Feb. 23, 1971 D. S. FRASER 3,564,727

FREEZE DRYER USING AN EXPENDABLE REFRIGERANT

Filed March 3, 1969

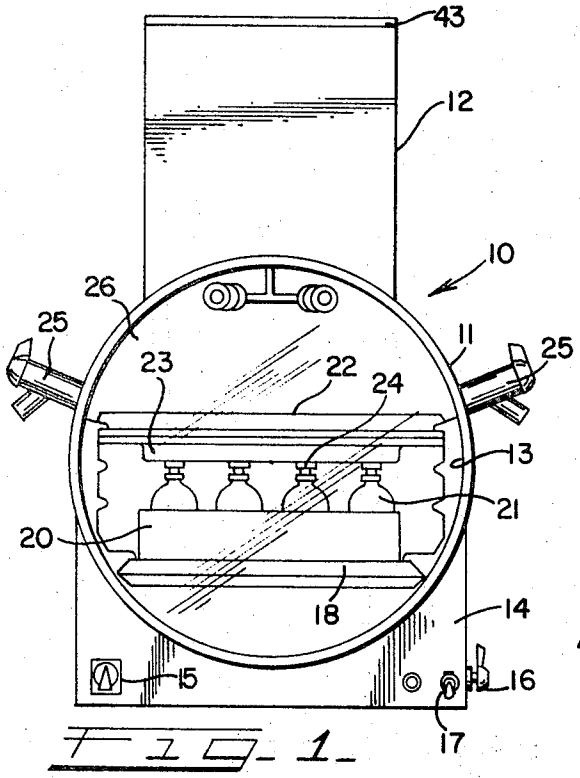


FIG. 1

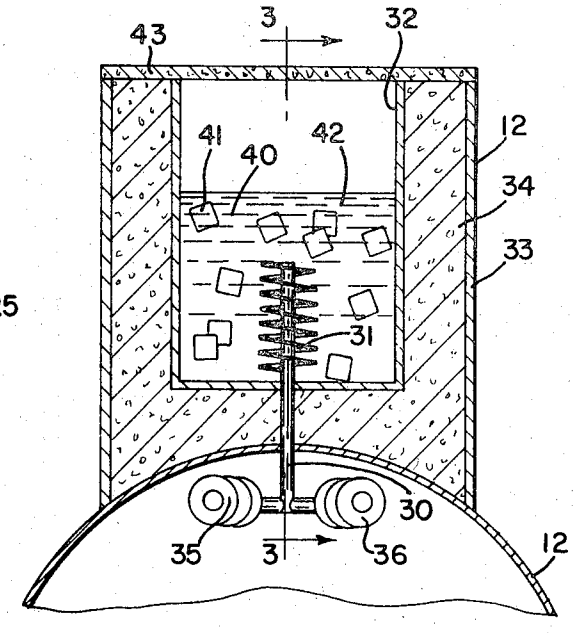


FIG. 2

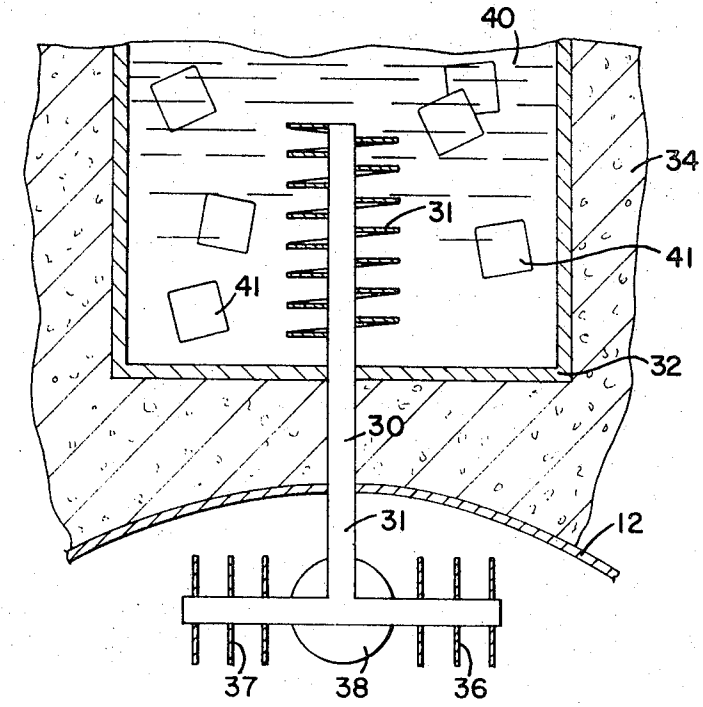


FIG. 3

INVENTOR

DOUGLAS S. FRASER

BY Robert E. Wagner ATT'Y.

United States Patent Office 3,564,727
Patented Feb. 23, 1971

3,564,727

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Douglas S. Fraser, New Paltz, N.Y., assignor to The Virtis Company, Inc., Gardiner, N.Y., a corporation of New York

Filed Mar. 3, 1969, Ser. No. 803,760
Int. Cl. F26b *13/30*
U.S. Cl. 34—92 4 Claims

ABSTRACT OF THE DISCLOSURE

A freeze drying arrangement having means to permit the use of an expendable refrigerant. A transparent door is also disclosed having a metallic layer thereon to permit viewing therethrough while reflecting radiant energy which could affect the temperature level in the chamber.

This invention relates to improvements in freeze drying chambers and, more specifically, to a new and improved refrigeration system adapted for use with an expendable refrigerant and a novel reflective door for said drying chambers.

In the practice of freeze drying, the material is first frozen and then subjected to a high vacuum and controlled heat input. Under these conditions, the frozen water content of the material is removed through sublimation which, by definition, consists of ice going to vapor, bypassing the intermediary liquid phase. In the accomplishment of freeze drying, vacuum chambers have been utilized which are cylindrical or tubular in shape. They are fabricated with a transparent or clear access door which is properly sealed to be vacuum tight. Within the vacuum chamber is provided a product shelf having means to permit it to be heated under controlled circumstances. Ideally, refrigeration should be provided to permit the prefreezing of the materials to be dried and thus eliminating the need for a separate freezing chamber. Even if freezing is done elsewhere, refrigeration should be provided to form a condensing surface to collect the water vapor and prevent it from gaining access to the vacuum pump which continuously evacuates the vacuum chamber during the freeze drying process.

In the past, chambers of this general type have been used with a mechanically operated refrigeration system. Because of the size of the refrigeration system required, the complete freeze drying unit, of necessity, was fairly sizable. Oftentimes, the space required and the cost of such a unit was beyond the space available and the budget of smaller laboratories having only an occasional need for a freeze dryer.

The present invention relates to a freeze dryer of the chamber type which may also be included with connections for manifold-type drying as well as chamber drying. The present design includes a novel refrigeration system which utilizes an expendable refrigerant located outside the freezing chamber. A novel exchange system of closed circuit design is used to transfer the heat from the expendable refrigerant to the interoir of the vacuum chamber. This system may be also utilized to maintain the products in a frozen state or freeze dry products while positioned on the shelf. The exchange system also acts as a condensing surface to collect the water vapor during freeze drying. As an additional feature, the present invention contemplates the use of a novel door which reflects heat energy while permitting viewing of the specimens in the chamber.

The present system provides a refrigeration unit of good efficiency which is considerably less expensive than the mechanical refrigeration units heretofore known. It

does not have the drawbacks of previously used techniques of positioning chunks of Dry Ice in receptacles adjacent the vacuum chamber.

The refrigeration unit also serves multiple functions, freezing and condensing out the water vapors. All of the advantages of more expensive systems are gained without a significant sacrifice in refrigeration efficiency. Other advantages will be seen upon description of a preferred form of the invention.

It is an object of this invention to provide a new and improved freeze drying chamber having a novel door permitting viewing of the contents while reflecting any heat energy which ordinarily would pass through said door.

It is a further object of this invention to provide a new and improved freeze drying arrangement having a novel refrigeration system which will be relatively inexpensive to manufacture and assemble, thereby placing it within the reach of users having only occasional need for freeze drying equipment.

It is a further object of this invention to provide a new and improved freeze drying arrangement having a novel refrigeration system which utilizes an expendable refrigerant.

It is a still further object of this invention to provide a new and improved refrigeration system for use with freeze drying equipment which may be readily manufactured and assembled.

Objects other than those specifically set forth will become apparent to the person skilled in the art upon reference to the accompanying drawings and following description.

In the drawings:

FIG. 1 is a front elevational view of the freeze drying apparatus of the present invention;

FIG. 2 is an enlarged cross-sectional view of the upper portion of the freeze drying chamber shown fragmentarily and the refrigeration system shown in cross section; and

FIG. 3 is a further enlarged cross-sectional view taken generally along the line **3—3** of FIG 2.

Referring now to FIG. 1, a freeze drying arrangement of the present invention is indicated generally by reference character **10**. The freeze drying arrangement includes a vacuum chamber **11** and refrigeration system **12** which is disposed on top of the vacuum chamber **11**.

The vacuum chamber **11** consists of a cylindrical drum **13** supported on a housing **14**. The housing **14** may include suitable controls such as a temperature control **15**, stoppering control **16** and shelf heat switch **17**, all of which function in a known manner.

The chamber **11** includes a bottom shelf **18** on which is positioned a tray **20** having bottles **21** of material to be freeze dried. A stoppering plate **22** is located above the bottles **21** and is provided on the underside with an elastomeric diaphragm **23** which, through the use of the stoppering control, expands downwardly to seat stoppers **24** in the mouth of the bottles or containers **21** after freeze drying is completed. This is a conventional or known type of stoppering which is known to the art.

Manifold units in the form of valves **25** may be provided along opposite sides of the vacuum chamber **11**. Each valve may be of the type described in co-pending application Ser. No. 650,134, assigned to the assignee of the present invention. The disclosure of that application is incorporated herein by reference. The front of the chamber is closed off by a transparent door **26** of plastic having vacuum metal deposited thereon. This permits the operator to observe the freeze drying process while reflecting heat from outside sources and maintaining the heat level in the chamber fairly constant. External heat is reflected while full viewing of the specimens being dried is available. The total reflectance should be of

the order of about 75%. The chamber **11** is provided with suitable connections (not shown) for attachment to a vacuum pump capable of evacuating the interior of the chamber **11**. Ideally, the vacuum connection is located where the flow of vapor will be past the condensing surface in the chamber.

The expendable refrigeration unit **12** is of box-like form and disposed on the top of the vacuum chamber **11**. As seen in FIGS. 2 and 3, the refrigeration unit includes a closed tube **30** having heat exchange means **31** located centrally of a receptacle **32** which is located within a housing **33**. An insulating foam **34** is formed in the space between the receptacle **32** and the housing **33**, both of which may be rectangular as shown or of any other suitable shape. The closed tube **30** extends within the vacuum chamber **12** and communicates with a generally X-shaped condenser having a plurality of condensing fins **35, 36, 37** and **38** at the respective ends. Obviously, the condenser may be of any desired configuration. The heat exchange surfaces **31, 35, 36, 37** and **38** are formed by a plurality of metallic fins which are joined to the closed tube **30** by suitable means such as soldering, welding or the like to provide for optimum heat transfer.

The heat exchange means **31** is surrounded in the well **32** by an expendable refrigerant **40** which may include Dry Ice chunks **41** immersed in an alcohol or acetone bath **42**. Inasmuch as the well **32** is well insulated from the outer housing **33** by a layer of foam several inches thick and covered by a suitable cover **43**, the expendable refrigerant will last for approximately 24 hours.

The tube **30** forms a closed refrigeration circuit and is filled with a suitable refrigerant such as Freon 13 which boils at about —115° F. Gases which would serve as a refrigerant in this system are nitrous oxide, refrigerant 503, ethane ethylene and possibly others. The pressure range would vary between about 200 p.s.i. to 700 p.s.i., depending upon the refrigerant.

The refrigeration cycle which occurs is as follows: As the refrigerant is cooled at the upper end of the tube **30** by the expendable refrigerant **40**, it goes to a liquid state, draining down to the bottom of the tube and into the condensing area of the heat exchangers **35–38**. As the heat exchange means in the chamber **12** is warmed through loss of heat during the freeze drying or refrigeration of the material to be freeze dried, the refrigerant is heated to above its boiling point and assumes a gas form, rising in the tube **30** to the area of the heat exchange means **31**. There the refrigerant is again cooled and the cycle repeats itself.

It is contemplated that the refrigerant will be maintained in the tube **30** under a pressure. As indicated, this will vary with the type of refrigerant used, however, in the preferred form, when a refrigerant known as Freon 13 is utilized, it is under a pressure of the order of 300 p.s.i.

It can be appreciated from consideration of the foregoing embodiment that the freeze drying system of the present invention is complete with the exception of a suitable vacuum source. Because of its simplified construction, it may be produced economically to place it within the economic range of small laboratories. With the refrigeration system shown, it may be used as a freezer to freeze the materials to be dried and thereafter used as a condenser during the freeze drying process. Because of the

novel construction of the refrigeration system, it will operate efficiently and utilize a minimum of Dry Ice. Heat is transferred in the present invention by latent heat of liquid used, permitting large amounts of heat to be transferred through a relatively small tube which, in one embodiment, had an outside diameter of five-eighths (⅝) inch. The location of the condenser permits the temperature to be maintained until the last fragment of Dry Ice has been expended. The reflective door prevents heat loss or gain by radiant heating exterior of the chamber while permitting a full view of the chamber contents during drying.

Upon a consideration of the foregoing, it will become obvious to those skilled in the art that various modifications may be made without departing from the invention embodied herein. Therefore, only such limitations should be imposed as are indicated by the spirit and scope of the appended claims.

I claim:

1. A freeze drying unit comprising a closed vacuum chamber adapted to be evacuated, condensing and refrigeration means for cooling said chamber and condensing vapors during freeze drying, said refrigeration and condensing means including a housing for a receptacle, said receptacle being located adjacent said vacuum chamber and adapted to receive an expendable refrigerant, a closed tube forming a refrigeration transfer medium, said closed tube extending from said receptacle into said vacuum chamber, a first heat exchange means formed on said closed tube within said chamber, a second heat exchange means on said tube in said receptacle and adapted for directly contacting said expendable refrigerant and a pressurized gas in said closed tube to maintain said first heat exchange means in said chamber at a temperature below the temperature required to condense the vapor out of the materials being dried.

2. The freeze drying unit of claim **1** wherein said refrigerant in said closed tube is under a pressure in the range from about 200 pounds per square inch to about 700 pounds per square inch.

3. In a chamber-type freeze dryer having a door thereon to gain access to said chamber, while maintaining said chamber vacuum-tight, the improvement comprising said door having a metallic substance thereon to reflect heat energy on both sides thereof while permitting viewing therethrough.

4. The improvement in chamber-type freeze dryers as defined in claim **3** wherein said door is about 75% reflective.

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U.S. Cl. X.R.

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