Jan. 24, 1967 E. THUSE 3,299,525

CARRIER GAS SUBLIMATION

Filed June 29, 1964 2 Sheets-Sheet 1

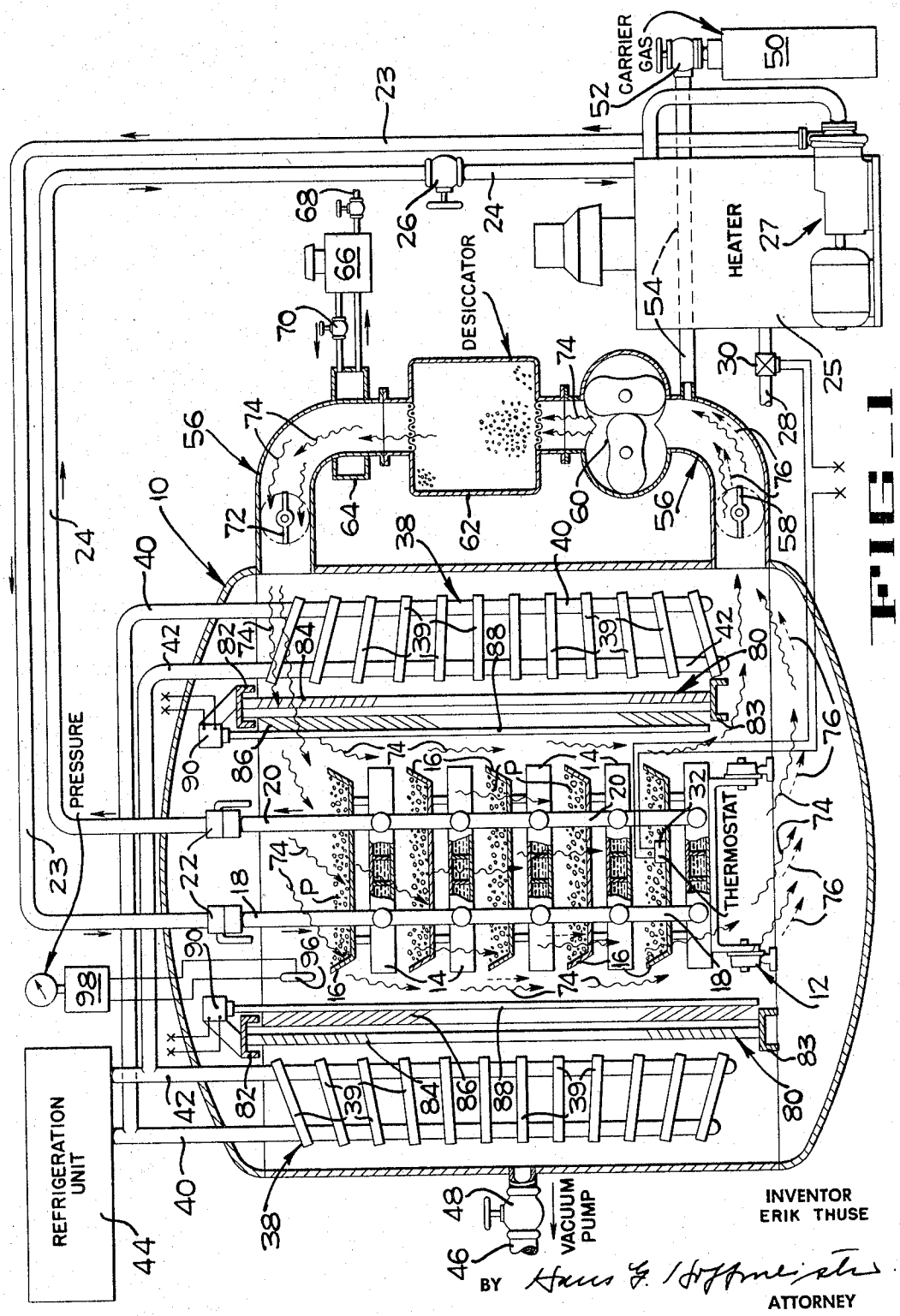


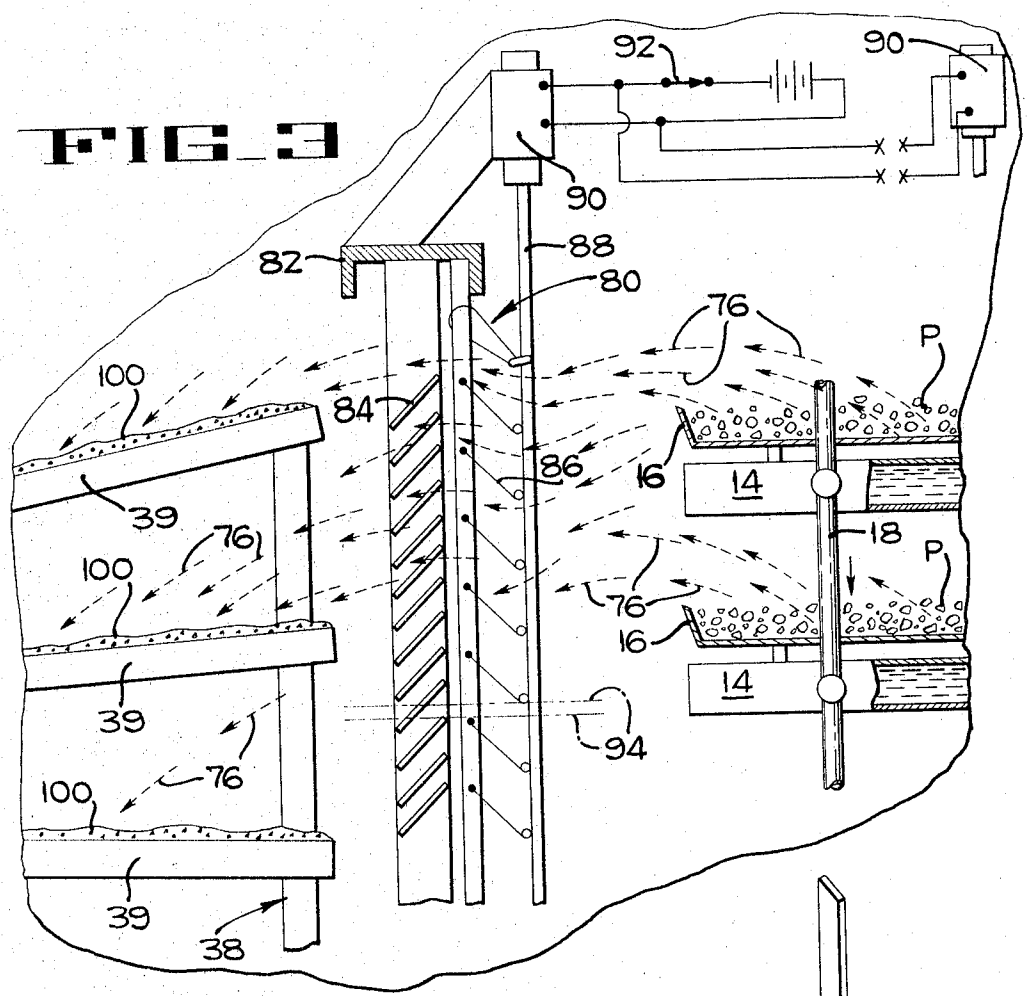
FIG. 1

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CARRIER GAS SUBLIMATION

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FIG_3



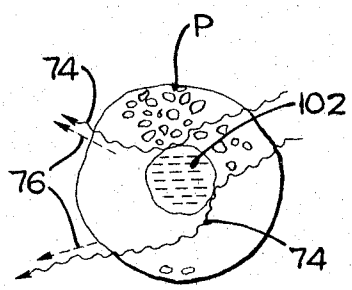


FIG_4

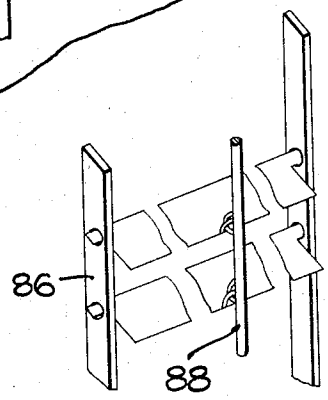


FIG_2

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United States Patent Office 3,299,525
Patented Jan. 24, 1967

3,299,525

CARRIER GAS SUBLIMATION

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Filed June 29, 1964, Ser. No. 378,883
6 Claims. (Cl. 34—5)

This invention relates to freeze drying of cellular material such as food products, biologicals, etc., and more particularly to a method and apparatus for freezing such products which involves the use of a carrier gas.

One of the principal problems in freeze drying processes of the type referred to is that the drying cycle may be quite prolonged. This renders the process costly, particularly when high tonnage, low cost materials are being dried, such as various food products. Attempts to shorten the drying cycle by increasing the heat applied to the product must be carefully controlled, because if the temperature of the dried portion of the product is permitted to rise above a certain value, which depends upon the nature of the product, then the product will be scorched or otherwise down graded during the drying process. It has been proposed to shorten the drying cycle by using various heat carrier gases and circulating them to the drying chamber. Although this is an effective process, its use throughout an entire drying cycle represents additional economic loss in the form of pumping costs, additional refrigeration costs where refrigeration is used in the drying chamber for condensing entrained water vapor, and other factors.

It is an object of the present invention to shorten the drying cycle in a freeze drying process.

Another object is to shorten the drying cycle by circulating an inert or non-oxidizing carrier gas through the drying chamber, after a large proportion of the water vapor has been removed from the product by conventional evacuation and vapor condensation methods.

Another object is to reduce the pumping and other costs attendent with the use of a carrier gas.

Still another object of the present invention is to eliminate the need for cooling or refrigerating the carrier gas.

It is an object of the present invention to permit the use of a desiccator in the carrier gas circulation system which is relatively small in capacity compared to the size of desiccators usually employed in this service.

A further object of the invention is to provide a drying chamber for receiving carrier gas as described, which chamber also contains the condenser plates for condensing the water vapor, and which includes means for shielding the condenser plates from heat radiated from the heating shelves, and from the carrier gas in the chamber, when present.

A further object of the present invention is to provide adjustable carrier gas shielding louvers between condensing plates and product shelves and trays in a freeze drying chamber.

Another object of the invention is to provide a method of operating a freeze drying chamber including vapor condensing means, and a carrier gas circulating system.

The manner in which these and other objects may be obtained will be apparent in the following detailed description of the invention.

In the drawings:

FIGURE 1 is a diagram of a freeze drying system embodying the invention.

FIGURE 2 is a fragmentary perspective showing a portion of the louver assembly.

FIGURE 3 is an enlarged fragmentary diagram showing the operation of the system during an initial part of the freeze drying cycle.

FIGURE 4 is a section through a sample of the material being dried, including the ice core.

The system of FIG. 1 includes a drying chamber **10** which has a loading door (not shown) for introducing and removing the product. Details of the shape, construction, etc., of the drying chamber **10** are not critical to the present invention. It is contemplated that the drying chamber will be basically like that of the patent to Abbott et al., No. 3,132,930, issued May 12, 1964. The product is introduced and removed from the drying chamber **10** on a car, indicated diagrammatically at **12**, which car is also like that disclosed in the aforesaid Abbott et al. patent. The car is provided with heated shelves **14**, through which a heated liquid is circulated for supplying the heat of sublimation to the product. Before the car **12** is introduced into the drying chamber **10**, trays **16** are loaded with the product P. These trays are supported on the heated shelves **14** by feet, which separate the trays from the heated shelves and cause the heat from the heated shelves to reach the product largely by radiation from the shelves. The heating liquid for the trays, which may be Propylene glycol, is introduced and removed from the trays by lines **18** and **20**. Quick disconnect couplings **22** are provided, to permit removal of the car **12** from the drying chamber. These couplings incorporate shut-off valves, which are closed when the couplings are disconnected. Couplings of this type are conventional in the art, and the details thereof are not critical to the present invention.

The heated liquid is circulated through the heating shelves **14** in the drying chamber by lines **23** and **24**, which connect to a liquid heater unit **25**. A shut off valve **26** is provided in one or both of the lines **23**, **24**. As illustrated, a pump assembly **27** circulates the liquid through the heating coils (not shown) in the heater **25**. The details of the heater **25** are not critical to the invention. As illustrated, it is a gas heater which receives heating gas from a gas inlet line **28** controlled by a remotely controlled or solenoid operated valve **30**. The valve **30** is in turn controlled by a thermostat unit **32** resting on one of the trays in the drying chamber. Heat control in this manner, where the product is heated radiantly, gives a more precise indication of the moisture content of the product as described in the aforesaid Abbott et al. patent.

A condenser plate assembly **38** is mounted on each side of the car bearing the product laden trays. Each assembly comprises a plurality of hollow condenser or cold plates **39**, which by manifolds and headers receive refrigerant for circulation by lines **40** and **42** from a refrigeration unit **44**. The details of the connections to condenser assembly **38** are not critical to the present invention and are preferably like those described in the aforesaid Abbott et al. patent.

Systems of the type described must be provided with means for removal of air and other non-condensable gases initially in the chamber, as well as for making up for any leakage of such gases into the chamber as may occur during the drying cycle. Thus, a line **46** is connected to a vacuum pump (not shown) and controlled by a vacuum valve **48**. At all times, the pressure within the drying chamber **10** must be below that corresponding to the pressure of water vapor at the triple point, to avoid wetting the product.

A feature of the system of the present invention is the use of an inert or non-oxidizing carrier gas during a terminal portion of the drying cycle, for assisting in conducting heat to the ice core of the product particles, and for sweeping away the additionally sublimed water vapor. As illustrated in the diagram of FIG. 1, the carrier gas, which may be helium for example, is supplied in a pres-

sure vessel **50**, which carries a gas valve **52** that controls a gas admission line **54** leading into the system. A carrier gas circulating conduit **56** connects between the lower and upper portions of the drying chamber **10**. This conduit has an inlet valve **58**, which may be a conventional butterfly valve, and the gas is circulated by a blower **60**. Since the pressure within the system will be quite low, in the order of 2 to 30 millimeters of mercury, it is contemplated that a positive displacement blower be employed, such as the well known Roots blower. After leaving the blower, the carrier gas is directed through a desiccator **62**. The desiccator is filled with a water absorbent agent, such as silica gel or the like, and is removable for drying during the downtime of the system. Alternatively, and in accordance with conventional drawing practice, two desiccators may be provided, with suitable valving so that one can be removed for dehydrating while the other is in service.

It is contemplated that the carrier gas may be heated by a heater, the details of which are not critical to the invention. As illustrated, the heater is in the form of a heating chamber **64** surrounding the carrier gas circulating conduit **56**. When in use, hot air is circulated through chamber **64** from a gas heater **66**, supplied with gas by a gas line **68**, manually controlled by a valve **70**. Usually, however, the carrier gas picks up sufficient heat from the heated shelves **14**.

The end of the carrier gas circulating conduit that connects to the upper portion of the drying chamber **10** is also provided with a butterfly valve **72**. Obviously, closing of both the butterfly valves **58** and **72** isolates the carrier gas system from the drying chamber. In FIG. 1, the wavy arrows **74** indicate the flow of carrier gas through the system, when the carrier circulating system is in operation. Also a convention has been adopted wherein the entrained water vapor is indicated by the dashed arrows **76** in the drawings.

It is a feature of the present invention that means are provided to isolate the condenser assemblies **38** from receiving radiant heat from the heated shelves **14**, and for discouraging the flow of carrier gas into the condensing zones, when the carrier gas is being circulated through the chamber. In order to accomplish this, a louver assembly indicated generally at **80** is provided between each condenser assembly **38** and the car with its trays and shelves within the vacuum chamber. The louver assembly is mounted by means not critical to the invention, such as upper and lower channels **82, 83**. Two sets of louvers are employed—a set of fixed louvers **84** and a set of adjustable louvers **86**. Referring to FIGS. 2 and 3, it can be seen that the adjustable louvers **86** include a louver adjusting bar **88** which is actuated by an electric solenoid **90** mounted on the channel **82**. An electric circuit for supplying energy to the solenoid can be controlled by a switch **92**, which switch is actually mounted outside of the drying chamber.

In FIG. 3, the broken lines **94** show that even when the louvers **86** are in their open position, there is no line of sight path between the heated shelves **14** and the condenser assemblies **38**. This blocks off radiant heat from the shelves, and prevents such heat from adding to the load of the refrigeration system when removing heat from the condenser assemblies. In the form of the invention illustrated, this line of sight exclusion of radiant heat is accomplished by selecting the relative spacing and positioning of the fixed louvers **84**, and the adjustable louvers **86**.

Pressure within the drying chamber is sensed by a pick up **96** in the chamber, and indicated by an indicator unit **98**. A low pressure unit of this type is sold as the absolute pressure recorder unit (series 500), by the Bristol Co. of Waterbury, Conn.

In operation, the car **12** will have been removed from the drying chamber and its trays will have been loaded with the product P and placed on the car. The condenser plates **39** will have been flushed clear of ice from the previous cycle. The car is pushed into the drying chamber and the heating liquid connections **22** are made. The butterfly valves **58** and **72** in the carrier gas circuit will be closed. The door (not shown) to the drying chamber is closed and sealed, and the vacuum pump connected to line **46** is started up with the vacuum valve **48** open. Of course, as in conventional drying systems the product P may have been pre-frozen before loading, although if desired the freezing can take place in the drying chamber if the vacuum is drawn low enough by the vacuum pump. However, this adds to the cycle time, it is more efficient to freeze the product outside of the vacuum chamber before it is introduced therein.

With the vacuum pump in operation, the noncondensable gases in the vacuum chamber are soon exhausted and the pressure drops to well below the triple point. In fact, the pressure can be brought down to a millimeter of mercury or less, if desired at this stage. Heated liquid is now circulated through the shelves **14**. As soon as the pressure thus drops, water sublimes from the frozen product, and as illustrated in FIG. 3 by the dashed arrows **76**, this sublimed water vapor passes freely through the adjustable louvers **86** and the fixed louvers **84**, to the condensing plates **39**. Here the water vapor is at once frozen to form ice particles **100**, deposited on the condenser plates. This freeze drying by sublimation continues and the ice core of the product P, FIGURE 4, which is illustrated diagrammatically at **102**, gradually is reduced in size and recedes from the surface of the product particles. This recession of the ice core leaves a surrounding volume of dried cellular material, that acts as a good heat insulator.

Toward the end of the drying cycle, the ice core has become so small and the insulating volume so large, that the effectiveness of heat radiation in penetrating to the ice core is reduced, thereby prolonging the drying time unnecessarily. When this condition occurs, which is approximately after 90% of the water vapor has been sublimed from the product, the adjustable louvers **86** are adjusted to their substantially closed position, as illustrated in FIG. 1. Also, the butterfly valves **58** and **72** are opened and the blower **60** is started. Carrier gas such as helium is admitted by the opening of the gas valve **52**, and circulation of the carrier gas begins. The admission of the carrier gas continues until the pressure gauge unit **98** indicates the desired pressure, which may be very low, in the order of 10 millimeters of mercury. As disclosed in the co-pending application of Barth et al., Serial No. 271,083, filed April 8, 1963, a good operating zone is at a pressure in the order of 2–30 millimeters of mercury. This makes an effective compromise of the conflicting requirements as to heat transfer into the ice core and diffusion of the water vapor through the dried shell of product from the ice core.

As illustrated in FIG. 4, the carrier gas, indicated by wavy arrow **74**, sweeps through the shell of dried material and contacts the ice core **102**, thereby giving up heat to the ice core and assisting in further sublimation of water vapor. The carrier gas picks up heat from the shelves **14**, which continue to receive heating liquid. The water vapor, indicated by the dashed arrows **76** in FIG. 4, is picked up and entrained by the carrier gas, and is carried down past the inlet valve **58** through the blower **60** and into the desiccator **62**. Here the water vapor is removed from the carrier gas, and the dry carrier gas is recirculated through the outlet valve **72**, back into the drying chamber and across the product and shelves **14**. If desired, heat may be added by the carrier gas heater **66**, previously described.

The refrigeration system can be kept in operation, since the louvers **86** and **84** do not completely block off vapor from passing therethrough, if large amounts of water vapor are evolved, some water vapor can be attracted by the usual pressure differential to the condenser plates **39**. During the operation of the carrier gas system as shown in FIG. 1, the vacuum pump can be shut off, or

at least the valve 48 controlled to limit the flow through the vacuum to keep the pressure in the chamber within the selected range, say 10 millimeters of mercury. As mentioned, the carrier gas, if it is not heated by the heater 66 will pick up heat from the heated shelves 14 and conduct it directly to the ice core, as illustrated in FIG. 4. Thus, during the last 10% of the drying cycle, the product is thoroughly and rapidly dried, without requiring an increase in the temperature of the liquid supplied to the heating shelves 14, with attendant scorching of the product.

As described in the aforesaid Abbott et al. patent, the thermostat 32, which measures the product temperature will control the liquid heater 25, and as the product becomes dryer it will reduce the heat delivered to the heating shelves 14, and prevent scorching of the substantially dried product. When the product is completely dried, this temperature will rise rapidly, which is an indication that the process should be stopped. Valves 72 and 58 in the carrier gas system are closed, and the vacuum line is shut off or directed to atmosphere to break the vacuum. Circulation of heating liquid through the heated shelves is stopped, and the refrigeration unit is turned off. With the vacuum broken, the door to the drying chamber is opened, and the dried product removed with the car 12. The amount of carrier gas such as helium, actually required for a drying cycle, is relatively small. As can be seen, with the pressure as low as 10 millimeters of mercury, the gas expands and it requires but a small quantity of gas to fill the circulating system. Thus the saving in drying time more than offsets the slight additional cost of the carrier gas, such as helium, required during the last 10% or so of the drying cycle.

Having completed a detailed description of the invention so that those skilled in the art may practice the same, I claim:

1. The method of freeze drying cellular materials comprising the steps of placing frozen cellular materials in a chamber, supplying heat of sublimation to the ice core of the frozen material by radiation and conduction, reducing the partial pressure of the water vapor in the chamber below the triple point pressure by condensing the water vapor sublimed from the frozen product directly in the chamber, initiating the circulation of a dry, heated, non-oxidizing, heat carrier gas through the chamber and across the materials after a majority of the ice core in the product has sublimed to sweep away water vapor and increase the conductivity of the shell of the dried material, partially shielding the vapor condensing elements in the chamber from the carrier gas, removing water vapor entrained by the gas, reheating the gas, and re-circulating the gas through the chamber until the material has fully dried.

2. Freeze drying apparatus comprising a drying chamber, a plurality of product supporting shelves in said chamber, means for heating said shelves for subliming water vapor from a frozen product, means for withdrawing non-condensible gases from said chamber, means in said chamber for condensing water vapor sublimed from the ice core of the product, means for circulating a dry-non-oxidizing gas through said chamber to provide heat of sublimation to the produce ice core by diffusion through the product shell, control means for introducing the gas into said chamber after a majority of the water vapor has been sublimed from the product, and louvers between said product shelves and said condensing means for accommodating the flow of water vapor to the condensing means while shielding the condensing means from said gas.

3. The apparatus of claim 2, wherein said louvers are adjustable.

4. Freeze drying apparatus comprising a drying chamber, a plurality of product supporting shelves in said chamber, means for heating said shelves for subliming water vapor from a frozen product, means for withdrawing non-condensible gases from said chamber, means at each side of said chamber for condensing water vapor sublimed from the ice core of the product, means for circulating a dry-non-oxidizing gas through said chamber to provide heat of sublimation to the product ice core by diffusion through the product shell, control means for introducing the gas into said chamber after a majority of the water vapor has been sublimed from the product, and louver assemblies between said product shelves and said condensing means for accommodating the flow of water vapor to the condensing means while shielding the condensing means from said gas, said louver assemblies comprising a set of fixed louvers and an adjacent set of adjustable louvers, the individual louver elements of said sets cooperating to block line of sight heat radiation from said shelves when the adjustable louvers are in their open position.

5. Freeze drying apparatus comprising a drying chamber, a plurality of product supporting shelves in said chamber, means for heating said shelves for subliming water from a frozen product, means for withdrawing non-condensible gases from said chamber, means for condensing water vapor sublimed from the ice core of the product, means for circulating a dry-non-oxidizing, heated, heat carrier gas through said chamber to provide heat of sublimation to the product ice core by diffusion through the product shell, said gas circulating means comprising a conduit external of said chamber having a gas inlet at an upper portion of the chamber, a gas and water vapor outlet at a lower portion of the chamber, a blower, a desiccator, and a heater between said desiccator and said gas inlet, and control means for circulating said carrier gas through the chamber after a majority of the water vapor has been sublimed from the product.

6. The method of freeze drying cellular materials comprising the steps of placing frozen cellular materials in a chamber, supplying heat of sublimation to the ice core of the frozen material by radiation and conduction, reducing the partial pressure of the water vapor in the chamber below the triple point pressure by condensing the water vapor sublimed from the frozen product, initiating a substantially continuous circulation of a dry, non-oxidizing, gas through the chamber and across the materials after a majority of the ice core in the product has sublimed, to sweep away water vapor and increase the conductivity of the shell of the dried material, continuing said gas circulation until the material has fully dried, and heating said gas externally of the chamber to supply additional heat for sublimation.

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WILLIAM J. WYE, *Primary Examiner.*