April 6, 1965 M. E. FUENTEVILLA 3,176,408

FREEZE DRYING APPARATUS AND METHOD

Filed July 24, 1961 3 Sheets-Sheet 1

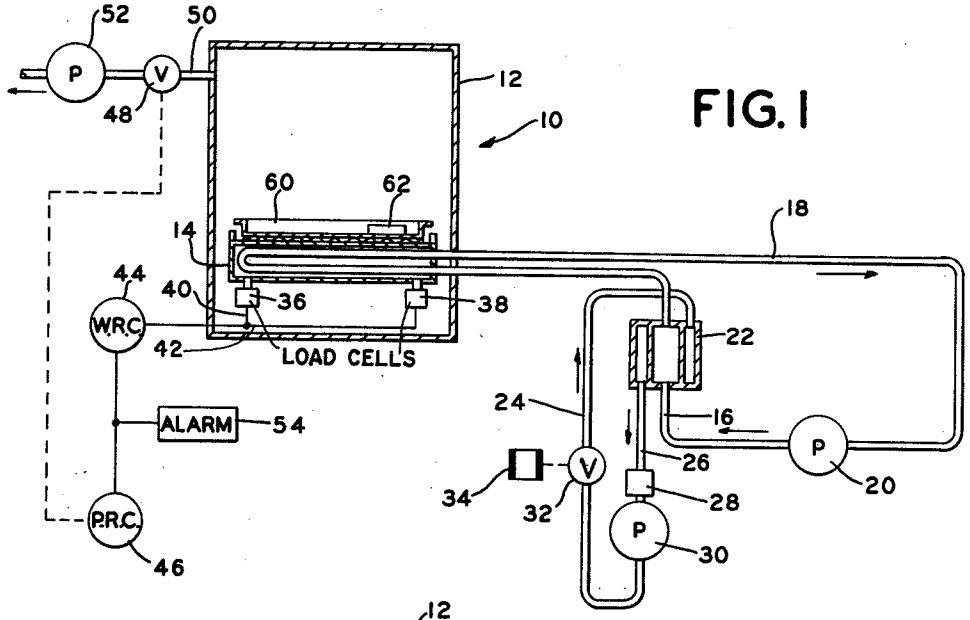

FIG. 1

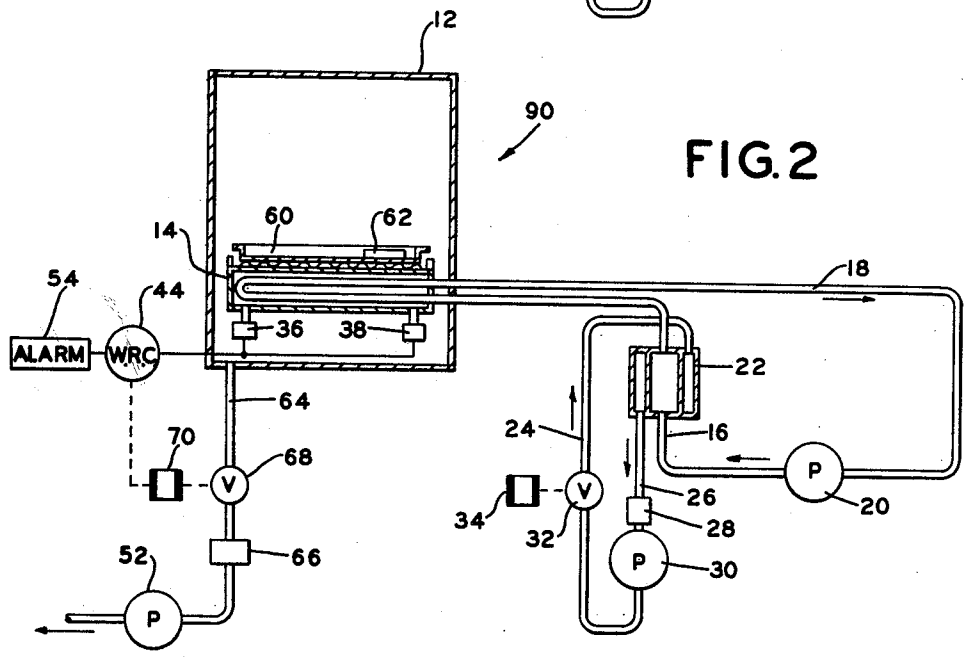

FIG. 2

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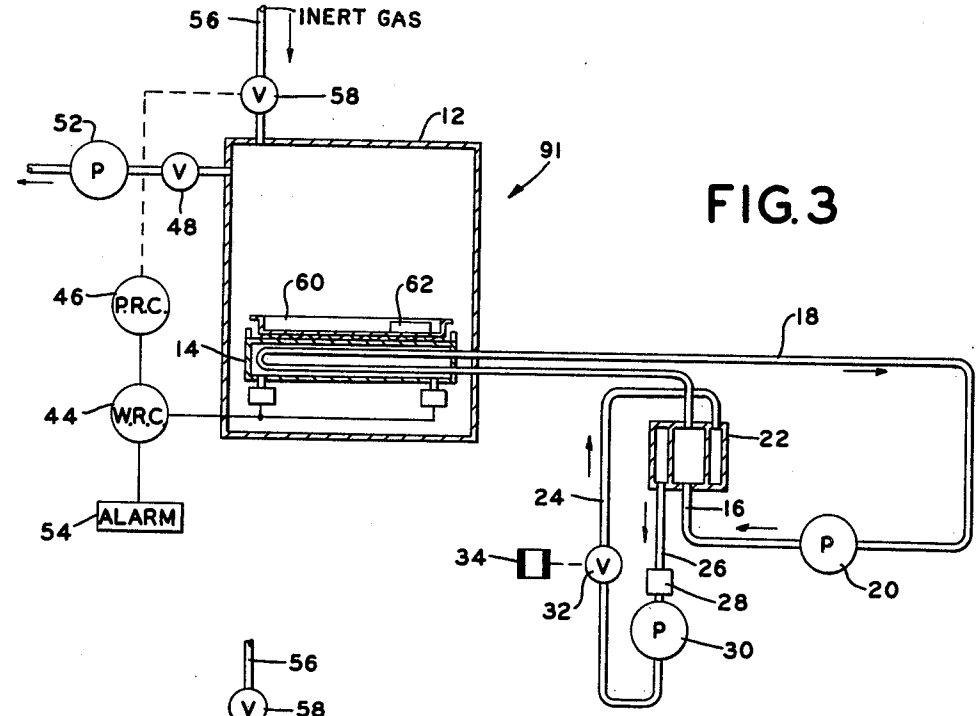

FIG. 3

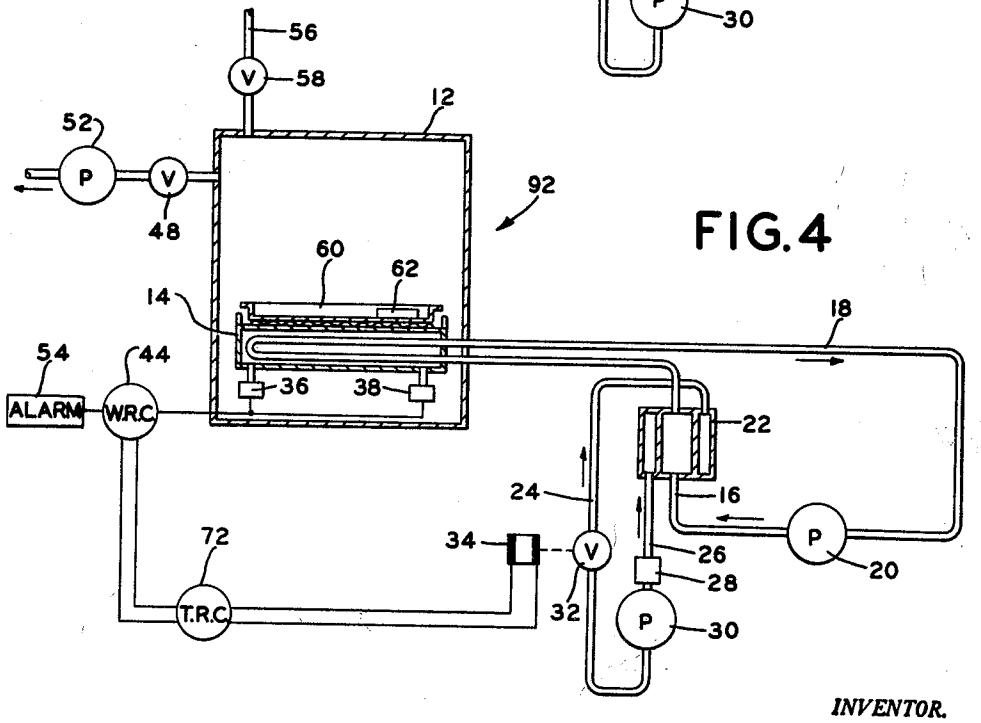

FIG. 4

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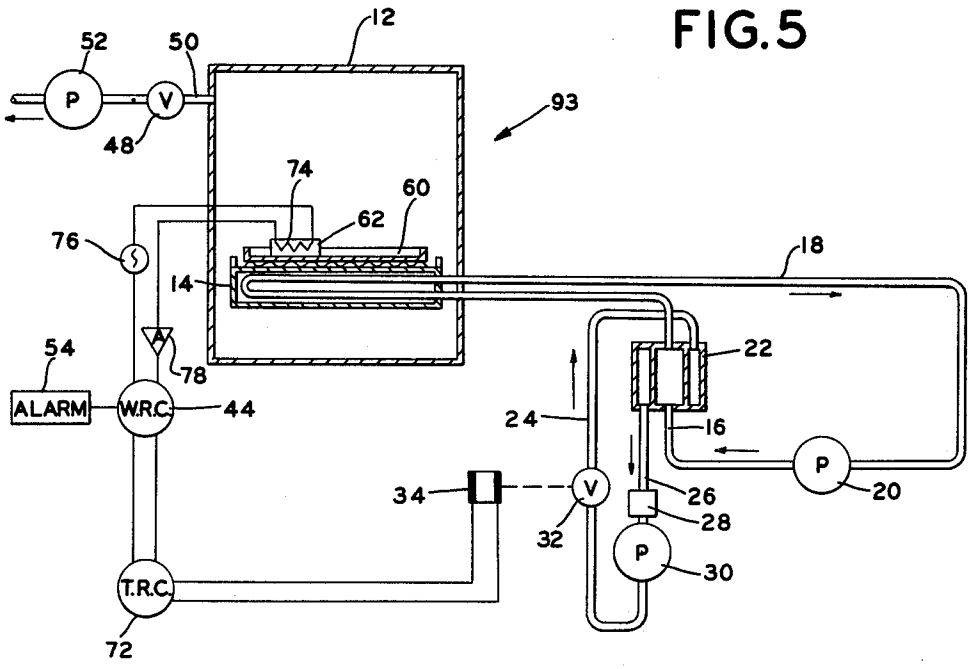
FIG.5



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FREEZE DRYING APPARATUS AND METHOD

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10 Claims. (Cl. 34—5)

This invention relates to a freeze drying apparatus and method, and more particularly to a freeze drying apparatus and method wherein the freeze drying cycle is automatically controlled in response to the loss of weight of the articles undergoing freeze drying.

Numerous processes and apparatus for freeze drying specimens have been proposed heretofore. In such devices and processes proposed heretofore, the length of the freeze drying cycle is based on empirical formulae. Accordingly, the cycle for the freeze drying process is nothing more than a calculated guess. Accordingly, it will be obvious that numerous inefficiencies are present in the apparatus and methods proposed heretofore.

The present invention is directed to a method and apparatus wherein the freeze drying cycle is automatically controlled in response to the loss of weight of the articles being freeze dried. When using the apparatus or method of the present invention, it is not necessary for an operator to set any controls as to the timing of the cycle nor periodically inspect the articles being freeze dried so as to ascertain the status of the articles. In accordance with the present invention, the loss of weight of the articles being freeze dried is ascertained in accordance with any one of the embodiments of the present invention and means are provided for automatically stopping or varying the freeze drying cycle while at the same time activating an alarm so as to alert the operator.

In accordance with the present invention, the change in the loss of weight of the articles being freeze dried is ascertained in response to load cells which support the articles or in response to the electrical resistivity of the articles. As a result of the change in the weight of the articles being freeze dried, the heat being supplied to the articles may be varied, or the pressure within the chamber enclosing the articles may be varied.

It is an object of the present invention to provide a novel apparatus for freeze drying articles.

It is another object of the present invention to provide a novel method for freeze drying articles.

It is still another object of the present invention to provide a novel apparatus for freeze drying articles wherein the freeze drying cycle is automatically controlled in response to the change in weight of the articles being freeze dried.

It is a still further object of the present invention to provide a novel method of freeze drying articles by varying the freeze drying cycle in response to the change of weight in the articles being freeze dried.

Other objects will appear hereinafter.

For the purpose of illustrating the invention there is shown in the drawings forms which are presently preferred; it being understood, however, that this invention is not limited to the precise arrangements and instrumentalities shown.

FIGURE 1 is a schematic illustration of freeze drying apparatus in accordance with the present invention.

FIGURE 2 is a schematic illustration of another embodiment of the present invention.

FIGURE 3 is a schematic illustration of another embodiment of the present invention.

FIGURE 4 is a schematic illustration of another embodiment of the present invention.

FIGURE 5 is a schematic illustration of another embodiment of the present invention.

Referring to the drawing in detail, wherein like numerals indicate like elements, there is shown in FIGURE 1 a freeze drying apparatus designated generally as **10**.

The freeze drying apparatus illustrated schematically as **10** comprises a housing **12** having a plurality of hollow shelves **14** disposed one above the other and supported by said housing. Only one such shelf is illustrated in the drawing. The shelf **14** is hollow so that a heating medium may be circulated therethrough. The interior of the shelf **14** is connected to an inlet conduit **16** and an outlet conduit **18**.

The conduits **16** and **18** are connected to a pump **20** which continuously circulates the medium through the shelf **14**. A heat exchanger **22** is disposed in the conduit **16** between the pump **20** and the shelf **14**. The temperature of the heat exchanger **22** is regulated by a controlling fluid such as steam which is circulated therethrough by means of the inlet conduit **24** and the outlet conduit **26**. The outlet conduit **26** is connected to a steam generator **28** having a pump **30**. The outlet side of the pump **30** is connected to the inlet conduit **24**. A valve **32** having a solenoid operator **34** is disposed within the conduit **24**.

The shelf **14** is supported by load cells **36** and **38**. Load cells **36** and **38** are commercially available devices which generate an electric current in response to pressure. Such load cells are extremely accurate and are not effected by the temperatures and pressures within the housing **12**. The load cells **36** and **38** are connected to the weight recorder and controller **44** by wires **40** and **42**. The weight recorder and controller **44** is a commercially available device which permanently records the electrical current generated by the load cells **36** and **38** on a graph which may be interpreted in terms of weight. In addition, the weight recorder and controller **44** relays the current received from the load cells **36** and **38** to a controller **46**.

The controller **46** regulates the flow of motive fluid to the diaphragm on a valve **48** in the conduit **50**. The conduit **50** is connected to the interior of the housing **12**. The conduit **50** is provided with a vacuum pump **52** for evacuating the interior of the housing **12**. An alarm **54** is connected to the weight controller and recorder **44** and is responsive to a minimal amount of current.

A tray **60** is supported by the shelf **14** with legs on said tray spacing the same from the shelf **14**. The tray **60** is filled with articles **62** which are to be freeze dried within the housing **12**. The condenser **66** is connected to the housing **12** by means of a conduit **64**.

The apparatus **10** operates as follows:

A tray **60** of articles **62** which are to be freeze dried is disposed within the housing **12** on the shelf **14**. The articles **62** are prefrozen. The door (not shown) providing access to the interior of the housing **12** is provided with a seal means to that the housing **12** is hermetically sealed in the closed disposition of the door. A heating fluid is circulated through the shelf **14** by means of conduits **16** and **18**. The circulating fluid within the shelf **14** heats the articles **62** by radiation. All surfaces

on the shelf 14 and the inner surface of the housing 12 are colored black while the inner surface on the door is colored white.

As the articles 62 are heated by radiation, they are subject to a vacuum within the housing 12 effected by the pump 52. The combination of heat and the vacuum causes the ice within the frozen articles 62 to sublime with the resultant vapor being removed by the pump 52. As the ice within the articles 62 sublimes, the weight supported by the load cells 36 and 38 decreases.

After a predetermined period of time, the weight of the articles 62 will become stabilized thereby indicating that substantially all of the ice is removed therefrom by sublimation.

During the entire cycle, the controller 44 is recording the change in the current generated by the load cells 36 and 38 and relaying the same to alarm 54 and controller 46. In response to a minimum current indicating that substantially all ice has been removed from the articles 62, the alarm 54 is activated and controller 46 is activated so as to close the valve 48. Thus, it will be seen that the freeze drying cycle is controlled so as to stop the cycle as soon as substantially all of the ice has been removed from the articles 62.

It has been determined that there is an optimum rate of change of weight of the articles 62 with respect to time. It is possible to utilize a controller 46 which is of the type which is capable of receiving a signal from the controller 44 so that the valve 48 is constantly varied during the freeze drying process. Thus, the apparatus 10 may be operated with optimum efficiency.

An alternative embodiment of the present invention is illustrated schematically in FIGURE 2 and designated generally as 90. The apparatus 90 is substantially identical with the apparatus 10 except as will be made clear hereinafter. The controller 44 relays a signal to a solenoid 70 which operates the valve 68 in the conduit 60. In response to a minimal signal, the solenoid 70 is actuated to regulate the valve 68. The outlet side of the condenser 66 is connected to the inlet side of the vacuum pump so that all condensate is removed from the air by the condenser 66 before being discharged to atmosphere by the pump connected thereto.

There is shown in FIGURE 3 another embodiment of the present invention designated generally as 91. The apparatus 91 is substantially identical with the apparatus 10 except as will be made clear hereinafter. In the apparatus 91, the signal received by the weight recorder control is transmitted to the controller 46 which controls the flow of motive fluid to the diaphragm actuated valve 58 in the conduit 56. The conduit 56 is utilized to inject an inert atmosphere into the housing 12. The injection of an inert atmosphere in the housing 12 slows down the freeze drying cycle since it effects the vacuum therein.

As shown more clearly in FIGURE 4, another embodiment of the present invention designated generally as 92 is illustrated. In response to an electrical signal from the load cells 36 and 38, the controller 44 actuates the alarm 54 and transmits a signal to the controller 72. The controller 72, in turn, actuates the solenoid 34 thereby closing the valve 32. When the valve 32 is closed, the heat exchanger 22 ceases to be effective.

Another embodiment of the present invention designated generally as 93 is illustrated in FIGURE 5. In order to ascertain the change in the weight of the articles 62 which are being freeze dried, an electrical resistor 74 is disposed within one or more of the articles 62. The resistor 72 is disposed in an electrical circuit having a source of power 76 with the change in resistance being fed to the controller 44 through an amplifier 78.

As the ice in the articles 62 sublimes, the resistance of the articles 62 is constantly changing. When substantially all of the ice has sublimed from the articles 62, the resistance through the articles 62 will be substantially

constant. At this point, the controller 44 will activate the alarm 54 and transmit a signal to the controller 72 which causes the solenoid 34 to close the valve 32.

Then, it will be seen that the present invention discloses a plurality of embodiments capable of controlling the freeze drying process in response to the change of weight of the articles being freeze dried. It will be appreciated by those skilled in the art that it may be desirable to combine one or more of the embodiments illustrated. For example, it is often desirable to connect the motor for the pumps 20 and 52 in series with the electrical circuit for the solenoid valve 34 so that the pumps 20 and 52 are stopped at the same time that the valve 32 is closed. Also, it is often desirable to close the valve 32 at the same time that the valve 68 is closed.

The present invention may be embodied in other specific forms without departing from the spirit or essential attributes thereof and, accordingly, reference should be made to the appended claims, rather than to the foregoing specification as indicating the scope of the invention.

I claim:

1. Apparatus for freeze drying articles of substantially the same size and kind comprising a housing having a chamber therein, a shelf within said chamber, a tray for frozen articles to be freeze dried within said housing, said tray being supported on said shelf, means for freeze drying the articles on said tray by causing ice in the frozen specimens to sublime, and means responsive to the loss of weight of the articles for automatically controlling said freeze drying means, said last-mentioned means including at least one load cell supporting the weight of said tray and the articles to be freeze dried, whereby an electrical signal may be generated which varies in accordance with the change of weight of the articles due to the sublimation of ice from the articles.

2. Apparatus in accordance with claim 1 wherein said freeze drying means includes a valved conduit through which the pressure in said chamber can be controlled, and the valve of said valved conduit being coupled to said load cell.

3. A method of freeze drying articles of substantially the same size and kind comprising the steps of freezing articles, disposing said frozen articles in a hermetically sealed enclosure, evacuating said enclosure while subjecting said articles to heat so as to cause ice in said frozen articles to sublime, and automatically continuously weighing the articles and generating a signal as a function thereof, and controlling the amount of heat supplied to said articles in repsonse to said signal.

4. A method of treating articles of substantially the same size and kind comprising freeze drying said articles in a hermetically sealed enclosure, automatically continuously weighing the articles to ascertain the change in loss of weight of the articles due to sublimation of ice therefrom and generating a signal as a function thereof, and varying said freeze drying process in response to said signal.

5. A method in accordance with claim 4 wherein said varying step includes controlling the pressure in said enclosure in response to said signal.

6. A method in accordance with claim 4 wherein said weighing step includes supporting the articles by load cells, and said step of generating a signal being accomplished by the load cells so that the signal generated will vary in accordance with the change of weight of the articles due to the sublimation of ice from the articles.

7. Apparatus for freeze drying articles of substantially the same size and kind comprising a housing having a chamber therein, a shelf within said chamber, said shelf being adapted to support frozen articles to be freeze dried within said housing, means for freeze drying the articles on said shelf by causing ice in the frozen articles to sublime, and weighing means directly responsive to the loss of weight of the articles for automatically controlling said freeze drying means.

8. Apparatus in accordance with claim 7 wherein said means responsive to the loss of weight includes a device for varying the pressure in said chamber.

9. Apparatus in accordance with claim 8 wherein said device is a valved conduit for introducing a gas into the chamber.

10. Apparatus in accordance with claim 8 wherein said device is a valved conduit through which the chamber may be evacuated.

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