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Gottfried

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[54] **CONTINUOUS FREEZE DRYER**

[76] Inventor: **Herbert Gottfried,** 3441 5th St., Ocean Side Long Island, N.Y. 11572

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[52] **U.S. Cl.** 34/5, 34/92
[51] **Int. Cl.** F26b 5/06
[58] **Field of Search** 34/5, 192

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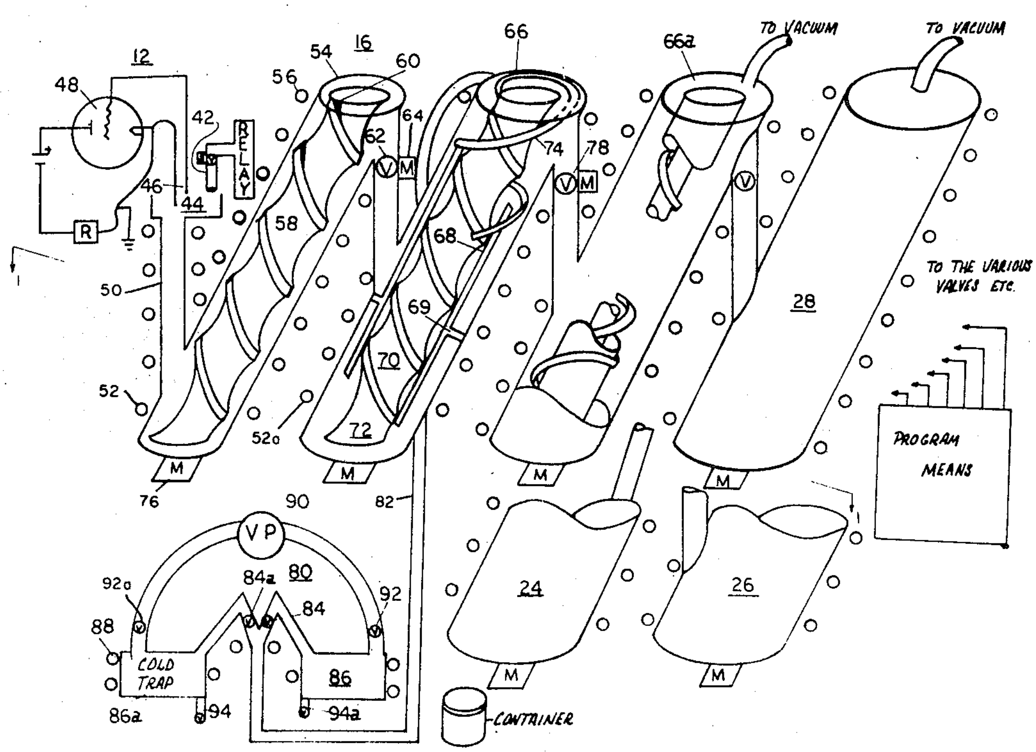
Primary Examiner—William F. O'Dea
Assistant Examiner—William C. Anderson
Attorney—George B. Oujevolk

[57] **ABSTRACT**

Arrangement for continuously and automatically lyophilizing material comprising freezing the material, pulverizing the frozen material, circulating the material along a screw conveyor while exhausting the ambient gas to heat the material and expose particles of material to heat, regulating the heat supplied by reversible refrigeration, alternately exhausting said ambient gas in first and second cold traps, sequentially finishing partially treated material in at least first and second finishing zones and discharging the material into containers without breaking the vacuum.

12 Claims, 9 Drawing Figures



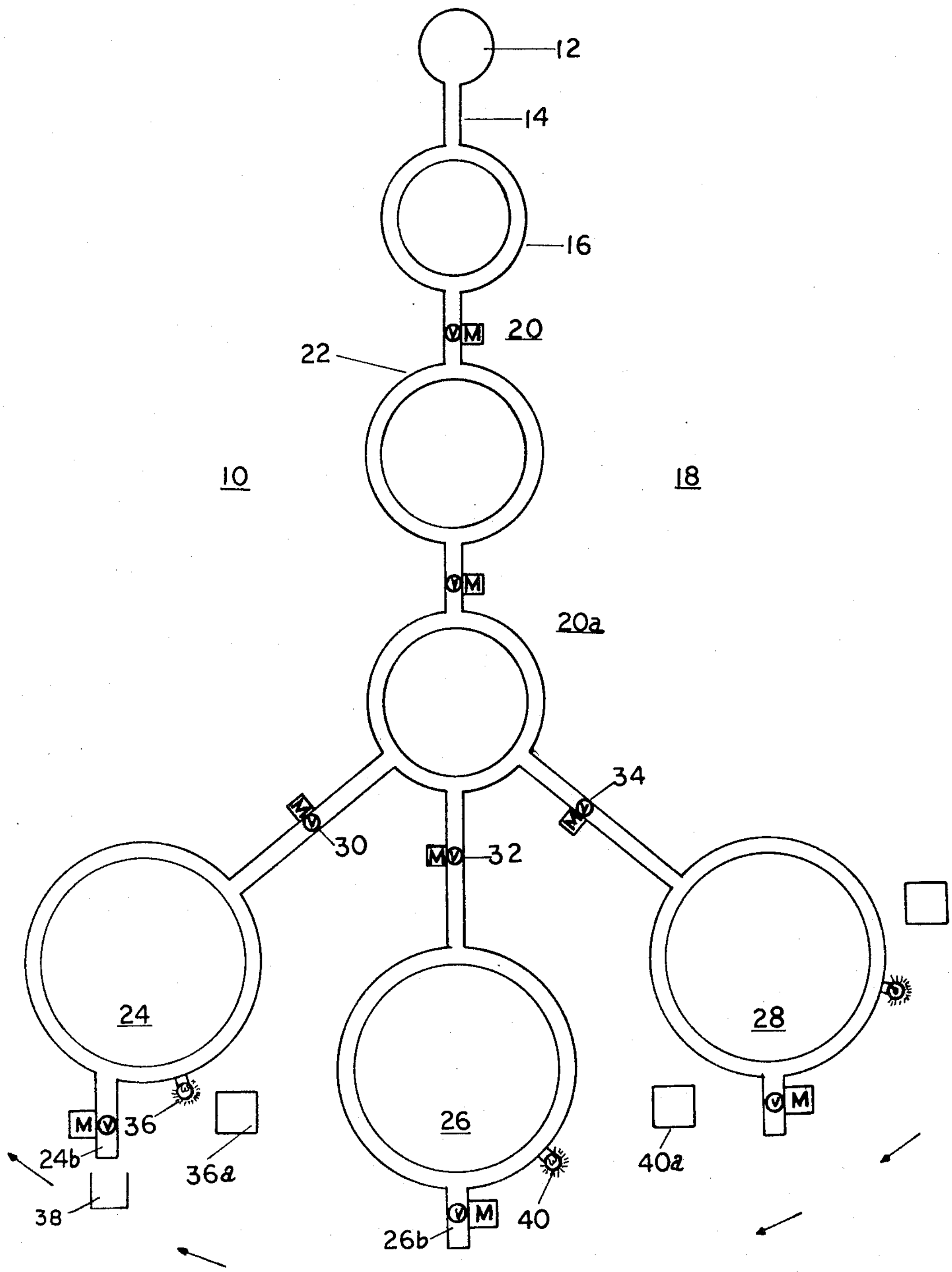


FIG. 1

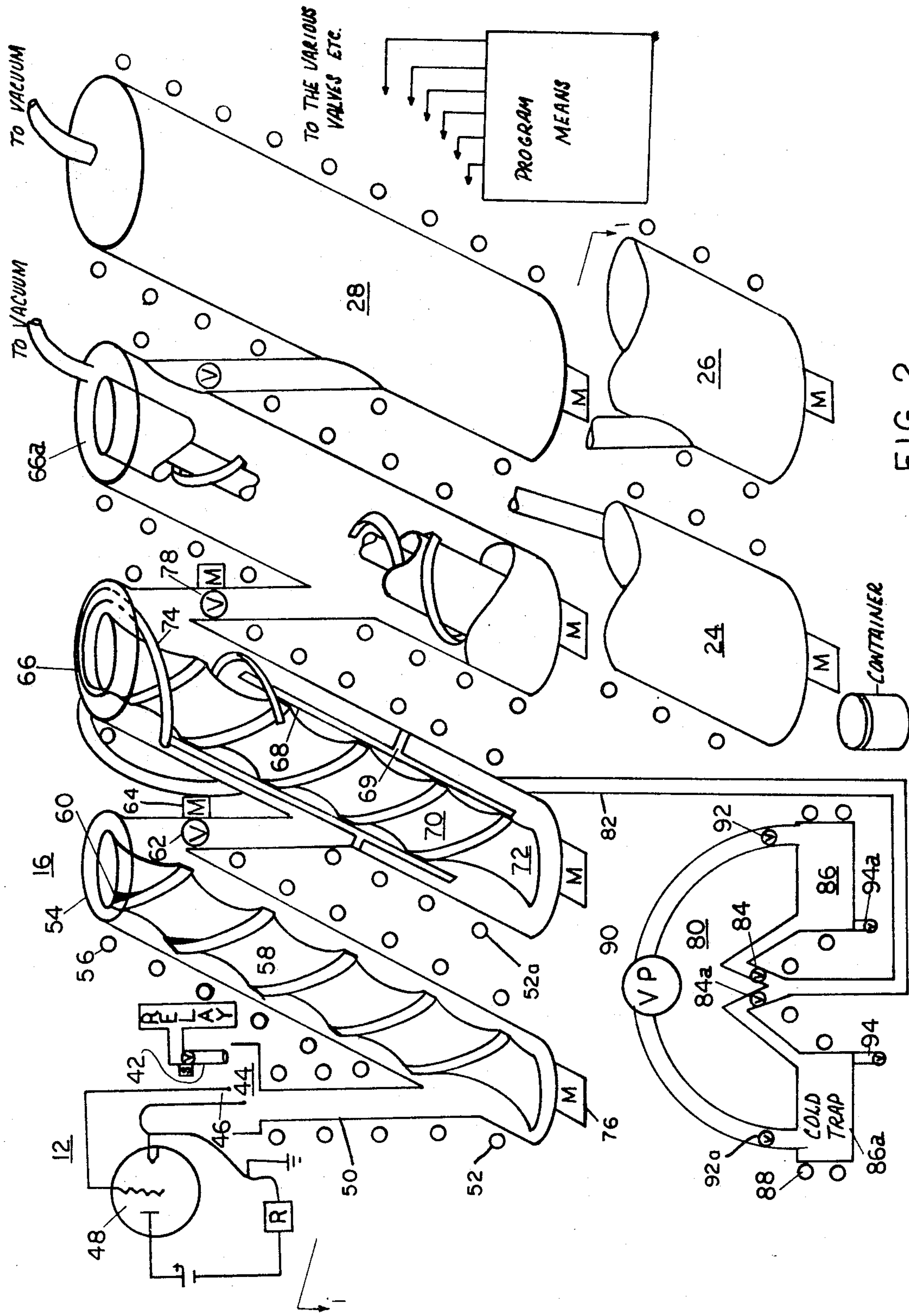

TO VACUUM
TO VACUUM
TO THE VARIOUS VALVES ETC.
PROGRAM MEANS
RELAY
CONTAINER
COLD TRAP
VP

FIG. 2

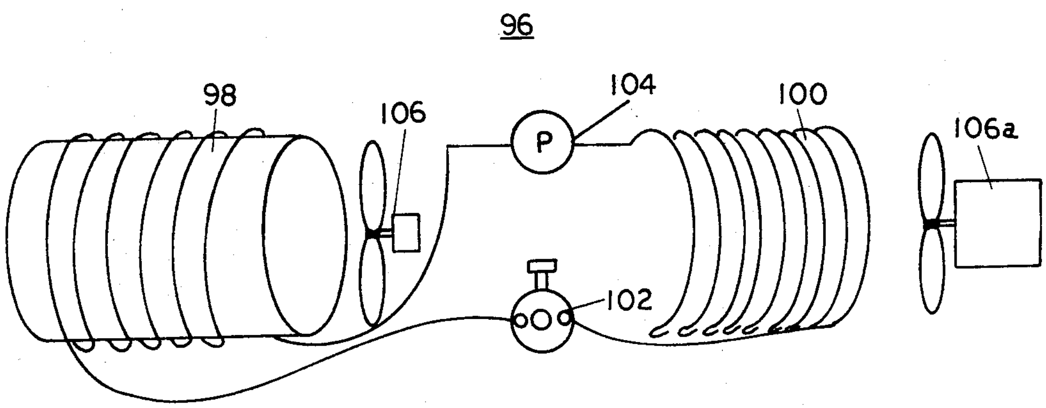


FIG. 2a

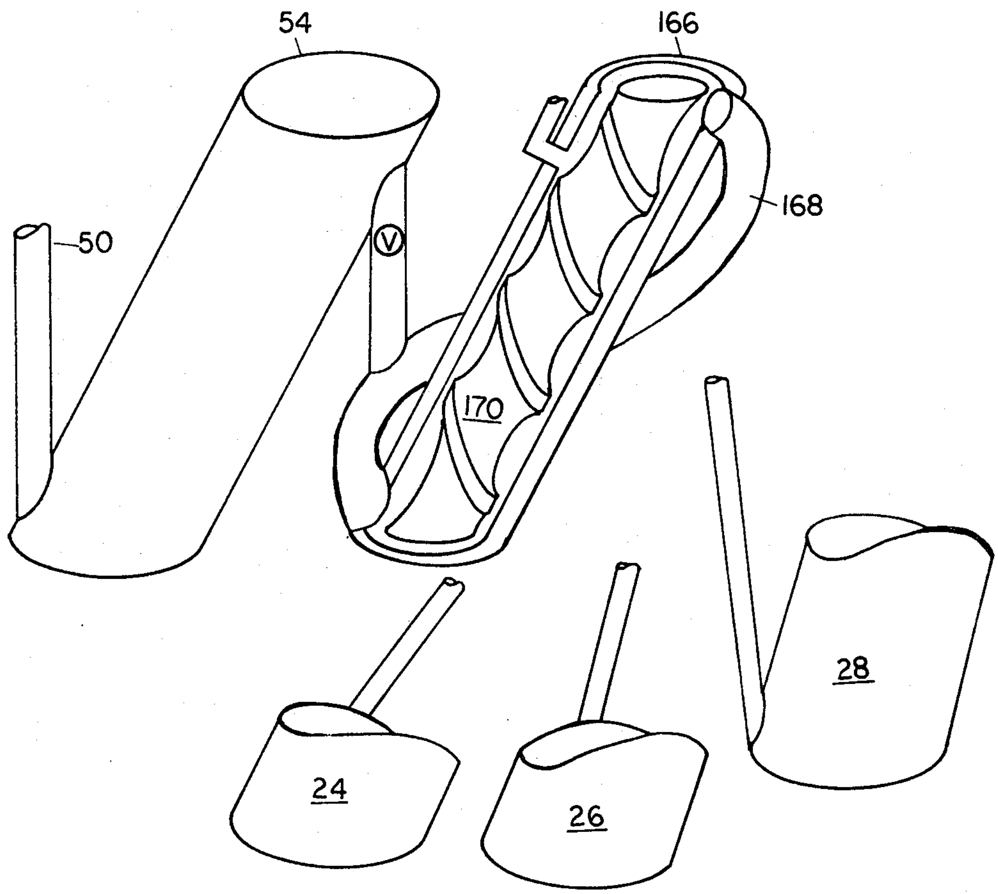


FIG. 3

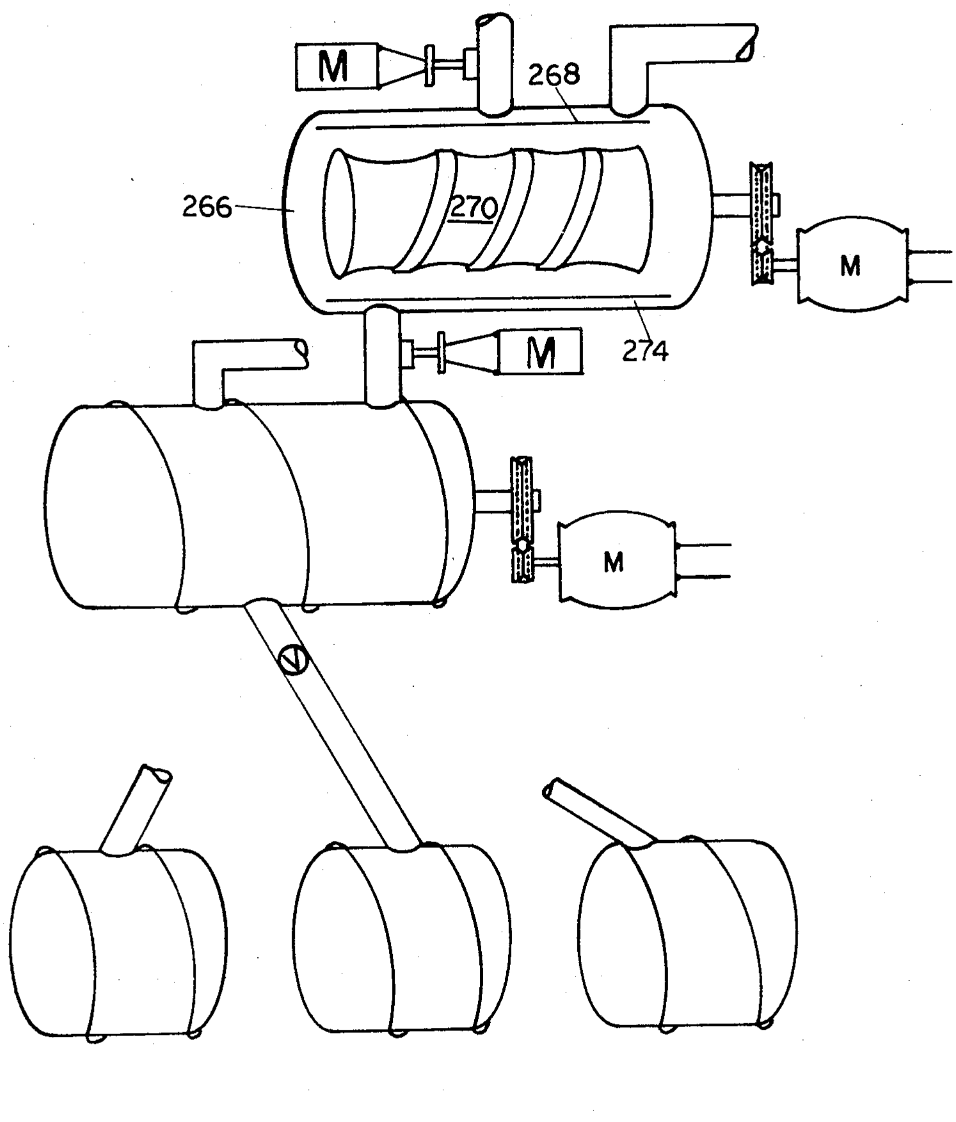


FIG. 4

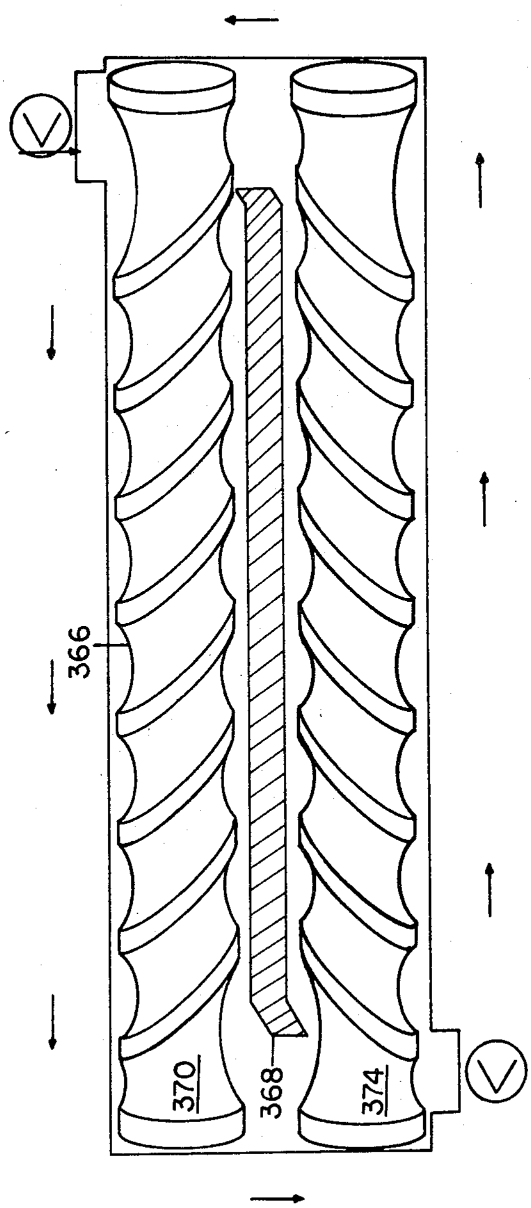
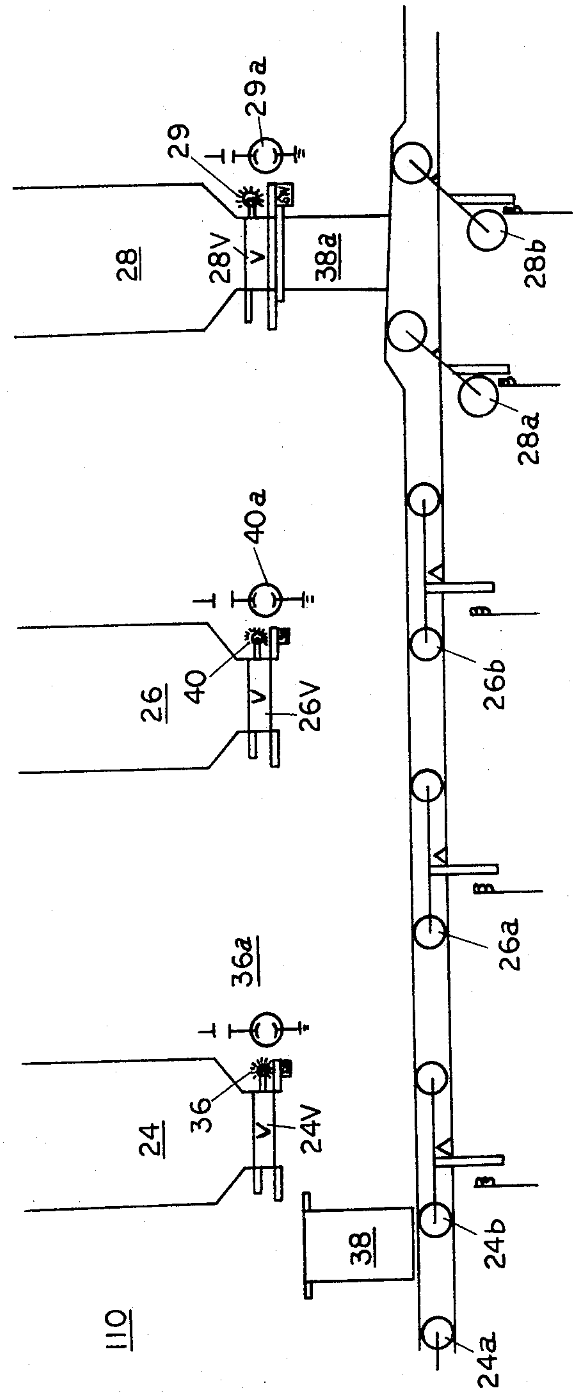


FIG. 4a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28
28V
38a
28b
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40a
40
26
26V
26b
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36a
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24
24V
24b
38
110
24a

FIG. 5

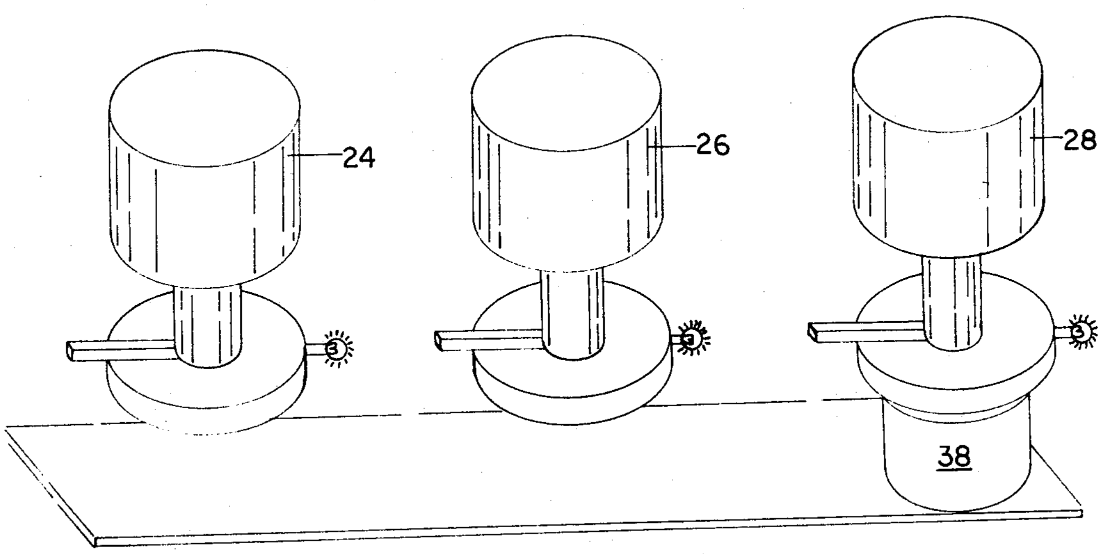
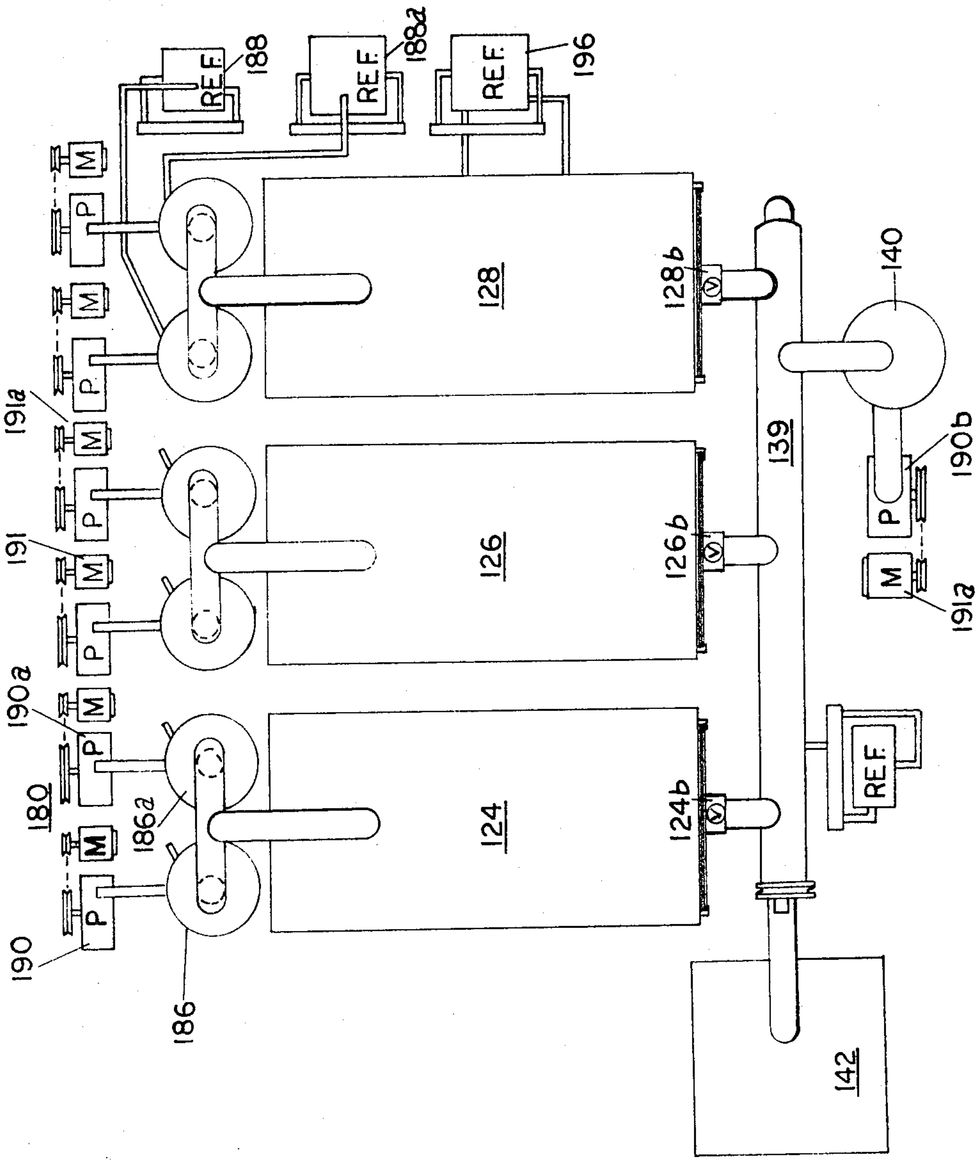


FIG 5a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128b
126b
124b
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190b
191a
REF.
142

FIG. 6

CONTINUOUS FREEZE DRYER

BACKGROUND OF THE INVENTION

The present invention relates to a system for drying substances which deteriorate when subjected to elevated temperatures; and more specifically, it concerns the process called freeze-drying, i.e., lyophilization.

BRIEF DESCRIPTION OF THE ART OF FREEZE DRYING

In a freeze drying process, material to be dried is subjected to a low temperature so that the water present in the material goes over to the solid state forming ice crystals. Ice, even though at low temperatures, has a substantial vapor pressure. If the pressure above the ice is lowered below this vapor pressure, then the ice will evaporate.

Freeze drying, as generally practiced, is a batch process. The frozen material is placed in pans or other containers, in a vacuum chamber where freeze drying proceeds. After evaporation of the water, the material is removed from the vacuum chamber for further processing. This is a slow process requiring a long period of time for complete drying. For example, fruit juices such as orange and lemon juice require several days to produce substantial amounts of dried material. In these cases, the residue, if not completely dry, is hygroscopic and will readily pick up moisture.

Material which is not completely dry will not age well, spoilage occurring even when kept in the refrigerator. It is essential that the water be removed so that the vapor pressure above the material is of the order of 25 microns or less at ambient temperatures before the material can be considered completely dry. Thus with present instrumentation, while one may fairly rapidly evaporate 80–90 percent of the water present, the last 10–20 percent takes long periods of time. Further, present instruments do not permit ready automation of the system.

OBJECTS OF THE INVENTION

It is therefore an object of this invention to provide an instrument for continuous freeze drying which starts from the raw material such as foods (e.g., fruit juices, meats, vegetables), and biological materials, which are heat sensitive, and automatically and continuously prepare a dried material, stable for extended periods of time.

Another object of the present invention is to provide an instrument for dehydrating materials more rapidly than by present day conventional processes, without lessening the quality or stability of the finished product.

Still another object of the present invention is to provide an instrument for preparing a product with an extremely low water content so that the material, in most cases, can be stored at ambient temperatures in sealed containers.

Yet another object of the present invention is to provide an instrument for preparing freeze dried materials without attendance being required by an operator, except to supervise the loading and unloading of the instrument.

REQUIREMENTS OF A FREEZE DRYING SYSTEM

A freeze drying system therefore requires:

A. A means for cooling the material to the frozen state.

B. A means for creating a vacuum, usually a vacuum pump.

An additional requirement is that the water evaporated does not enter the vacuum pump in substantial quantities so as to condense there. For this reason a cold trap at a lower temperature than that to which the material to be dried has been cooled, is needed. This requires a third component for the system, namely,

C. A cold trap.

In addition, as freeze drying proceeds it is required to supply heat to the material to be dried, at a controlled rate, so that the heat of evaporation is supplied, but the freezing point is not exceeded. This usually comprises exposing the outside of the container, where the material being dried is located, to a ambient temperature. In some cases, heated coils or infra-red rays are employed to supply this heat. A fourth component is then required.

D. Means for supplying heat to the material being freeze dried without raising the temperature above freezing.

SUMMARY OF THE INVENTION

Generally speaking, the object of the invention is to provide means for exposing, continuously, each and every particle of triturated frozen material to a source of controlled heat, in a vacuum, so that all moisture in the material can be removed, and to perform this function in a completely automatic manner. Thus, in the apparatus herein contemplated, material to be processed is first frozen and ground. It is then introduced under vacuum to a treating chamber. Within this treating chamber are agitating means, e.g., screw conveyor means for moving the material longitudinally across said chamber to one end thereof, during this process exposing all particles of the material to the blades of said conveyor and walls of the container. A return path for the material is provided to guide the material back from the one end to the other end of the chamber. Reversible refrigeration means are provided to cool or heat the chamber walls so as to maintain the temperature within a predetermined range. A vacuum pump and cold trap arrangement constantly evacuates gas formed by moisture in the chamber and, vacuum discharge means are provided to discharge the treated material from the chambers into packing containers.

The invention, as well as other objects and advantages thereof will become more apparent from the following detailed description when taken together with the accompanying drawings in which:

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a type of block diagram representation of the inventive concept when viewed along line 1 — 1 of FIG. 2, which cuts across the principal components of the instrument herein described;

FIG. 2 shows a partly schematic, partly perspective explanation of the instrument contemplated herein with parts broken away and in section to describe the operation of the components;

FIG. 2*a* illustrates the principles of a reversible refrigerator arrangement which is useful herein;

FIG. 3 presents a modification of the inventive concept described in FIG. 2 in a view somewhat similar to FIG. 2;

FIG. 4 again presents in perspective and schematic form another version of the inventive concept; while,

FIG. 4a illustrates a further modification of the concept shown in FIG. 4;

FIG. 5 depicts schematically the packaging of the product;

FIG. 5a is a perspective explanation relating to the vacuum packaging of the product; and

FIG. 6 illustrates another arrangement for packaging the product.

DETAILED DESCRIPTION

THE OVERALL APPARATUS

According to the present inventive concept as illustrated in FIG. 1, raw material to be processed in the apparatus **10**, such as citrus juices, meats, vegetables or biological materials are first introduced into an input zone **12** where the material is maintained at a constant level. From the input zone **12**, the raw material is cooled in a pipe **14** and goes to a freezing zone **16**. In the freezing zone **16**, the raw material is caused to travel across an elongated chamber by a screw conveyor. The travel path is surrounded by cooling coils and the screw conveyor terminates in a crushing area having specially made convolutions to crush and pulverize the raw material. The raw material is then transferred to a lyophilization zone **18** across a valve. In this lyophilization zone, the raw material is kept in constant movement while the zone is exhausted of moisture by means of a vacuum pump. This zone may consist of one or two chambers. If a two stage arrangement **20, 20a** is used, as shown in FIG. 1, about 50 percent of the moisture is removed in the first chamber while up to 90 percent of the moisture is removed in the second chamber. The lyophilization zone **18** is surrounded by the coils **22** of a reversible refrigerator. Heretofore, one of the problems in connection with freeze drying was the fact that only the outer layer of material was treated whereas the moisture in the middle of a lump of material was insulated from the heat source so that the moisture was retained. However, according to the present inventive concept the material is constantly moved, turned and agitated so that lumps are not formed and each and every particle of material is acted on by the heating coils **22** and the vacuum suction means. Generally, the material will remain in the two chambers of the lyophilization zone for upwards of several hours. During this time, about ninety percent of the moisture will be removed. The treated material is then transferred sequentially to one of three chambers of the finishing zone **24, 26, 28**, across motor operated valves **30, 32, 34**. The chambers in this finishing zone are similar to those in the lyophilization zone and also cooled or heated by reversible refrigerator coils. However, in the primary chamber **24** of the second zone the input material is already 90 percent moisture free. After the first batch of material from the lyophilization zone enters the primary chamber **24**, the lyophilization zone **18** will have an empty chamber which is then refilled with material from the freezing zone **16** so that the two chambers of the lyophilization zone **18** remain in operation, as well as the primary chamber of the finishing zone. When the lyophilization zone **18** has again removed about 90 percent of the moisture in the second chamber thereof, the material is again transferred, but this time across valve **32** to the secondary chamber **26** of the finishing zone. At this time, both primary chamber **24** and secondary chamber **26** are processing material. The two chambers of the lyophilization zone **18** are again kept filled, and again 90 percent of the moisture is removed from the material and this time the treated material is sent across valve **34** into last chamber **28**. Thus, after a while, the two chambers of the lyophilization zone **18**, as well as the three chambers of the finishing zone are processing material, the processing time in the lyophilization zone being about one-third the time required for the processing in any of the three chambers **24, 26, 28** of the finishing zone.

After the material in chamber **24** is completely dried, the temperature will tend to rise. When it reaches ambient temperature, the signal light **36** from chamber **24** will light up. This will act on photocell **36a** causing a container **38** to stop under valve **24b**. When the secondary chamber **26** is ready, light **40** of the secondary chamber will light up to act on photocell **40a**. This will cause the next container to stop under valve **26b** of secondary chamber **26**. The same thing takes place with the last chamber and the process is then repeated.

It will be noted that at all times a finishing chamber remains ready to accept the load from the lyophilization zone. Thus, the total process is continuous and automatic.

In the description of the invention which follows, three different embodiments are described in connection with the lyophilization and constant movement zones. For ease of presentation, the most complicated embodiment is described first. However, the other embodiments are equally important and indeed, the simplest embodiment is in many cases more advantageous, since it has less moving parts and consequently, is less apt to require repairs.

THE INPUT ZONE AND FREEZING AND CRUSHING ZONE

The input zone **12** is fed by a spigot **42** into a liquid container **44** maintained at a constant level. This is accomplished by first and second electrode **46**, one of which is located within and the other without the liquid. One electrode is tied to the grid and the other to the cathode of a vacuum tube **48**. Thus, if the liquid level does not touch the grid electrode, current flows from the cathode to the plate. When the liquid slurry level is such as to touch the grid electrode, grid current flows, a relay is enabled and the liquid level is shut off. Liquid slurry in container **44** drops along pipe **50** to a work unit. Pipe **50** is surrounded by cooling coils **52** designed to keep the slurry cool, the coils being at about 0° to about 5°C. This work unit is the freezing zone **16** and consists of an elongated cylindrical chamber **54**, disposed at about a 30° inclination from the vertical, also surrounded by cooling coils **56**. The temperature of these cooling coils is about −40°C. This causes the material to freeze to a solid state. In the center of cylindrical chamber **54** is a screw conveyor **58** disposed at an angle to the vertical. Thus, pipe **50** conveys the raw material to the top of the chamber. In the screw conveyor **58**, the last few upper convolutions **60** have a special cutting configuration to crush the icy material.

The material moving along screw conveyor **58** is to be transferred to the lyophilization zone **18**. However, after the transfer, while the material is being processed in the lyophilization zone **18**, the screw conveyor **58** of the freezing zone **16** does not stop turning. Indeed, if it were to stop, the material on the screw conveyor **58** would lump together and jam the conveyor. Thus, when not transferring material across valve **20**, the screw conveyor turns slowly at an idle speed of about 1 r.p.m. to keep the material from freezing solid. In this state, the contents merely slide around and does not advance.

From the foregoing, it can be seen that the liquid to be frozen and lyophilized arrives from a storage chamber through a spigot **44**. The opening of the spigot is controlled by a solenoid valve so that a constant level is maintained in one leg of what comprises a V tube. One leg of the V tube is a pipe leading from the constant leveling device to the second leg of the tube which comprises a combination driving screw conveyor and crushing heads, formed in several of the outer convolutions of the screw conveyor.

The constant level is maintained by two platinum wires, one of which dips into the liquid and is grounded and attached to the cathode of an electronic tube. The second wire is set at the level desired. This second wire is attached to the grid of the electronic tube. The electronic tube acts as an electronic switch to close a relay when the level of the liquid reaches the second wire, closing the circuit. A transistor circuit can be used in a similar fashion.

The closing of the relay activates the solenoid valve so that the liquid stops flowing into the V tube. This valve then opens when the liquid level drops. In this way a constant level is maintained.

The liquid travels down the V tube until it reaches the leg of the V where the start of the screw conveyor is located. This screw conveyor is driven by a motor arrangement **62**. It serves to lift the liquid while it is being cooled by the cooling coils.

As the liquid first descends and then ascends the V, it is cooled and eventually freezes. The stainless steel lifting screw conveyor serves to break up the ice into chunks as it is formed. These chunks are lifted into the grinding and flaking area. In this area, the convolutions of the screw take on the form of sharp pointed wedges or cutting blades. These flake and grind the ice so that when it reaches the exit zone of the V tube the material is in the form of a coarse ground powder.

FIRST LYOPHILIZATION ZONE

The material now enters the first chamber **66** of the lyophilizing zone **18** through a ball valve **62** with a 4 inch opening which is opened and closed by a motor **64**. The crushed ice passes through the ball valve and enters first lyophilizing chamber **66** having cooling coils **52***a*. After the lifting screw conveyor has turned a preset number of times, the first lyophilizing chamber **66** contains an amount of frozen material, approximately equivalent to one half its full capacity. At this point motor **64** is activated to close the ball valve **62**.

Thus, lyophilization zone **18** comprises an elongated cylindrical chamber **66** also inclined from the vertical at about 30° and held in this chamber **66** is a cylindrical partition **68** which is fastened to the chamber wall by spokes **69**. Within the cylindrical partition **68** is a central screw conveyor **70**. Central screw conveyor **70** has a central shaft **72**. The material treated travels up the central screw conveyor and over the cylindrical partition **68**. Disposed around the cylindrical partition **68** is an outer screw conveyor **74** which will push the material downwards along the partition wall. This outer screw conveyor **74** does not extend fully along the entire length of the chamber. There are at most one or two convolutions. The central partition **68** does not extend the full length of the chamber so that the wall does not prevent the first and second outer convolutions of the outer screw spiral **74** from passing from the central shaft **72** over the wall. The outer screw conveyor **74** extends only partly along the outer wall since it is only connected to the central shaft at one extremity. As the chamber **66** is substantially vertical, material must be pushed upwards by the inner screw conveyor **70** until past the partition wall. The material will tumble in the space between the partition outer side and the chamber inner wall and will travel back downwards to the bottom of the chamber along the partition wall.

In addition to the continuous movement just described by the screw conveyor arrangement, the first and second chambers of the lyophilization zone have two other features namely: a reversible refrigeration arrangement, and, a vacuum pump cold trap arrangement. Both of these will be described in greater detail herein.

Thus, as material is transferred from the freezing and crushing zone **16** to the first lyophilization zone **18** across valve **62***a*, a vacuum pump arrangement **80** is started so as to form a vacuum in first chamber **66**. At this time the chamber **66** is sealed.

When the crushed ice reaches the first lyophilizing zone **18**, it is only a few degrees below the freezing point. The application of the vacuum causes first a rapid evaporation process to proceed. This cools the ice further to temperatures below −10°C. The ice becomes "harder" as traces of liquid are frozen. During this process, the rate of evaporation of the ice is so rapid as to cool the ice rapidly. Contact with the rotating double screw conveyor supplies the heat required for the heat of vaporization.

As the screw conveyor rotates in the chamber, the mechanical energy is converted to heat. It is well known that mechanical energy generates heat. Thus, the rate at which heat is supplied is determined by the rate at which the rotor rotates. In addition cooling coils add or subtract heat and thus maintain a constant temperature at a constant speed of rotation.

When a balance has been reached between the cooling process due to evaporation, and the heating process due to rotation, so that evaporation is taking place without melting of the ice, and with the temperature remaining constant, the lyophilizing conditions or process conditions have then been established.

A motor **76** drives the screw conveyor in the vacuum chamber. This motor **76** is connected to the shaft of the rotor which extends out of the chamber through a vacuum seal similar to the one described in U.S. Pat.

Nos. 2,419,074 and No. 2,454,340, or some other similar vacuum seal.

After permitting the lyophilization process to be established at ambient room temperature, the temperature is slowly raised in the lyophilization chamber by means of the coils from the reversible refrigeration means, so that the wall temperature reaches 30° to 70°C, depending upon the lyophilization rate and stability of the material. It must be pointed out that while the wall temperature is above room temperature, the temperature of the material remains well below freezing because of the rapid evaporation process.

Treatment in the chamber continues until the gas pressure in the chamber reaches 200 microns or less at 50°C. This usually takes approximately two hours, the exact time depending on the material, the capacity of the chamber, the operation of the vacuum pump and cold trap subsequently described herein.

Thus, in the lyophilization chamber **66**, the material is circulated forward, up, back down, and forward again. In addition, the material is being lifted and circulated against the walls of the chamber in a spiral fashion. This results in intimate contact of the particles of material with the screw conveyor and container walls permitting efficient access of the heat to the material to be lyophilized. This solves a major problem with present systems.

It is to be noted that the problem of getting heat to each particle is solved in this invention by bringing the heat to each particle by the mechanical motion of a mixing and circulating device. This produces a readily controlled temperature at the surface of each particle.

In the present invention, a central rod supports a first screw conveyor and, attached to the first screw conveyor is a second screw conveyor so that both move together. Rigidly attached to the wall of the container is a stainless steel cylindrical partition containing the first screw conveyor, while the second screw conveyor, which does not completely extend across the entire chamber, and indeed, may have only one or two convolutions, goes around the cylindrical partition. The inner screw conveyor drives the material forward and, when it reaches the end of its travel, it piles up and is soon picked up by the outer screw conveyor which moves it in the opposite direction. In this way, the material circulates from front to back. Since the outer and inner screw conveyors also lift material on each rotation, the material describes a spiral motion forward and the material comes repeatedly in contact with the screw blades and the walls of the container.

The center stainless steel separation may also be omitted. In this case the inner and outer screw conveyors, running counter to each other, are both continuous and rigidly attached. In this case the material pushed by the outer spiral packs and forms a wall so that the material being pushed in the inner spiral moves counter to this wall of material.

While the ball valve **62** is closed, operation of the screw conveyor in the freezing zone **16** is in an idling position, rotating only slowly at 1 r.p.m., or less to prevent impaction of the ice, but not fast enough to lift it. At the same time no new liquid is coming in because the spigot **22** is closed. This permits the lyophilization chamber to cause circulation of the material until a vacuum of less than 200 microns is reached. This low pressure starts a timer switch. Lyophilization and circulation is continued for about an additional 1 hour. At this point valve **78** opens.

Communication then exists between the first and second lyophilization chambers **66** and **66*a***. The second lyophilization chamber is at less than 100 microns, so that the vacuum is not broken. All the material in the first lyophilization chamber **66** now empties into the second lyophilization chamber **66*a***. This chamber is almost identical to the first chamber and need not be described in detail. In fact, for some applications this second chamber of the lyophilization may be omitted.

After about 50 percent of the moisture has been removed in the first chamber, the material is transferred to the second chamber kept under a higher vacuum. Here, the process proceeds as in the first chamber, except that upwards of 90 percent of the moisture is then removed.

Up to this point, two features of the first lyophilization and constant movement zone have not as yet been described. These features are the reversible refrigeration, and the vacuum pump and cold trap.

THE COLD TRAP AND VACUUM PUMP ARRANGEMENT

The cold trap and vacuum pump arrangement **80** is connected to the chamber **66** of the first zone by an exhaust pipe **82**. Pipe **82** splits with first and second branches each with a valve **84**, **84*a***. Each branch has a cold trap **86**, **86*a*** surrounded by reversible refrigeration coils **88**, **88*a***. Each cold trap **86**, **86*a*** is in turn connected to a central pump **90** across valves **92**, **92*a***. Valves **92**, **92*a*** operate together with valves **84**, **84*a***. Thus, in effect, there is an exhaust line from the chamber **66** to the vacuum pump **90** alternately across cold trap **86** and **86*a***. The cold trap is nothing more than a hollow vessel with input and exhaust openings at the top of the vessel, surrounded by reversible refrigeration coils **88**, **88*a***, and drains with valves **94**, **94*a***.

During operation, the vacuum pump continues to operate uninterrupted. At first valve **92*a*** and valve **84*a*** are closed. Valves **92** and **84** are open. Condensation of ice takes place in cold trap **86**. Cold trap **86** is of such magnitude that it can take all of the condensate from one charge of the lyophilizing chamber. When the lyophilizing chamber **66** begins to unload, ball valves **92** and **84** close. Drain valve **94** opens. Cooling coils around cold trap **86** now become heating coils due to reversal of the action of the reversible refrigeration. The ice begins to melt and the water goes down a drain across drain valve **94**. In the meantime, valves **92*a*** and **84*a*** are opened, and drain valve **94*a*** has closed. The coils around the second cold trap **86*a*** become cooling coils. Lyophilization of the next charge in the lyophilizing chamber results in condensation in the second cold trap **86*a***. When this trap is full the cycle is repeated, the first cold trap **86** becoming cold, while the second cold trap **86*a*** is defrosted.

THE REVERSIBLE REFRIGERATING ARRANGEMENT

A reversible refrigerating unit **96** has two sets of coils **98**, **100** separated by a narrow orifice **102** with a pump

104 to compress the gas contained in the system. As the gas is compressed in coil 100 preceding the narrow orifice 102, its temperature is elevated. As the gas expands into a second coil 98 on the other side of the orifice 102, it cools. Thus, there is a cool set of coils 98 and a hot set of coils 100, and a pump 104 maintaining the differential in pressure.

The hot set of coils 100 is cooled by outside means (air or water), shown as a fan 106, and thus, the cold set of coils gets colder in each pass so that soon it can be used for freezing materials.

Now the pump may be reversed. In which case, the function of the two sets of coils is reversed. The hot set of coils now becomes the cold and the cold set of coils becomes hot. Normally, a fan 106, 106*a* serves to cool the hot coils. When the inlets and outlets of the compressor are reversed, the coils around the container contain the gas under pressure. These then become the hot coils. By alternately reversing the compressor direction, a constant temperature can be readily maintained in a chamber from −80°C to +75°C, that is from very cold to hot. This is the range which is used in the various chambers of the present invention. To enhance the heating and cooling, coils can be passed through the screw conveyor.

OPERATION OF THE LYOPHILIZATION PROCESS

Thus, to briefly review the operation of the apparatus hereinbefore described, it can be stated that material to be lyophilized needs first to be frozen and then pulverized. This is done in the freezing and crushing zone 16. Two general methods may be used. The preferred procedure is to cool the material to the solid state by refrigeration and pulverize it. As the material travels along a screw conveyor, an alternative procedure is to drop the material, one drop at a time, or particle by particle into liquid nitrogen. The frozen pellets so formed are then pulverized.

The material is transferred by means of the screw conveyor to a lyophilizing zone 18 of constant movement. Here the material is subject to high vacuum. A reversible refrigeration unit herein described, maintains the wall temperature at 25° to 75°C. This requires heat and the reversible refrigeration supplies this heat by being operated in a reverse manner. The reason for this is that the lyophilization process removes the heat of vaporization from the material. While the temperature on the walls of the container is above ambient temperature, the temperature of the ice crystals in the container is at −20°C, or less. An electric heating coil is not satisfactory at this stage since in the event that the temperature becomes too high, the reversible refrigeration needs to be reversed to cool the chamber 66. Thus, a cooling and heating system is needed at the lyophilization stage. The agitation and movement of the material also generates heat and this is the main source of heat utilized in the process. This also may supply excessive heat under certain circumstances and then the reversible refrigerator needs to act as a coolant.

The lyophilizing zone 18 communicates with a vacuum pump to create the vacuum. Interposed between the vacuum pump and the lyophilizing zone is a condensing zone consisting of two cold traps used alternately where the evaporated water is condensed.

Here the temperature is maintained at from −40°C to −80°C in order to ascertain that the vapor pressure of the ice in the condensing zone is lower than that in the lyophilizing and constant movement zone.

It is apparent that after a period of operation, the cold trap in the condensing zone will become full of ice and require defrosting. For this reason the condensing zone consists of two cold traps, each connecting to the pump and to the lyophilizing zone, through ball valves and each supplied with a separate reversible refrigerator unit.

The first cold trap is defrosted by the reversible refrigeration raising the temperature to above freezing to melt the ice therein. A drain, supplied in the cold trap, opens and the water runs down the drain. When all the water has drained out, the drain valve closes and the refrigerator reverses to cool the cold trap to −40°C to −80°C. The first cold trap is now ready to accept a second load of ice after the second cold trap has become full. The process is then repeated with the second cold trap. In this manner, in a continuous automatic fashion the condensate is frozen and removed.

During the initial operation, the chamber being loaded is at ambient pressure, while the other chambers in the system are separated from this chamber by ball valves, vacuum being maintained in these other chambers. When the first chamber has been fully loaded with ice, it is sealed off from the atmosphere and evacuated. Loading from one chamber to the next does not break the vacuum in this manner, when ball valves between the chambers are opened.

The lyophilization zone 18 is preferably a complex of two or more similar chambers 66, 66*a*. In the first chamber, vacuum is applied until a pressure of less than 200 microns is reached. This signifies that lyophilization is in progress. Establishment of lyophilization means that the rate of evaporation is sufficient to maintain the material in a frozen state, without outside cooling. This also means that the ice is usually at least 10°C below the freezing point and is frozen hard. The function then of the first chamber is to establish firmly the conditions of lyophilization.

The material is now transferred to the second chamber where the vacuum is maintained at below 100 microns until approximately 90 percent of the moisture has been removed. Thus, after treatment in the first lyophilization chamber 66, and after about 50 percent of the moisture has been removed, ball valve 78 which is also motor driven, opens and the material in chamber 66 is transferred to chamber 66*a*. Valve 78 then closes and valve 64 opens admitting air and the material from the grinding and flaking area into the first lyophilizating chamber. Valve 64 closes, the vacuum pump is activated, and motor 62 goes to idling speed. The cycle for the first lyophilizating chamber is now repeated.

The second lyophilizating chamber is constructed in a manner similar to the first lyophilizating chamber. The volume of the powder is markedly reduced in the first lyophilizating chamber since approximately two-thirds of the water has been removed. For this reason, the second lyophilization chamber may be smaller for many applications. As an alternative, if of the same size, the second lyophilization chamber may take several successive loads from the first lyophilization chamber.

In the second lyophilization chamber, lyophilization continues until the pressure is brought down to less than 75 microns at temperatures ranging from 30° to 70°C, depending upon the stability of the material.

Second lyophilization chamber **66a** communicates with three finishing chambers. These are called finishing chambers because it is in this area that the material is finally dried and ground before being packaged. The structure of the finishing chambers is similar to first and second lyophilizating chambers.

THE FINISHING ZONE

When the material in the second lyophilizating chamber of the lyophilization zone has reached a vapor pressure of less than 75 microns at 30° to 70°C it usually indicates that its moisture content has been lowered to less than 10 percent. More time is required to remove the last traces of moisture than is required to reduce the moisture content in the first two lyophilization chambers. The contents of the second lyophilization chamber are therefore transferred to a finishing primary chamber **24** under vacuum. As described before, chamber **24** is one of three finishing chambers. When the product in the second lyophilization chamber is again ready for transfer, this is then transferred to the finishing secondary chamber **26**. When a third charge is ready from the second lyophilization chamber **66b**, this is transferred to last finishing chamber **28**. The finishing chambers are maintained at temperatures ranging from 4° to 40°C. The reason for using the lower temperatures is because of the fact that as the material dries, its temperature rises to wall temperatures. For certain biological products, chambers **24, 26, 28** are maintained below 10°C. For fruit juices the temperature may be elevated to 40°C. In any case the wall temperature of chambers **24, 26** and **28** are lower than that of the chambers in the first zone where ice is present at all times.

The outer screw conveyors of the finishing chambers have a few turns which are wedge-shaped so as to pulverize the product, since some dried powders obtained from materials such as orange juice, tend to cake and form a compact mass as they approach dryness. This prevents the complete drying of the material within the cake. For this reason the material needs to be powdered.

After loading of finishing primary chamber **24**, chamber **66**a receives a charge from chamber **66** and chamber **66** is loaded from chamber **54**. During this process the last chamber **28**, is unloaded. In this case a ball valve opens and the contents of chamber **28** are emptied into an evacuated plastic coated metal container on an endless belt arrangement.

The treating time in the finishing chambers is much longer than in the lyophilization chambers. Indeed, treatment in the lyophilization zone may be about 4 hours and 12 hours in the finishing chambers. The only significant difference between the finishing chambers **24, 26, 28** and lyophilization chamber **66** is that they have a vacuum discharge arrangement.

PACKAGING ZONE

After the material in chambers **24, 26, 28** are successively freeze dried, the contents of these chambers must be transferred to appropriate containers. Two proceedures may be followed. The packaging zone may be kept under an inert gas, such as nitrogen. Or, the empty containers may first be placed in a tray lyophilization apparatus as described in the Natelson and H. Gottfried et al. U.S. Pat. No. 3,293,772, and then sequentially released and moved out under vacuum conditions on an endless belt. Thus, as the container enters the packaging zone, it either is under substantial vacuum conditions, or under inert gas ambient conditions.

The discharge of the product into containers **38** is done in packaging zone **110**. Here, the containers **38, 38a** will leave the entrance chamber and go along a belt and roller conveyor **39**. Each chamber **24, 26, 28** in the finishing zone has a light **36, 40**, and **29** and a corresponding photocell **36a, 40a**, and **29a**. Below each chamber is a lift for the belt and roller conveyor consisting of pairs of rollers **24a, 24b; 26a, 26b; 28a, 28b**. These roller pairs are held by a pivot and arm. As each photocell is enabled, it operates a solenoid pulling down two arms under the chamber, thus lifting the roller pairs under the chamber. The arriving container **38a** hits a switch as it comes into position which stops the belt and roller conveyor drive long enough to fill the containers and opens a valve **24V, 26V, 28V** at the same time. As the container lifts, it meets a flare on the mouth of the exit of the finishing chamber. This flare on the mouth is covered with a rubber mat. Thus a close fit, mouth to mouth, is made. As the exit valve of the finishing chamber opens, a vacuum forms in the container due to the vacuum in the finishing chamber. This causes the container to adhere, as in the Natelson and H. Gottfried et al. U.S. Pat. No. 3,293,772. The vacuum pump continues to operate, a vacuum then forms in the container. The container is filled by the motion of the screw in driving the powder forward. When the container is full, the exit valve closes. The bleeder valve opens and the platform under the container is lowered. This causes the container to fall away.

This portion of the instrument operates somewhat as described in the aforesaid U.S. Pat. No. 3,293,772, except that in the present case the containers move to the discharge orifice, whereas in the aforesaid U.S. Pat. No. 3,293,772, the material treated is stationary. During the filling of the containers the suction pump arrangement of the finishing chamber keeps operating to remove any moisture in the containers. However, it is important to maintain either a partial vacuum or an inert atmosphere in the packaging zone to avoid contamination of the product.

Attention is directed to the fact that the lifting height of the containers to the discharge orifice of the finishing chambers has been greatly exaggerated in the drawing to explain the operation. In practice, the roller pairs merely lift the belt a fraction of an inch and, there is no chance of the containers tipping over because of a high lift.

Another example of a packaging zone is shown in FIG. **6** where the finishing chambers are shown as chambers **124, 126, 128**. Each finishing chamber is connected to a cold trap and vacuum pump arrangement **180**, similar to the corresponding arrangement **80**, herein before described. Each cold trap and vacuum pump arrangement **180** has two cold traps **186**, 186*a*, but each cold trap has its own pump 190, 190*a*, and each pump has its own motor 191, 191*a*. Each cold trap has a reversible refrigeration unit 188, 188*a*, and each chamber 124, 126, 128, likewise has its own reversible refrigeration unit 196. The chambers 124, 126, 128 operate exactly as described, with reference to chambers 24, 26, 28. Each chamber has an output valve 124*b*, 126*b*, 128*b*, which leads to a screw conveyor 139. The screw conveyor 139 is kept under vacuum by a condensing unit 140 operated by a pump 190*b* driven by a motor 191*a*. The containers are filled in a vacuum chamber 142 connected to the output of the screw conveyor 139. The filling of the containers has already been described in connection with FIG. 5.

PROGRAMMING AND COMPONENTS

To somewhat condense the lengthly description and to avoid describing matters known to those skilled in the art, the programming and certain components have not been described at length. The valves between the various chambers are all ball valves operated by a motor. This permits a perfect vacuum to be maintained since when the ball valve is shut there is substantially no leakage. Since many of the components operate at low temperatures, electric motors to operate the valves have been found to be more satisfactory than relays and solenoids in most cases. Also, not described in detail are program means. The respective chambers are discharged when the gas pressure therein reaches a certain pressure level. Then pressure gages actuate the respective motors opening and closing the proper valves. Since this function is well known in the arts, an extensive description of these program means have been omitted from the description. If necessary, the separate functions can be performed by hand.

OTHER EMBODIMENTS

The treating chambers described and shown in FIG. 2 all have an inner and outer screw conveyor separated by a cylindrical partition. A simple version is shown in FIG. 3. Here, chamber 166 has only a single screw conveyor 170 and a return chute 168. With chamber 166 inclined at an angle of between 15° and 30° from the vertical, the goods reaching the top of the chamber readily fall down the chute back to the bottom of the chamber. It is possible to have a single horizontal chamber 266 with an inner cylindrical partition 268 and only an inner screw conveyor 270. The return path 274 is over the inner cylindrical partition 268. FIG. 4*a* shows a different version of the horizontal chamber 366 having twin screw conveyors 370, 374 separated by a partition 368. The configuration of the partition wall 368 and the screw convolution permits the cycling of the material being treated. The arrows in the figure show the path of the material as it moves during lyophilization.

It is to be observed therefore, that the present invention provides for a freeze drying instrument having input means to introduce a controlled amount of raw material to a freezing zone 16 having a freezing chamber 54 with constant agitating means, e.g., a screw conveyor 58 therein. The material is then discharged into the first chamber 66 of a lyophilization zone 18 where the treated material is continuously circulated by a screw conveyor arrangement. The screw conveyor arrangement may consist of two screw conveyors 70, 74, one an inner, the other an outer conveyor, separated by a cylinder 68, or just a single chamber 266 with a single screw conveyor 270 and a cylinder separation 168*a* defining a return path 274*a*. It may also consist of two parallel screw conveyors 370 and 374 separated by a partition wall 368 or a single substantially vertical chamber 166 with a single screw conveyor 170 therein and a return chute 168. Material is treated in the first chamber for several hours to remove about 50 percent of the moisture. It is then transferred to a second chamber 66*a* similar to the first chamber where the treatment is continued until up to 90 percent of the moisture is removed. The material is then sequentially transferred to one of three finishing chambers 24, 26, 28, similar to chambers 66, 166, 266, 366 where the treatment is continued for upwards of ten hours to remove the remaining moisture. The finished product is then discharged into containers under vacuum conditions.

EXAMPLE

A typical example, on a pilot plant scale, is described as follows:

Freshly pressed orange juice is cooled and partially frozen. The liquid portion is forced through a stainless steel screen and the ice which separates is washed with small quantities of cold water. In this manner, 40 to 50 percent of the water is removed. This is the orange juice concentrate.

The instrument described in FIG. 3 is utilized. All chamber sizes are 2 feet long by 16 inch diameter for a volume capacity of 2.5 cubic feet or approximately 28 quarts.

The concentrate is placed in a storage vat cooled to 4°C which communicates with the solenoid valve and spigot of the instrument. When the instrument is turned on the solenoid valve opens and the orange juice is added to the V tube. There it is frozen and ground.

Twelve quarts of frozen and ground material are added to the first lyophilization chamber which is cooled to −20°C. The motor turns the screw assembly at 20 r.p.m. during the loading cycle. The ball valve closes and the vacuum is applied. For the next twenty minutes the outer wall is cooled to −20°C while the screw rotates slowly (20 r.p.m.) until a vacuum of less than 300 microns is reached indicating that the lyophilization process has been established. The screw assembly now is rotated at 60 r.p.m. As the pressure continues to fall, the wall temperature is slowly increased. When the pressure drops to 200 microns the wall temperature is now raised to 70°C with the reversible refrigerator coils.

The temperature is raised slowly so that no increase in pressure is caused, the pressure remaining at less than 200 microns. If the presence should rise, the temperature is lowered rapidly since it signifies a leak, and danger of melting of the material. The temperature is kept at 70°C, usually for approximately 3 hours. At this point, the temperature is lowered to 50°C and maintained there for approximately one-half hour when the pressure is now at 150 microns or less. If maintained at 70°C the pressure may rise indicating less water in the material and therefore, less evaporation and cooling per unit time.

When the total elapsed time is 4 hours, the contents of the first lyophilization chamber are automatically transferred to the second lyophilization chamber (maintained at 50°C) by the action of the screw conveyor in driving the material forward. The screw assembly in the second lyophilization chamber also rotates at 60 r.p.m. The first lyophilization chamber is now automatically reloaded with a second 12 liters of frozen orange juice concentrate.

The second lyophilization chamber is maintained for four hours at 50°C at which time the material is transferred to one of the three finishing chambers.

In the finishing chambers the wall temperature is programmed to be steadily reduced from 50° to 25°C or ambient temperature. The reason for this is that as the material dries, the rate of cooling decreases and no cooling is occurring when the material is dry. The screw operates at 30 r.p.m. in the finishing chambers. The material remains for 12 hours in the finishing chamber. At the end of 12 hours, a timer activates the belt drive which carries the empty glass or plastic coated metal containers. A light and photocell arrangement causes the glass container to stop under the chamber ready for unloading. The container is filled with the amount of powder equivalent to one quart of orange juice. Since the powder represents 12 liters of frozen orange juice concentrate or 24 liters of orange juice, 24 containers are so filled.

The total elapsed time for this pilot plant is 20 hours (4 + 4 + 12) to produce the first batch of lyophilized powder. Subsequent batches come out at a rate of once every 4 hours. Subsequent batches are fed to alternate finishing chambers, the first finishing chamber being re-utilized after emptying.

I claim:

1. The method of continuously and automatically lyophilizing material comprising the steps of:

a. freezing the material;

b. pulverizing the frozen material;

c. circulating the material from an inlet zone along a screw conveyor while exhausting the ambient gas to heat the material and exposing particles of material to heat and recycling the material back to the inlet zone;

d. regulating the heat supplied by the rate of rotation of the circulating screw and by reversible refrigeration;

e. alternately exhausting said ambient gas in first and second cold traps;

f. sequentially finishing partially treated material in at least first and second finishing zones; and,

g. discharging the material into containers without breaking the vacuum.

2. In an automatic and continuous freeze drying instrument, in combination,

a. input means to introduce under vacuum a material to be processed in the instrument;

b. an elongated lyophilization chamber, connected to said input means, said chamber having screw conveyor means therein for conveying and agitating said material longitudinally in said chamber to one end thereof, including means for adjustably rotating said screw conveyor means at predetermined speeds, said rotation also supplying heat to the chamber and means for controlling said adjustable rotating means for controlling the temperature in the chamber;

c. a return path coupled to said chamber for guiding material from said one end to the other end of said chamber;

d. reversible refrigeration means to cool or heat said chamber and thus maintain the wall temperature within a predetermined range;

e. a vacuum pump and cold trap arrangement to constantly evacuate gas formed by moisture in said chamber; and,

f. vacuum discharge means to discharge treated material from the chamber while maintaining the vacuum in the chamber.

3. A device for the continuous dehydration of materials which are sensitive to heat which comprises,

a. a conveyor to continuously feed said material to be dehydrated to a first freezing and crushing compartment;

b. a reversible first refrigeration unit attached to said freezing and crushing compartment; said freezing and crushing compartment having means for pulverizing the frozen material, said first refrigeration unit supplying a constant reduced temperature to said freezing compartment so as to freeze and maintain the material in a frozen state;

c. a lyophilizing and constant movement zone connected to said freezing and crushing compartment; said lyophilizing zone being attached to a condensing zone; said condensing zone being maintained at a low temperature by a second reversible refrigeration unit so as to condense the moisture removed from the material being dehydrated;

d. a vacuum pump attached to said condensing zone so as to maintain a reduced pressure causing the material in the lyophilizing zone to give up its water and cool itself, thus remaining in a frozen state;

e. a third reversible refrigeration unit attached to said lyophilizing and constant movement zone so as to maintain a flow of heat to the material being dehydrated so as to supply energy required for lyophilization;

f. means for continuously agitating progressively advancing and recycling the material in the lyophilizing and constant movement zone; and,

g. a receiving and packaging zone attached to said lyophilizing and constant movement zone so that the material can be removed from the lyophilizing and constant movement zone without breaking the vacuum in the major portion of said zone in order for the material to be removed and packaged in a continuous mode;

h. and means for adjusting said continuously agitating means at predetermined speeds, said continuously agitating means also supplying heat to the lyophilizing and constant movement zone; and means for controlling said adjusting means for controlling the temperature in the lyophilizing and constant movement zone for controlling the temperature in the zone.

4. In a freeze drying instrument, in combination,

a. input means to introduce under vacuum a material to be processed in the instrument;

b. an elongated lyophilization chamber, connected to said input means, said chamber having agitation conveyor means therein for conveying said material longitudinally in said chamber to one end thereof at predetermined speeds, means for adjusting said agitation conveyor means at predetermined speeds, said agitation exposing all parts of said material to treatment and heating said material, and means for controlling said adjusting means for controlling the temperature in the chamber,

c. a return path coupled to said chamber for guiding material from said one end to the other end of said chamber;

d. refrigeration means to cool or heat said chamber and thus maintain the wall temperature within a predetermined range;

e. a vacuum pump and cold trap arrangement to constantly evacuate gas formed by moisture in said chamber; and,

f. vacuum discharge means to discharge treated material from the chamber while maintaining a vacuum in said chamber.

5. An instrument as claimed in claim **4**, wherein said refrigeration means are reversible.

6. An instrument as claimed in claim **4**, including

a. in said input means, constant leveling means and a cooled pipe (**14**) connected to a freezing zone (**16**); and,

b. said freezing zone comprising an elongated chamber having a screw conveyor therein and refrigeration means to heat and cool said freezing zone.

7. An instrument as claimed in claim **4**, wherein said lyophilization chamber is generally vertically inclined, said agitation means is a central screw conveyor and said return path is a chute from the top of said chamber to the bottom thereof on the outer side of said chamber, said material being carried upward by the screw conveyor and carried downwards by said chute.

8. An instrument as claimed in claim **4**, wherein said lyophilization chamber is substantially horizontal including first and second screw conveyors for conveying material in opposite directions, with a partition wall therebetween.

9. An instrument as claimed in claim **4**, wherein said lyophilization chamber is generally vertically inclined, said agitation means is a central screw conveyor in said lyophilization chamber and said return path includes a cylindrical partition in said chamber.

10. An instrument as claimed in claim **9**, including an outer screw conveyor with a few convolutions around said partition.

11. An instrument as claimed in claim **4**, including at least first and second finishing chambers connected to said lyophilization chamber, said finishing chambers being constructed similar to said lyophilization chambers and being coupled to said vacuum discharge means, the material being first partially lyophilized in said lyophilization chamber until ambient pressure in said lyophilization chamber reaches a predetermined level and then transferred to said finishing chamber when all remaining moisture is removed.

12. An instrument as claimed in claim **10**, including means for automatically transferring material between chambers under vacuum.

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