exerted by the stoppering means being operated reaches the pressure which is set in terms of receptacle height by the control knob 136, switch 162 automatically flips, and, in so doing, de-energizes relay 158. This in turn closes air line solenoid valve 134 and opens vacuum hold solenoid valve 132 to communicate bellows **118** of stoppering means **110** with the vacuum conduit **124.** A spring-loaded pressure-release valve **140,** which is disposed in the vacuum conduit **124,** senses the resulting surge of high-pressure air and opens at approximately one half pound of pressure to release most of the high pressure air to the atmosphere. It remains open until equilibrium with the atmosphere is reached at which point it closes. Any remaining pressurized air in the bellows **118** is then withdrawn by the secondary vacuum pump. This pump down and return of the platen **116** to an upper position is assisted by negator spring **120.**

If any difficulty might occur during the stoppering 0peration which could be damaging to the receptacles, a cancel switch or cancel actuator means **144,** which is a single-pole push-button switch in series with the pressure control switch **162,** is provided and may be pushed any time after switch **152** has been activated to completely deflate the bellows and terminate stoppering.

At the end of the stoppering operation, and when it is desired to remove samples from all modules **50,** the main vacuum system is switched off, which will deactivate the stoppering means control system shown in FIG. **19.** Since the rubber bellows **118** are under vacuum, in order to avoid stresses on the bellows, a vacuum break or third actuator means **142** is pushed. This opens a vacuum break solenoid valve **146** and all the air line solenoid valves for each module **134** by activating relay **150.** The vacuum break solenoid valve **146** acts on a single manifold, which, when open, admits air through the air line solenoid valves **134** into each of the stoppering means **110** in the individual modules **50,** thereby balancing internal and external pressure on the stoppering means **110.** The leads to the instrumentation switch on the upper right of the FIG. **19** go to an incidental switch on the control panel which acts as a source of power supply to operate the vacuum break and must be on in order to break vacuum in the stoppering means.

The pressure control is commercially available and consists of a pressure activated spring loaded piston disposed near micro-switch **162.** Micro-switch **162** itself pivots to and from the piston so that when receptacle height is adjusted by control knob **136,** the micro-switch is positioned so that it will be flipped by the piston upon the piston reaching the position corresponding to the maximum desired pressure. Terminal **164** is also provided in the event of a need for a stoppering limit switch in the chamber to either augment or eliminate the pressure control.

Upon consideration of the foregoing, it will become obvious that those skilled in the art that various modifications may be made without departing from the spirit of the invention embodied herein.

We claim:

1. An apparatus particularly adapted for freeze drying materials contained in receptacles and for stoppering said receptacles comprising, in combination:

a plurality of individual self-supporting drying chambers, each drying chamber having a lower wall which also forms a shelf to support a number of receptacles containing material to be freeze dried;

hinge means reciprocally mounted on each drying chamber adjacent an open end of said drying chamber, said hinge means having a door of substantially similar dimension to said open end of said chamber attached for rotation thereabout, said door being rotatable on said hinge means to cover said open end of said chamber, said hinge means allowing movement of said door relative to said open end of said chamber such that said door is self-locating with respect thereto;

latching means disposed on each drying chamber for opening and closing tightly said door mounted on said drying chamber, said latching means being always movable to a position that does not obstruct the opening and closing of similar doors on adjacent drying chambers:

door support means disposed adjacent said drying chamber door and movable with said door, said support means automatically operating to maintain said door in an open position and releasable to allow said door to be moved over the open end of said chamber in position for closing by said latching means;

an equipment unit separate from said drying chambers having heat transfer means, vacuum means, condenser means and stoppering power means each mounted thereon, said heat transfer means, said vacuum means and said condenser means in communication with each of said drying chambers independently and operable respectively to control the temperature and to evacuate and condense water vapor from each of said chambers independently, such that materials placed in each of said chambers may be freeze dried; and,

unitary stoppering means removably mounted within each of said drying chambers, said stoppering means in each chamber being movable, upon operation of said stoppering power means in communication therewith, to stopper the receptacles supported on said shaft in said chamber after the material contained in these receptacles has been freeze dried.

2. The combination set forth in claim 1 wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plates acting to directly conduct thermal changes to the receptacles and the materials contained therein within the chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

3. The combination set forth in claim 1 wherein said hinge means includes, a chamber portion mounted adjacent said drying chamber above the open end thereof and having an elongated shaft extending normally to the plane of the open end of said chamber, a door por-

tion attached to said chamber door and connected to said chamber portion for rotation therearound, a resilient means adjacent said shaft and urging said hinge means in the direction of the open end of said chamber.

4. The combination set forth in claim 1 wherein said latching means includes, locking rod means disposed adjacent the open end of said drying chamber and movable laterally of said wall forming a shelf of said drying chamber, said locking rod means having a plurality of upright protrusions disposed along its length, latching plate means disposed on said door of said drying chamber to engage said upright protrusions on said locking rod, adjustable connecting means having a first end attached to one end of said locking rod means, lever arm means attached to the opposite end of said connecting means and pivotal horizontally about a fixed point adjacent said open end of said chamber, and handle means to rotate said lever arm means about said fixed point to move said locking rod means laterally of said door and said latching means thereby locking and unlocking said door with respect to the open end of said drying chamber, said handle means being always rotatable to a vertical position to allow a door of an adjacent drying chamber to be moved to any position without obstruction.

5. The combination set forth in claim 4 wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plates acting to directly conduct thermal changes to the receptacles and the materials contained therein within said chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

6. The combination set forth in claim 1 wherein said equipment unit includes a frame, a condenser mounted on said frame, said condenser in communication with said plurality of drying chambers through a plurality of individually valved conduit means connecting each of said drying chambers with said condenser independently, vacuum means mounted on said frame in communication with said condenser to withdraw water vapor from each of said drying chambers into said condenser, refrigeration means mounted on said frame and in communication with said condenser, ehat transfer means mounted on said frame including heat exchanger means containing heat transfer fluid and proportionally controlled means to heat said fluid in valved communication therewith, and continually operating pump means in communication with said exchanger means to circulate said heat transfer fluid through conduit means to each of said drying chambers, and stoppering power means, including fluid pressure means and secondary vacuum means, mounted on said frame. heat

7. The combination set forth in claim 1 wherein said stoppering means includes a support means and a platen means connected and being maintained in parallel relationship to one another by resilient means disposed between said platen means and said support means and urging said platen means upwardly toward said support means and expansible means sandwiched between said support means and said platen means and inflatable to move said platen means and said support means in vertically opposite directions, said stoppering means being easily and quickly mounted in said drying chamber above said lower wall thereof by hanging said support means in said chamber, and said stoppering power means includes fluid pressure means mounted on said equipment unit in communication with said expansible means through conduit means to inflate said expansible means, control means in communication with said fluid pressure means to regulate the admission of pressurized fluid into said expansible means so that a maximum constant stoppering force is always exerted by said platen means on the receptacles to be stoppered despite the height of such receptacles, valve means to release pressurized fluid contained in said expansible means after stoppering, and secondary vacuum means to evacuate said expansible means of residual pressuirzed fluid and thereby maintain equilibrium between said expansible means and the interior of said drying chamber, said stoppering means being operable by actuation of said stoppering power means such that said support means is moved adjacent an upper wall of said drying chamber as said platen means is moved adjacent said receptacles during stoppering to allow the external forces on said drying chamber to assist in balancing the internal forces created by said stoppering means.

8. The combination set forth in claim 7 wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said support means having brackets disposed on them on which said stoppering means may be slidably mounted, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plate acting to directly conduct thermal changes to the receptacles and the materials contained therein within said chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining together said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

9. The combination set forth in claim 1 wherein said door support means includes an elongated support arm having a lower end attached to said drying chamber at a point adjacent said open end thereof, said support arm pivotal about said point, and having an upper end with a notch formed therein and a stop means formed thereon, and a catch means slidably mounted on said support arm to travel from one end of said arm to the opposite end, said catch means pivotally attached to said chamber door, said catch means having a pawl pivotally attached thereto, said pawl engaging said notch on said support arm when said catch means is slidably moved toward the upper end of said support arm by the opening of said chamber door to maintain said door in an open position, said catch means having a friction slide means frictionally attached thereto for movement therewith relative to said support arm until, said door is moved to move said catch means and said friction slide means along said support arm such that said stop means bears against said friction slide means thereby causing said friction slide means to move relative to said catch means to disengage said pawl to permit said door to be closed.

10. The combination set forth in claim 9 wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said support means having brackets disposed on them on which said stoppering means may be slidably mounted, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plate acting to directly conduct thermal changes to the receptacles and the materials contained therein within said chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining together said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

11. The combination set forth in claim 10 wherein said latching means includes, locking rod means disposed adjacent the open end of said drying chamber and movable laterally of said wall forming a shelf of said drying chamber, said locking rod means having a plurality of upright protrusions disposed along its length, latching plate means disposed on said door of said drying chamber to engage said upright protrusions on said locking rod, adjustable connecting means having a first end attached to one end of said locking rod means, lever arm means attached to the opposite end of said connecting means and pivotal horizontally about a fixed point adjacent said open end of said chamber, and handle means to rotate said lever arm means about said fixed point to move said locking rod means laterally of said door and said latching means thereby locking and unlocking said door with respect to the open end of said drying chamber, said handle means being always rotatable to a vertical position to allow a door of an adjacent drying chamber to be moved to any position without obstruction, and said hinge means includes, a chamber portion mounted adjacent said drying chamber above the open end thereof and having an elongated shaft extending normally to the plane of the open end of said chamber, a door portion attached to said chamber door and connected to said chamber portion for rotation therearound, a resilient means adjacent said shaft and urging said hinge means in the direction of the open end of said chamber.

12. The combination set forth in claim 11 wherein said stoppering means includes a support means and a platen means being maintained in parallel relationship to one another by resilient means disposed between said platen means and said support means and urging said platen means upwardly toward said support means, expansible means inflatable to move said platen means and said support means in vertically opposite directions, said expansible means disposed between said support means and said platen means and stoppering power means, including fluid pressure means mounted on said equipment unit in communication with said expansible means through conduit means, to inflate said expansible means, control means in communication with said fluid pressure means to regulate the admission of pressurized fluid into said expansible means so that a maximum constant stoppering force is always exerted by said platen means on the receptacles to be stoppered despite the height of such receptacles, valve means to release pressurized fluid contained in said expansible means after stoppering, and secondary vacuum means to evacuate said expansible means of residual pressurized fluid and thereby maintain equilibrium between said expansible means and the interior of said drying chamber.

13. The combination set forth in claim 12 wherein said equipment unit includes a frame, a condenser means mounted on said frame, said condenser means in communication with said plurality of drying chambers through a plurality of individually valved conduit means connecting each of said drying chambers with said condenser means independently, vacuum means mounted on said frame in communication with said condenser means to withdraw water vapor from each of said drying chambers into said condenser means, refrigeration means mounted on said frame and in communication with said condenser means, heat transfer means mounted on said frame including heat exchanger means containing heat transfer fluid and proportionally controlled means to heat said fluid in valved communication therewith, and continually operating pump means in communication with said exchanger means to circulate said heat transfer fluid through conduit means to each of said drying chambers, and stoppering power means, including fluid pressure means and secondary vacuum means, mounted on said frame.

14. An apparatus particularly adapted for freeze drying various materials comprising in combination, a plurality of individual self-supporting drying chambers, each drying chamber having a lower wall which also forms a shelf to support a number of receptacles containing material to be freeze dried, and an equipment

unit separate from said drying chambers having heat transfer means, vacuum means, unitary condenser means and stoppering power means each mounted thereon, said heat transfer means, said vacuum means and said condenser means in communication with each of said drying chambers independently and operable respectively to control the temperature and to evacuate and condense water vapor from each of said chambers independently, such that said materials placed in each of said chambers may be freeze dried.

15. The combination set forth in claim **14** wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall jointed together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plates acting to directly conduct thermal changes to the receptacles and the materials contained therein within the chamber in resposne to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining said rectangular plates at corresponding end thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

16. The combination set forth in claim **15** wherein said equipment unit includes a frame, a condenser means mounted on said frame, said condenser means in communication with said plurality of drying chambers through a plurality of individually valved conduit means connecting each of said drying chambers with said condenser means independently, vacuum means mounted on said frame in communication with said condenser means to withdraw water vapor from each of said drying chambers into said condenser means, refrigeration means mounted on said frame and in communication with said condenser means, heat transfer means mounted on said frame including heat exchanger means containing heat tranfer fluid and proportionally controlled means to heat said fluid in valved communication therewith, and continually operating pump means in communication with said exchanger means to circulate said heat transfer fluid through conduit means to each of said drying chambers, and stoppering power means, including fluid pressure means and secondary vacuum means, mounted on said frame.

17. The combination set forth in claim **14** additionally including unitary stoppering means removably mountable within each of said drying chambers when stoppering of receptacles is desired, said stoppering means in each chamber being movable, upon operation of said stoppering power means in communication therewith, to stopper the receptacles supported on said shelf in said drying chamber after the material contained in these receptacles has been freeze dried, said stoppering means being supported, during stoppering, by the walls of each drying chamber such that the external forces on each of said drying chamber assist in balancing the internal forces created by said stoppering means.

18. The combination set forth in claim **17** wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said support means having brackets disposed on them on which said stoppering means may be slidably mounted, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plate acting to directly conduct thermal changes to the receptacles and the materials contained therein within said chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining together said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

19. The combination set forth in claim **18** wherein said stoppering means includes a support means and a platen means being maintained in parallel relationship to one another by resilient means disposed between said platen means and said support means and urging said platen means upwardly toward said support means, expansible means inflatable to move said platen means and said support means in vertically opposite directions, said expansible means disposed between said support means and said platen means and stoppering power means, including fluid pressure means mounted on said equipment unit in communication with said expansible means through conduit means, to inflate said expansible means, control means in communication with said fluid pressure means to regulate the admission of pressurized fluid into said expansible means so that a maximum constant stoppering force is always exerted by said platen means on the receptacles to be stoppered despite the height of such receptacles, valve means to release pressurized fluid contained in said expansible means after stoppering, and secondary vacuum means to evacuate said expansible means of residual pressurized fluid and thereby maintain equilibrium between said expansible means and the interior of said drying chamber.

20. The combination set forth in claim **19** wherein said equipment unit includes a frame, a condenser means mounted on said frame, said condenser means in communication with said plurality of drying chambers through a plurality of individually valved conduit means connecting each of said drying chambers with said condenser means independently, vacuum means mounted on said frame in communication with said condenser means to withdraw water vapor from each of said drying chambers into said condenser means, refrigeration means mounted on said frame and in communication with said condenser means, heat transfer means mounted on said frame including heat exchanger means containing heat transfer fluid and proportionally controlled means to heat said fluid in valved communication therewith, and continually operating pump means in communication with said exchanger means to circulate said heat transfer fluid through conduit means to each of said drying chambers, and stoppering power means, including fluid pressure means and secondary vacuum means, mounted on said frame.

21. An apparatus particularly adapted for use as a chamber unit in the freeze drying and stoppering of samples contained in receptacles comprising, in combination, a plurality of individual self-supporting drying chambers, each drying chamber having a lower wall which also forms a shelf to support a number of receptacles containing material to be freeze dried, and a stoppering means removably mounted within each of said drying chambers, said stoppering means in each chamber being movable, to stopper the receptacles supported on said shelf in said chamber after the material contained in these receptacles has been freeze dried, said stoppering means being supported by said lower wall and an upper wall of each of said drying chambers during stoppering such that the internal forces created by the stoppering means are at least partially balanced by the external forces on said chamber walls.

22. The combination set forth in claim **21** wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said support means having brackets disposed on them on which said stoppering means may be slidably mounted, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plate acting to directly conduct thermal changes to the receptacles and the materials contained therein within said chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining together said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

23. The combination set forth in claim **21** wherein said stoppering means includes a support means and a platen means connected and being maintained in parallel relationship to one another by resilient means disposed between said platen means and said support means and urging said platen means upwardly toward said support means and expansible means sandwiched between said support means and said platen means and inflatable to move said platen means and said support means in vertically opposite directions, said stoppering means being easily and quickly mounted in said drying chamber above said lower wall thereof by hanging said support means in said chamber, and said stoppering power means includes fluid pressure means mounted on said equipment unit in communication with said expansible means through conduit means to inflate said expansible means, control means in communication with said fluid pressure means to regulate the admission of pressurized fluid into said expansible means so that a maximum constant stoppering force is always exerted by said platen means on the receptacles to be stoppered despite the height of such receptacles, valve means to release pressurized fluid contained in said expansible means after stoppering, and secondary vacuum means to evacuate said expansible means of residual pressurized fluid and thereby maintain equilibrium between said expansible means and the interior of said drying chamber, said stoppering means being operable by actuation of said stoppering means such that said support means is moved adjacent an upper wall of said drying chamber as said platen means is moved adjacent said receptacles during stoppering to allow the external forces on said drying chamber to assist in balancing the internal forces created by said stoppering means.

24. The combination set forth in claim **23** wherein each drying chamber includes a top wall and a bottom wall, two side walls and a rear wall joined together to form an elongated hollow shell having an opening at its front end to provide access to its interior, said top wall and said bottom wall formed by parallel rectangular plates held in spaced relationship by support means vertically disposed between them, said support means having brackets disposed on them on which said stoppering means may be slidably mounted, said rectangular plate forming said bottom wall also forming a shelf to support the receptacles containing materials to be freeze dried, said rectangular plate acting to directly conduct thermal changes to the receptacles and the materials contained therein within said chamber in response to the temperature of a heat transfer fluid circulated about their outside surfaces, said side walls formed by cylindrically-shaped members joining together said rectangular plates at corresponding ends thereof, said rear wall of said drying chamber fixedly attached to said top wall, said bottom wall and said side walls and having an opening formed in it to provide communication to said condenser means mounted on said equipment unit, said door mounted in hinged relationship to said front end of said chamber being movable over the front end of said chamber to close and hermetically seal the interior of said drying chamber from the atmosphere.

25. An equipment containing unit particularly adapted for use in freeze drying various materials including a frame, a condenser mounted on said frame, said condenser in communication with a plurality of drying chambers through a plurality of individually valved conduit means connecting each of said drying chambers with said condenser independently, vacuum means mounted on said frame in communication with said condenser to withdraw water vapor from each of said drying chambers into said condenser, refrigeration means mounted on said frame and in communication with said condenser, heat transfer means mounted on said frame including heat exchanger means containing heat transfer fluid and proportionally controlled means to heat said fluid in valved communication therewith, and continually operating pump means in communication with said exchanger means to circulate said heat transfer fluid through conduit means to each of said drying chambers, and stoppering power means, including fluid pressure means and secondary vacuum means, mounted on said frame.

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