

[54] **LABORATORY FREEZE DRYER**

[76] Inventor: **Samuel M. Broadwin**, 60 East 8th St., New York, N.Y. 10003

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[58] Field of Search **34/92; 62/165, 166, 384**

[56] **References Cited**

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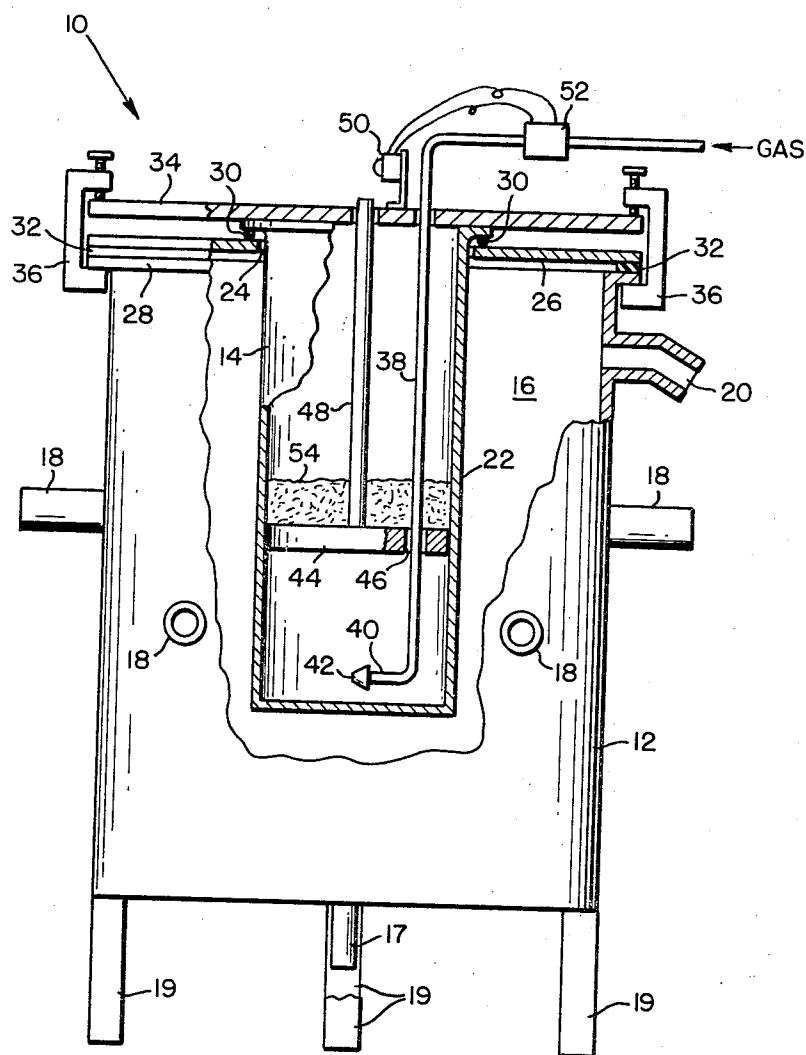
Primary Examiner—John J. Camby

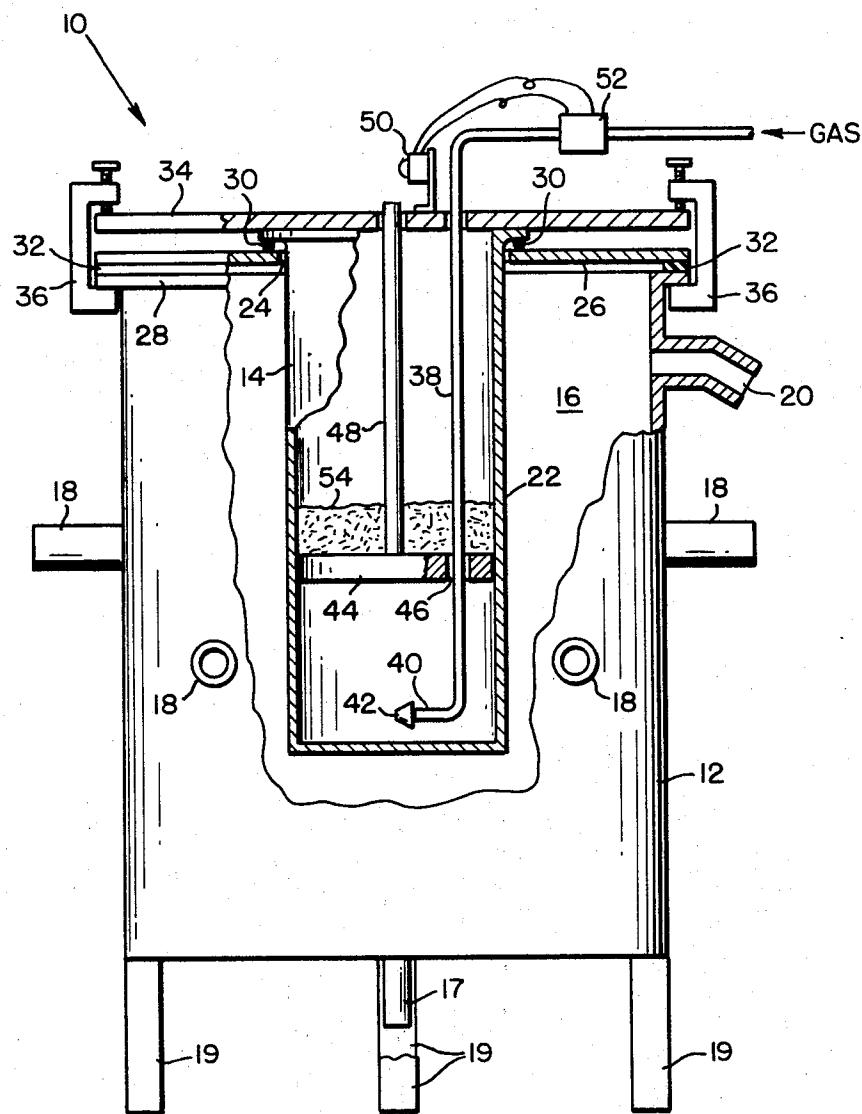
Attorney, Agent, or Firm—Theodore B. Roessel; Roger Aceto

[57] **ABSTRACT**

A vacuum trap useful as a laboratory freeze dryer in which the condensing surface is formed by an aluminum container. The surface is cooled by the snow formed when high pressure carbon dioxide gas is expanded into the container. A piston in the container rises and falls in response to the level of the snow and activates a switch to stop and start respectively the introduction of gas into the container.

9 Claims, 1 Drawing Figure





LABORATORY FREEZE DRYER**BACKGROUND OF THE INVENTION**

The present invention relates generally to freeze dryers and more specifically to a relatively small, compact freeze dryer suitable for laboratory use.

Freeze dryers are well-known in the art and are utilized to dehydrate various materials without destroying the physical characteristics of the material. Briefly, freeze dryers utilize a vacuum trap to subject the material to be dried to a vacuum. External mild heat is then applied to the material so that vapor pressure causes the removal of moisture by sublimation from the material; the moisture thereafter condensing and collecting on a super cooled condensing surface which may or may not be corrugated. In laboratory work, freeze dryers may be used to desiccate such items as blood plasma and animal tissue or other heat sensitive products.

While large commercial freeze dryers have automatic refrigeration systems for cooling the condensing surface, laboratory freeze dryers are usually manually operated. Laboratory freeze dryers of the prior art are typically constructed as double shell structures, wherein the space between the shells can be evacuated. The inner shell forms a reservoir which is filled with dry ice or a mixture of dry ice and acetone. This cools the shell wall which forms the condensing surface. As the dry ice sublimates and the condensing surface warms, more dry ice is manually placed into the inner shell as necessary to keep the condensing surface super cooled. Such devices, then, required some sort of refrigerated storage for the dry ice needed to fill the reservoir of the freeze dryer. Also, repeated inspections were required to insure that the proper amount of dry ice was contained in the freeze dryer and, of course, any dry ice lost through sublimation had to be manually replaced.

The present invention overcomes these drawbacks of the prior art by providing a laboratory freeze dryer in which the cooling medium is a snow formed by the expansion of a compressed gas such as carbon dioxide. The freeze dryer of the present invention also provides an automatic regulating means for maintaining the snow at a relatively constant level to insure that the condensing surface remains super cooled throughout the freeze drying process.

SUMMARY OF THE INVENTION

The present invention may be characterized in one aspect thereof by the provision of a freeze dryer for laboratory use formed with inner and outer shells arranged so that the space therebetween can be evacuated, the inner shell being metallic and providing a condensing surface; valved means for introducing a suitable pressurized gas such as carbon dioxide into the inner shell adjacent the bottom thereof where the gas expands to form a snow for super cooling the condensing surface; and control means which acts responsive to the level of snow in the inner shell to open and close the valve means, thereby regulating the amount of gas introduced into the inner shell.

OBJECTS OF THE INVENTION

One object of the present invention is to provide a laboratory freeze dryer, wherein the snow formed by the expansion of pressurized gas provides the cooling medium.

Another object of the present invention is to provide a laboratory freeze dryer of the type described having means to automatically regulate the amount of snow which is formed.

A further object of the present invention is to provide a freeze dryer of the type described, having means acting responsive to the level of the snow for controlling the introduction of the pressurized gas.

These and other objects, advantages and characterizing features of the present invention will become more apparent upon consideration of the following detailed description thereof when taken in connection with the accompanying drawings depicting the same.

DESCRIPTION OF THE DRAWINGS

The sole FIGURE is a elevation view partly broken away and in section schematically representing the freeze dryer of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to the drawings, the FIGURE shows the freeze dryer of the present invention generally indicated at 10 to include an outer shell 12 and an inner shell 14. The outer and inner shells form a space 16 therebetween which can be evacuated. For this purpose, outer shell 12 is provided with a plurality of ports 18 in a conventional manner which may be attached to any suitable vacuum source not shown. The outer shell is also provided with a fitting 20 to which can be attached in a conventional manner, a container (not shown) of the goods which are to be freeze dried. The outer shell also has a drain 17 and is supported on legs 19.

Inner shell 14 is preferably made of a metallic material having good thermal conducting characteristics, such as stainless steel or aluminum. This inner shell acts as a reservoir for containing the cooling media and the outer surface 22 of the shell defines a condensing surface on which the vapors drawn from the material to be freeze dried are condensed and frozen.

The inner shell is suspended in shell 12 through an opening 24 in a plate 26. Plate 26 merely rests on the upper edge 28 of the outer shell and suitable gasket means 30 and 32 between the plate and inner shell and the plate and outer shell, respectively, provide a vacuum tight seal when space 16 is evacuated. Plate 26 and inner shell 14 can be clamped to the top edge of the outer shell by means of a cover 34 and C clamps 36 as shown.

Extending down through cover 34 and into inner shell 14 is a valved gas inlet line 38. One end (not shown) of the gas line is connected to a source of pressurized gas such as a tank of carbon dioxide. The other end 40, of the inlet line, terminates in a nozzle 42 adjacent the bottom of inner shell 14. Positioned above this nozzle is a piston 44 having a bore at 46 permitting the passage of gas line 38. A piston rod 48 extends up through cover 34 to a point adjacent a micro switch 50 mounted on the cover. Micro switch 50 is in turn operatively connected to a valve 52 located in the gas line.

Completing the structure of the freeze dryer is a disc or pad of foam rubber material 54. While the FIGURE shows that this pad is carried on the piston, it may be located and attached instead either to the under surface of the piston or as an annular piece surrounding

the piston. The purpose of this material is set out herein below.

An operation, then, a container (not shown) containing the object or material to be dehydrated by freeze drying is attached to fitting 20. Space 16 is then evacuated by any suitable means such as a vacuum pump (also not shown) communicating with ports 18. The vacuum pump operates continuously in order to maintain the vacuum in space 16 as vapors are drawn from the article being freeze dried.

The introduction of gas under pressure through line 38 is then started. As the gas is discharged from nozzle 42 and into shell 14, the gas expands to form a snow which fills the space beneath the piston. The piston should rise slightly responsive to the initial increase in pressure in shell 14 and then full again. While gas can escape through the porous structure of pad 54, the pad acts as a seal to prevent the solid particles of snow from escaping around piston 44 and out of the inner shell. As more gas is introduced and more snow formed, the level of the snow rises which causes the piston to rise. When the piston has risen a predetermined amount, rod 48 will contact switch 50 to terminate the flow of gas. The snow, which fills the volume of shell 14 beneath piston 44, super cools the shell wall so that the outer surface 22 of the shell acts as a condenser. Water vapor drawn from the article to be freeze dried contacts this super cooled condensing surface and forms ice crystals which collect on the outer surface 22.

As the dry ice snow within the inner shell sublimates, gaseous carbon dioxide escapes by passing upwardly through and around the porous structure of pad 54. Sublimating carbon dioxide will reduce the snow level and therefore, the level of piston 44 will drop within the inner shell. This eventually carries rod 48 out of engagement with switch 50 so that valve 52 is again opened to permit more gas to be introduced into the inner shell. In this manner, the level carbon dioxide snow within shell 14 is maintained substantially constant in order to maintain condensing surface 22 super cooled.

Thus, it should be appreciated that the present invention accomplishes its intended objects in providing a freeze dryer for laboratory use which is simply operated and in which cooling of the condensing surface is accomplished by the expansion of a gas under pressure within the condenser. The expanding gas forms a snow which cools the condenser walls. Furthermore, a simple mechanism is provided for controlling the introduction of gas into the condenser responsive to the level of the snow which is formed, so that the condensing surface is maintained at a super cooled temperature throughout the freeze drying cycle of operation.

Having thus, described the invention in detail, what is claimed as new is:

1. Vacuum trap apparatus useful as a laboratory freeze dryer comprising:

- a. inner and outer shells forming a closed spaced therebetween which can be evacuated, said inner shell being metallic and providing a condensing surface on which vapors from the goods being freeze dried are condensed;
- b. valved means for introducing a gas under pressure into said inner shell adjacent the bottom thereof,

said gas expanding in said inner shell to form a snow for cooling the walls of said inner shell, and
c. control means acting to open and close said valve means responsive to the level of snow in said inner shell respectively falling below and reaching a predetermined level for controlling the introduction of said gas into said inner shell and maintaining the level of snow at substantially said predetermined level.

2. Apparatus as set forth in claim 1 wherein said valved means includes:

- a. a gas line having one end extending down into said inner shell and terminating in an outlet nozzle adjacent the bottom thereof, the other end of said line being connectable to a source of pressurized gas; and
- b. a flow control valve intermediate said ends.

3. Apparatus as set forth in claim 1 wherein said valved means includes a gas line having an outlet opening into said inner shell adjacent the bottom thereof, said control means including a piston in said inner shell disposed above said gas line outlet, said piston moving responsive to the level of snow in said inner shell to open and close said valved means.

4. Apparatus as set forth in claim 3 including a pad of porous material disposed above said piston to seal against the passage of snow while permitting the passage of sublimating gas.

5. Apparatus as set forth in claim 4 wherein said porous material rests on and moves with said piston.

6. Apparatus as in claim 3 including a micro switch operatively connected to said piston and valved means wherein movement of said piston beyond predetermined limits opens and closes said switch to operate said valved means.

7. Apparatus as in claim 6 including a cover for said inner shell, said switch being mounted on said cover.

8. Apparatus as in claim 7 including a rod upstanding from said piston, said rod adapted to pass through an opening in said cover responsive to movement of said piston for operating said switch.

9. In a laboratory freeze dryer comprising an inner and an outer shell adapted to be joined so that the space therebetween can be evacuated, the inner shell forming a reservoir for the containment of a cooling medium and presenting a super cooled condensing surface to said space, the improvement comprising:

- a. valved means for introducing pressurized carbon dioxide into the reservoir formed by said inner shell, the expansion of said pressurized carbon dioxide in said inner shell forming a snow therein to cool said condensing surface;
- b. a piston in said inner shell, said piston rising and falling in said inner shell responsive to the level of carbon dioxide snow therein; and
- c. control means operatively connected to said piston and valved means and operating to close and open said valved means in response to movement of said piston between predetermined high and low level limits respectively, for maintaining the level of said snow at a substantially constant level between said limits.

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