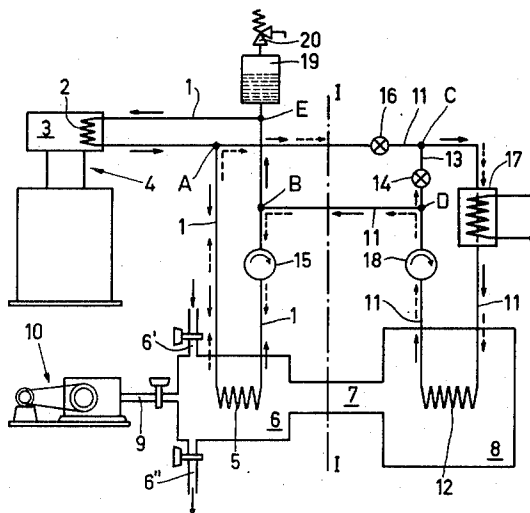


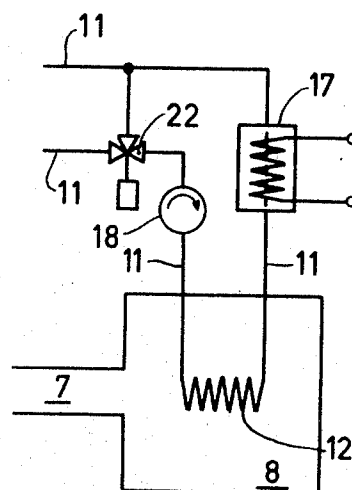
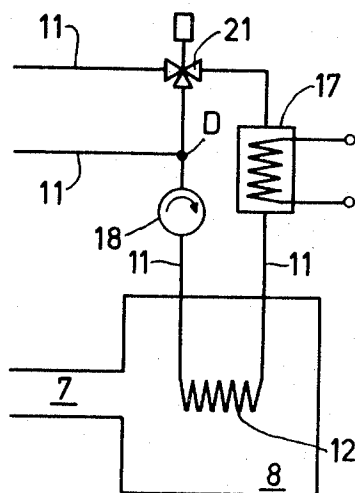
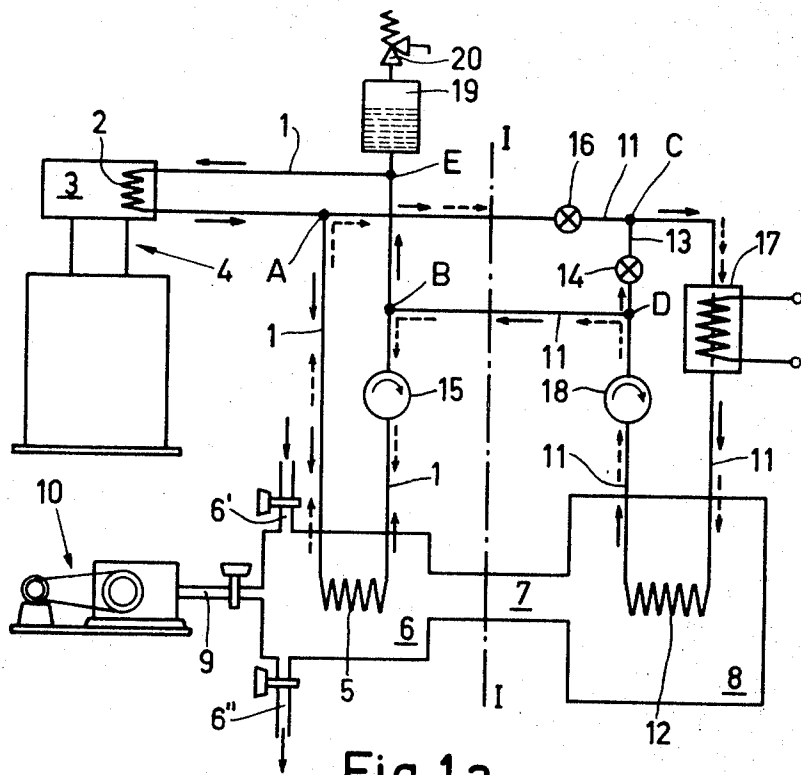
Van Dijk

[45] **Apr. 18, 1972**

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The invention provides a freeze-dryer of a simple construction in which the freeze-drying temperature of the vacuum chamber and the temperature of the condenser chamber which is at a lower level are adjusted by means of a liquid conducted along the cold head of a cold-gas refrigerator. This liquid is also advantageously used for defrosting the ice mass formed in the condenser chamber in a very short period of time, after the freeze-drying process.





INVENTOR.
NICOLAAS A.VAN DIJK

BY

Frank R. [Signature]
AGENT

FREEZE-DRYER

The invention relates to a freeze-dryer comprising at least one vacuum freeze-drying chamber communicating with a condenser chamber in which a lower temperature prevails during operation than in the vacuum chamber; a cold-producing device for supplying cold to the chambers, including a heating device is present for controlling the operating temperature of the vacuum chamber.

In a known freeze-dryer the cold producing device is formed by an evaporation-condensation refrigerator in which a gas is compressed to a high pressure by one or more compressors, is then condensed in a condenser and expanded in one or more throttle devices. On the one hand, liquid medium is supplied to a vaporizer arranged in the condenser chamber where it evaporates by the withdrawal of heat from the condense chamber and flows back to the compressor. On the other hand, liquid medium is conducted to a second vaporizer which is in a heat exchanging contact with a liquid present in a closed system of ducts, supplies its cold to said liquid and flows back to the compressor again in the gaseous phase. By means of a pumping device incorporated in the closed system of ducts, the liquid can circulate and again supply cold taken up from the second vaporizer in one part of the system of ducts, to the vacuum chamber which is in heat exchanging contact with another part of the system of ducts. Furthermore a heating device is present in the closed system of ducts by means of which the temperature of the cooled liquid and hence the temperature of the vacuum chamber can be controlled. The vacuum chamber is evacuated by means of a vacuum pumping device communicating with the condenser chamber.

The operation of this known freeze dryer is further described as follows: aqueous preparations which have been frozen at -50°C ., for example outside the freeze-dryer, are placed in the vacuum chamber which has previously been cooled, to -30°C ., for example. The vacuum chamber is then evacuated and it is ensured, by supplying thermal energy to the vacuum chamber via the liquid in the closed system of ducts, that the ice present in the preparations sublimates to water vapor. This water vapor is drawn off to the condenser chamber cooled at a lower temperature, condenses therein and freezes to the cold surface of the condenser chamber.

After a few batches of preparations have thus been freeze-dried, a large quantity of ice has deposited in the condenser chamber which necessitates the defrosting of this chamber before the freeze-drying process can be repeated. The cooling of the condenser chamber generally occurs to temperatures of approximately -80° while the temperature of the vacuum chamber is controlled from $+30^{\circ}\text{C}$. to -30° to -40°C .

This known freeze dryer has a few drawbacks. The dryer is comparatively complicated, which is due to the fact, that in order to be able to reach cooling temperatures to -80°C ., at least one low-pressure compressor and one high pressure compressor are required to obtain a high compression/expansion pressure ratio of the gas. An additional difficulty is that special precautions are necessary to return lubricating oil originating from the compressors and taken along by the compressed gas to said compressors.

Furthermore this freeze-dryer is complicated due to the fact that a separate liquid-filled (for example, glycol) system of auxiliary ducts is necessary to be able to control the temperature of the vacuum chamber. Owing to the above-mentioned temperature requirements, only a small number of gases is to be considered for use in the evaporation-condensation refrigerator. The choice is furthermore restricted by the properties of the compressors which in connection with their dimensions require a given minimum suction pressure. The sucked gas originates from the vaporizers in which the liquefied gas evaporates. It is also desirable that the refrigerator condenser can be cooled with cooling water which means a further restriction in the choice of the gas.

A further drawback is that the gases which can be used, for example, the medium known as Freon 22, have a vapour pressure which is lower than 1 atm. at comparatively low tempera-

tures. This means that, since the gas pressure in the system of ducts is hence lower than the atmospheric pressure, air can easily leak in with all the detrimental consequences of this (reduction in efficiency, pollution, corrosion).

In addition, with the known freeze dryer the time necessary to defrost the ice formed in the condenser chamber is very long. This is caused by the fact that the defrosting should be carried out at comparatively low temperatures since otherwise the gas pressure in the system of ducts would become inadmissibly high. In practice the defrosting is carried out by means of water of, for example, 40°C . The condition, whereby the water can transfer its heat only to the outside of the ice mass (the ice surrounds the vaporizer arranged in the condenser chamber for the greater part) results in a further unfavorable influencing of the defrosting time.

It is the object of the invention to provide a freeze-dryer of a considerably simplified construction with which the said drawbacks are avoided. For that purpose, the freeze-dryer according to the invention is characterized in that the cold-producing device is formed by a cold gas refrigerator, a first closed system of ducts being present which comprises a first heat exchanger for exchanging heat with the cold-gas refrigerator, and also comprises a second heat exchanger arranged in the condenser chamber, a liquid and a first pumping device being present in said system, a system of ducts being furthermore present which communicates on either side of the first heat exchanger with the first system of ducts and in which a third heat exchanger is present arranged in the vacuum chamber, the same liquid being present in the second system of ducts and a bypass duct being present which communicates on either side of the third heat-exchanger with the second system of ducts, a second pumping device being present which, together with the heating device, is incorporated in one part of the second system of ducts which is situated between the two connection places of the bypass duct with said system and which part also comprises the third heat exchanger, one or more control cocks being present in the second system of ducts and the bypass duct, respectively, for controlling the flow of liquid through said system and said duct, respectively, the first system of ducts being furthermore constructed so that in a position of the said control cocks in which the bypass duct is substantially entirely closes and when only the second pumping device is in operation, the liquid mainly flows through the second system of ducts and the part of the first system of ducts which is situated between the connection places of the second system of ducts hereto and which part comprises the second heat-exchanger.

In this manner a freeze-dryer is obtained which presents great advantage as compared with the known freeze-dryer.

By using the cold-gas refrigerator in combination with liquid in the systems of ducts it is possible to bring both the vacuum chamber and the condenser chamber each at their desirable temperatures in a simple, rapid and reliable manner.

For circulating the liquid in the system of the ducts, simple circulating pumps will do. The systems of ducts and heat exchangers cannot be polluted by lubricating oil, since the lubricating oil circuit forms no part of the independently operating cold-gas refrigerator. By using liquid in the systems of ducts, the danger of air leak is no longer present and a good heat transfer is ensured between said liquid and the cold and warm places respectively, along which it is conducted.

Whereas in the known device a separate system of auxiliary ducts with liquid is necessary which can be used only for controlling the temperature of the vacuum chamber by heating the liquid circulating in said system, the liquid present in the freeze-dryer according to the invention can also be used for defrosting the condenser chamber. By conducting the liquid, after heating in the second system of ducts, through the second-heat-exchanger, a considerable reduction of the defrosting time of the ice deposited in the condenser chamber is obtained. Actually, the temperature of the liquid, suitably chosen for that purpose, is the systems of ducts may now be high without the danger of too high a pressure in the ducts.

The ice mass is now defrosted from within. In addition it is possible to use water of likewise a high temperature to defrost the ice mass again from the outside.

By choosing a second heat-exchanger which has a considerably lower resistance to flow than the first heat exchanger, or by closing, for example, cocks incorporated in the two sections of the first system of ducts situated between the first heat-exchanger and the connection places of the second system of ducts, it is achieved that upon defrosting the condenser chamber, when the warm flow of liquid is circulated by the second pumping device, said flow of liquid flows substantially entirely or entirely through the second heat exchanger or does substantially not flow or does not flow at all through the first heat exchanger. The overall flow of liquid hence is available for the defrosting process which owing to a maximum heat transport through the liquid, is favorable for a rapid defrosting. Since warm liquid does not pass the cold place of the cold-gas refrigerator, the additional advantage is obtained that unnecessary heating of the cold-gas refrigerator is prevented. So during the defrosting period the freeze-dryer obtains its low operating temperature and remains ready for operation.

In a favorable embodiment of the freeze-dryer according to the invention at least one of the systems of ducts communicates with a liquid expansion container in which a gaseous medium is present above the liquid under such a pressure that, at any operating temperature of the liquid, boiling of said liquid is prevented. As a result of this strong evaporation of an formation of vapor bubbles in the liquid in the systems of ducts is prevented, so that a good heat transfer between the liquid and the heat exchangers remains ensured. By choosing the pressure of the gaseous medium in the expansion container to be sufficiently high, it is possible to produce an increase of the boiling point of the liquid which is of advantage upon defrosting the condenser chamber. The liquid can then be heated to an extra high temperature by the heating device without the danger of boiling phenomena occurring.

A further favorable embodiment of the freeze-dryer according to the invention in which the operating temperature of the vacuum chamber is controlled within the range of from approximately -30° to $+4^{\circ}$ C. and the operating temperature of the condenser chamber is approximately -80° C. is characterized in that the liquid is R11 (trichloromonofluoromethane- CCl_3F). The use of this known liquid presents various advantages. First of all the viscosity of said liquid (boiling point 23.8° C.) in the operative temperature range is rather constant. This means that in the temperature range considered a good heat transfer is maintained between the liquid and the heat exchangers. Actually, the danger that the normally turbulent flow of liquid in the systems of ducts decreases at comparatively low temperatures of the liquid owing to the increased viscosity and changes into a laminar flow is absent here.

Owing to the comparatively low viscosity a small pumping action is sufficient while the pumps are always subjected to a substantially constant load as a result of the fact that the viscosity remains substantially constant in the active temperature range. All this enables the use of small, comparatively cheap pumps in which the constant load favorably influences the lifetime.

The use of R11 also makes it possible to heat the liquid to comparatively high temperatures upon defrosting the moisture receiving chamber while at the same time the pressure in the expansion container and the systems of ducts can be comparatively low so that thin-walled and low-weight ducts can be used. It is possible, for example, to heat R11, at a pressure of 2.5 atmospheres in the systems of ducts, to 60° C. without the danger of boiling occurring.

BRIEF DESCRIPTION OF THE DRAWING

In order that the invention may be readily carried into effect, it will now be described in greater detail, by way of exam-

ple, with reference to the accompanying diagrammatic drawing.

FIG. 1a shows not to scale an embodiment of the freeze dryer.

FIGS 1b and 1c shows a slightly different embodiment of the part of the freeze-dryer shown in FIG. 1a on the right hand side of the lines I—I.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Reference numeral 1 in FIG. 1a denotes a first closed system of ducts in which a heat-exchanger 2 is incorporated which is in a heat exchanging contact with the cold head 3 of a cold-gas refrigerator 4, and in which a heat exchanger 5 is arranged in a condense chamber 6. The condenser chamber communicates via a duct 7 with a vacuum freeze-drying chamber 8, and communicates via a duct 9 with a vacuum pumping device 10. It furthermore comprises a water inlet 6' and a water outlet 6''. At the places A and B, a second system of ducts 11 communicates with the first system of ducts 1; the second system of ducts a heat exchanger 12 is incorporated which is arranged in the vacuum freeze-drying chamber 8.

At the places C and D a bypass duct 13 with controllable cock 14 communicates with the second system of ducts 11. A pumping device 15 is present in the first system of ducts 1, while in the second system of ducts a controllable cock 16, a heating device 17 and a pumping device 18 are incorporated. At the place E an expansion container 19 comprising a valve 20 communicates with the first system of ducts 1.

The systems of ducts are filled with a liquid which may be, for example, R11. Above the liquid in the expansion container 19 a gas under pressure, for example, nitrogen, is present. The heat exchanger 2 has a resistance to flow which is considerably larger than that of the heat exchanger 5.

The operation of this device is as follows: Cooling of the condenser chamber 6 takes place by circulating liquid in the system of ducts 1 by means of the pumping device 15 in the direction denoted by the solid-line arrows. Cold absorbed by the liquid at the cold head 3 of the refrigerator 4 in the heat exchanger 2 is delivered again to the condenser chamber in the heat exchanger 5. For cooling the vacuum freeze-drying chamber 8, a part of the flow of liquid which is conducted along the cold-gas refrigerator is tapped at A and conducted, by the pumping device 18, through the second system of ducts 11 and heat exchanger 12 in the direction likewise denoted by the solid-line arrows. In this heat exchanger 12 the liquid supplies its cold to the vacuum freeze-drying chamber 8. During cooling of the vacuum freeze-drying chamber 8, the heating device 17 is not in operation and the cock 14 is fully or partly opened. A part of the flow of liquid originating from the heat exchanger 12 will then enter the bypass duct 13 at the point D and mix at the point c with the cold flow of liquid passed by the cock 16 so that there a mixed temperature of the liquid is obtained. The remaining part of the flow of liquid originating from the heat exchanger 12, flows to B and is mixed there with the flow of liquid originating from the heat exchanger 5. By controlling the passage of the cocks 16 and 14, respectively, the vacuum freeze-drying chamber can be brought to the desirable temperature, a temperature which is higher than that so which the condenser chamber is adjusted. If it is desired that much cold be supplied to the heat exchanger 12, the cock 14 can be closed so that the overall quantity of liquid displaced by the pumping device 18 flows to point B and then to the heat exchanger 2.

After the vacuum freeze-drying chamber 8 and the condenser chamber 6 have each reached their operating temperatures, said chambers are evacuated by means of the vacuum pump device connected thereto and the actual freeze-drying of the preparations can take place. For that purpose, the heating device 17 is switched on and supplies as much thermal energy to the flow of liquid flowing to the heat exchanger 12 as is necessary to sublime the ice present in the preparations in the vacuum freeze-drying chamber to water vapor, which

water vapor condenses and freezes in the condenser chamber 6 kept at a lower temperature. After a few batches of preparations have thus been freeze-dried, a large quantity of ice has deposited in the condenser chamber, mainly on the outside of the heat exchanger 5. The defrosting of the condenser chamber can now be effected in a simple and rapid manner as follows: the pumping device 15 is switched off. The cock 14 is closed and the cock 16 is fully opened. The liquid which is heated to a comparatively high temperature by the heating device 17 is now circulated by the pumping device 18, along the path denoted by the broken-line arrows. The warm liquid supplies its thermal energy in the heat exchanger 5 to the ice in the condenser chamber. As compared with the process during the cooling, there are two striking differences. First of all, the flow of liquid during the heating process flows through the pumping device 15 and the heat exchanger 5 in a direction which is opposite to that during the cooling process. By the use of circulation pumps this is no problem. Secondly, the flow of liquid substantially does not flow through or does not flow at all through the heat exchanger 2. This is caused by the fact already mentioned above that the resistance to flow of the heat exchanger 2 is considerably larger than that of the heat exchanger 5 so that the flow of liquid chooses the easiest way through the last-mentioned heat exchanger. Of course it is also possible to prevent the flow of liquid through the heat exchanger 2 by means of cocks incorporated in the sections of the ducts situated between the heat exchanger 2 and the points A and E.

Since substantially little or no liquid flows through the first heat exchanger, the whole flow of liquid is available for the heat transport from the heating device 17 to the condenser chamber 6 which is in favor of a rapid defrosting. The liquid is heated to a comparatively high temperature, as gas under pressure for example, nitrogen, present in the expansion container 19 above the liquid ensuring that the liquid does not start boiling. When the operating temperature of the vacuum chamber is approximately in the temperature range of from -30° to $+30^{\circ}$ C., and when the operating temperating temperature of the condenser chamber is from -70° to -80° C. the liquid, when R11 is used, can be heated without boiling to 60° C. at a pressure of 2.5 atmospheres in the expansion container and the systems of ducts. During normal operation of the freeze-dryer, the pressure may vary between 1 and 1.5 atmospheres. Should, during operation, the pressure in the system of ducts increase too strongly by some cause, the valve 20 opens. Since a large flow of liquid of high temperature is conducted through the heat exchanger 5, the ice mass

deposited on it is rapidly defrosted from within. In addition, said mass can be defrosted from without by conducting water of a high temperature in the condenser chamber via the inlet 6'. Melting water can flow away out of the condenser chamber via the outlet 6''. During the defrosting process the warm liquid does not pass the cold place of the cold-gas refrigerator, so the cold supplied by said machine is not lost. Whereas in FIG. 1a two cocks 16 and 14 are present in the duct 11 and the bypass duct 13, respectively, FIGS. 1b and 1c show that it is also possible to use a mixing valve 21 and a distributing valve 22, respectively. For the rest the operation remains equal to that of the freeze-dryer shown in FIG. 1a. It will be obvious from the above that the invention provides a simple and readily operating freeze-dryer.

What is claimed is:

1. In a freeze-dryer apparatus including a vacuum freeze-drying chamber and a condensor chamber and vacuum pump means in communication with both chambers, and a cold-gas refrigerator for producing cold, the apparatus further comprising first second, and third heat-exchanger means for heat exchange respectively with said refrigerator, the condensor chamber, and the freeze-drying chamber, first closed duct system which includes said first and second heat exchangers and first pump means, second closed duct system which includes said third heat exchanger and a portion of the first duct system, second pump means, and heating means, liquid circulated in both of said duct systems by said corresponding pump means, a by-pass duct in said second duct system for bypassing said portion of the first duct system within said second duct system, control means for controlling liquid flow in said first and second duct systems and in said by-pass duct, whereby said circulated liquid cools said chambers and said liquid when heated in the second duct system also defrosts ice formed in the condensor chamber.

2. A freeze-dryer according to claim 1, further comprising a liquid expansion container, in which a gaseous medium is present above the liquid under such a pressure that, at any operating temperature of the liquid, boiling of said liquid is prevented, and at least one of said duct systems communicates with said container.

3. A freeze-dryer as claimed in claim 2 in which the operating temperature of the vacuum chamber is controlled within the range of from approximately -30° C. to approximately $+30^{\circ}$ C. and the operating temperature of the condenser chamber is approximately -80° C., characterized in that the liquid is R11 (trichlore monofluoro-metane $-CCl_3F$).

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3656240 Dated April 18, 1972

Inventor(s) NICOLAAS ANTONIE VAN DIJK

It is certified that error appears in the above-identified patent
and that said Letters Patent are hereby corrected as shown below:

Col. 3, line 43 "++°C" should be --+30°C--

Col. 5, line 14 "tow" should be =-two--

Signed and sealed this 1st day of May 1973.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

ROBERT GOTTSCHALK
Commissioner of Patents

Signed and Sealed this day of 1973.
