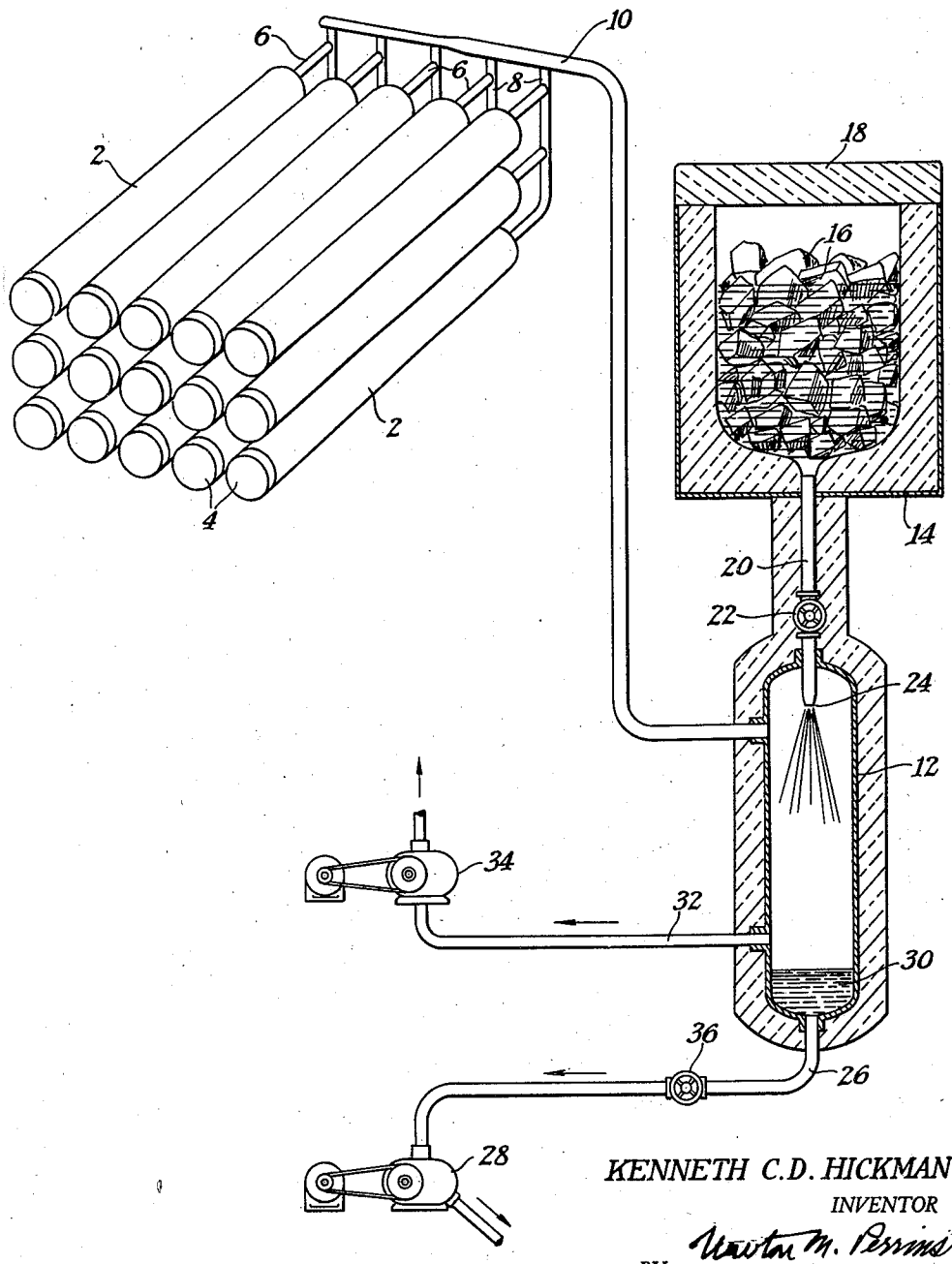
June 18, 1946. K. C. D. HICKMAN 2,402,401

METHOD AND APPARATUS FOR DEHYDRATING IN THE FROZEN STATE

Filed June 25, 1942 2 Sheets-Sheet 1

FIG. 1.



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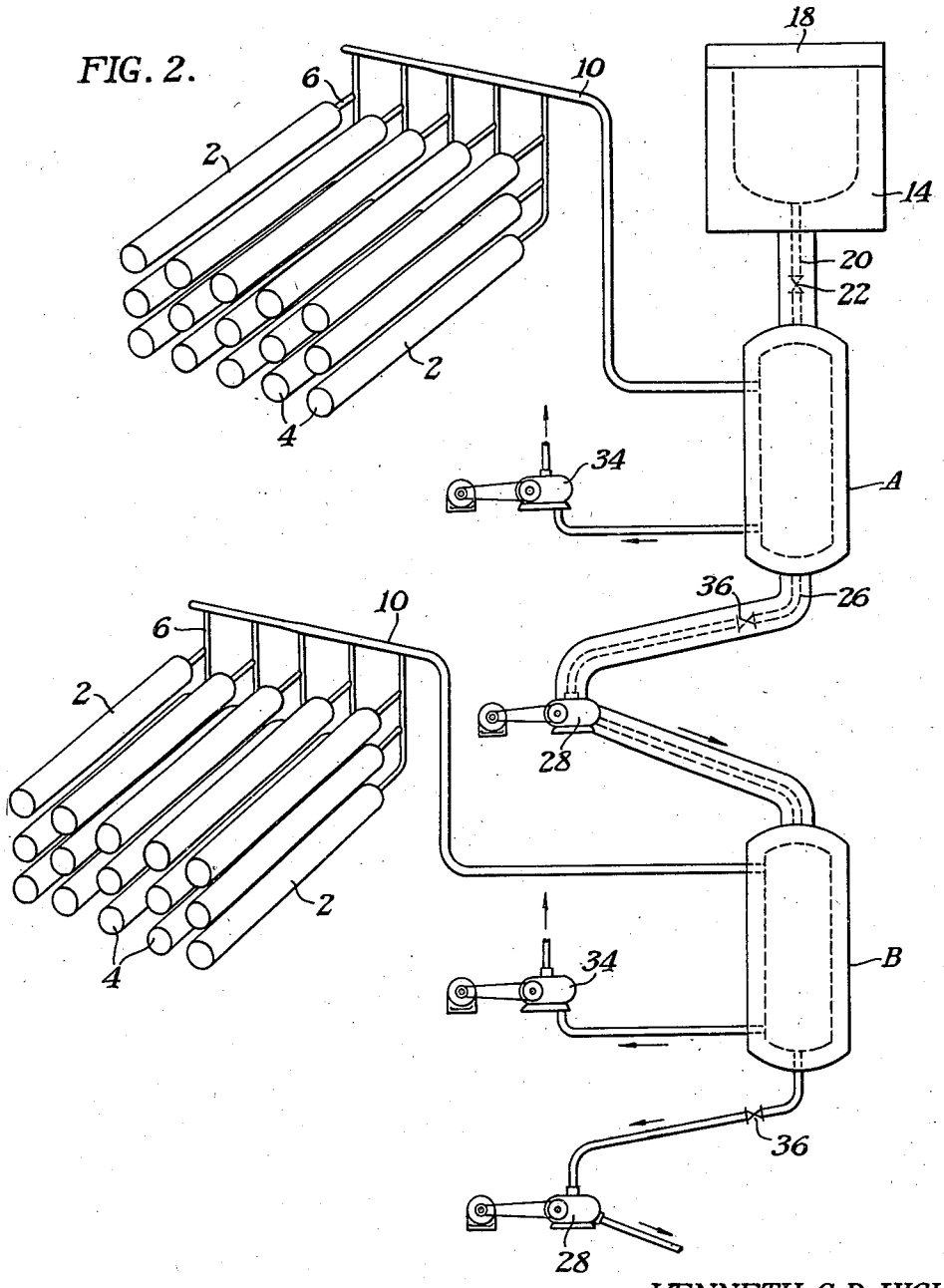
BY Newton M. Perrins
Warren H. Cannon
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FIG. 2.



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UNITED STATES PATENT OFFICE

2,402,401

METHOD AND APPARATUS FOR DEHYDRATING IN THE FROZEN STATE

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Application June 25, 1942, Serial No. 448,407

4 Claims. (Cl. 34—5)

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This invention relates to improved procedure for the vacuum dehydration of water-containing materials and apparatus for practicing such a process.

It is known to subject water-containing materials to dehydration at low pressures. See, for instance, Flosdorf 2,225,627 and Reichel Reissue 20,969. The procedure involves removal of water vapor at low pressures, usually 3 to 4 mm. of mercury when dehydrating from the frozen state. The procedure requires very low pressures since if the pressure is allowed to rise the material will melt causing undesirable changes therein, and also water vapor will cease to be given off at a suitable rate. The removal of water vapor at low pressures involves difficult pumping problems because enormous volumes of water vapor are given off at these low pressures. It has been suggested in the above-mentioned Flosdorf and Reichel patents to absorb the water vapor on a solid absorbent or to provide a trap with very cold surfaces upon which the water vapor is frozen. These procedures are expensive and inconvenient to use under many circumstances.

This invention has for its object to provide improved procedure and apparatus for the vacuum dehydration of water-containing materials. Another object is to provide improved procedure and apparatus for removing water vapor from frozen materials under relatively high vacuum. A still further object is to provide improved procedure and apparatus for the pumping of water vapor from a dehydrating chamber containing materials in the frozen state. Other objects will appear hereinafter.

These and other objects are accomplished by my invention which includes evacuating a closed system, which contains the frozen material to be dehydrated, to a pressure sufficiently low that water vapor is rapidly evolved therefrom, introducing into the closed system and into contact with the evolved water vapors a cold aqueous solution and removing spent solution from the closed evacuated system.

In the following description I have given several of the preferred embodiments of my invention, but it is to be understood that these are set forth for the purpose of illustration and not in limitation thereof.

In the accompanying drawings wherein like numbers refer to like parts I have illustrated in Fig. 1 a perspective view partly in section of a preferred embodiment of my invention and in Fig. 2 a similar view of two dehydrating machines illustrated in Fig. 1 connected in series in order to conserve ice.

Referring to Fig. 1 reference numeral 2 designates a bank of cylindrical dehydrating tubes which are preferably constructed of heat-conducting material such as steel or glass, which

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tubes are provided at one end with removable gas tight caps 4 and at the other end with an integral closed plate which is attached to conduits 6. These conduits connect with a header 8 which in turn connects to a main conduit 10 leading to the water vapor removal receptacle 12 which is preferably insulated as illustrated.

Numeral 14 designates a container or reservoir for ice 16 which container is suitably insulated and provided with a cover 18. Numeral 20 designates a conduit communicating with reservoir 14 and terminating inside absorbing chamber 12. This conduit is provided with a valve 22 and a spray nozzle 24. Numeral 26 indicates a conduit connected to the base of chamber 12 which leads to a liquid pump 28 which serves to remove liquid 30 from the system. Numeral 32 indicates a conduit connected to 12 at a point well above the level of liquid 30 which conduit connects to the intake of gas pump 34.

Referring to Fig. 2 letters A and B designate dehydrating units the same as those illustrated in Fig. 1 and connected in series by conduit 26. This conduit terminates in a spray nozzle the same as nozzle 24 shown in Fig. 1. The construction of the apparatus designated by the letter B is in all respects the same as that designated in letter A and described in detail in connection with Fig. 1.

In operating the apparatus illustrated in Fig. 1, caps 4 are removed and the tubes 2 are filled, or preferably partially filled, with material to be dehydrated. The caps 4 are then replaced and evacuating pump 34 is put into operation. This pump should be capable of evacuating the permanent gases in the system to a pressure sufficiently low that water vapor will be rapidly evolved from material being dehydrated. It is preferred that the pump be capable of removing gases to a sufficiently low pressure that the material will be self-frozen in the dehydrating tubes 2 without application of refrigerating means to cause freezing.

Ice is introduced into receptacle 14 and a suitable substance such as calcium or sodium chloride, which will form a low temperature solution on contact with ice, is introduced into receptacle 14 and thoroughly mixed with the ice. Valve 22 is then opened and a spray of cold aqueous solution issues from the spray nozzle 24. Water vapor evolved from the material being dehydrated in chambers 2 passes through conduits 6, 8 and 10 into chamber 12. Here the water vapor comes into contact with the cold aqueous liquid sprayed from 24 and becomes absorbed and condensed therein. This spent liquid solution is then collected at the base as indicated at 30. When sufficient liquid has accumulated at 30, valve 36 is opened and pump 28 is put into operation. The liquid is withdrawn from 12 at a rate such as to maintain a

substantially constant level at 38. However, sufficient liquid should not be permitted to accumulate at 30 that its temperature will rise and water vapor be evolved as a consequence. In other words, the liquid 30 should be withdrawn at a sufficient speed that its temperature will not be high enough to cause evolution of water vapor and destruction of the vacuum. Spent solution withdrawn from pump 28 may still have a rather low temperature and can be used for refrigeration purposes or for preliminary water removal from a material to be dehydrated. The solution can be treated to recover the salt or like constituent for re-use.

Pump 34 should be capable of producing a very low pressure in the system such as less than approximately 10 mm. and preferably less than about 3 to 4 mm. When such a pressure is established the water vapor will rapidly evolve from the frozen material being dehydrated. Also, lower pressures than those mentioned are advantageous at least in connection with the initial freezing of the material being dehydrated. Pump 34 can be a pump capable of handling water vapor, in which case no provision need be made for preventing water vapor from passing from receptacle 12 through conduit 32 into the pump. If the pump is not designed for handling water vapor it might be advantageous to place a cold trap in conduit 32 to remove small amounts of water which may be passing therethrough at the start of the dehydration procedure. After the process has once been established the spray from 24 will effectively remove the water vapor from the system and hence prevent it from passing into pump 34. Ejectors can be used in place of the mechanical pump illustrated.

The apparatus illustrated in Fig. 2 is operated in the same way that the apparatus is operated as described in connection with Fig. 1. However, instead of introducing cold brine from the ice into dehydrating apparatus B, the partially spent brine removed from dehydrator A by pump 28 is sprayed into dehydrator B where it at least partially absorbs the moisture from the material contained in the dehydrating tubes 2. This brine containing absorbed moisture is then withdrawn from chamber B by the removal pump 28 as described above.

The ice and substance which will form a low temperature solution on contact with ice are suitably proportioned to give a low temperature i. e. substantially below 0° C., to the liquid issuing from 24. Suitable substances for addition to ice to form the low temperature solution are ordinary salt, sodium thiosulfate, calcium chloride, sodium nitrate, ammonium chloride or nitrate and the like. The temperature of the solution is preferably the lowest which can be obtained from such mixtures. However, somewhat higher temperatures operate satisfactorily but are not as efficient.

Examples of water-containing materials which can be dehydrated are biological products such as blood plasma, antitoxins, and the like; meats and vegetables such as peas, beans, liver, soups; and fruit or fruit juices such as apricots and orange juice.

The advantages of my invention will be readily appreciated. The equipment necessary is of a simple and inexpensive type which can be readily installed in any desired locality and readily dismantled and moved when dehydrating procedure in that locality is no longer of interest. The operating materials required are very little more than supplies of ice and crystalline salts such as calcium and sodium chloride. The invention is inherently independent of specialized sources of vacuum. The equipment can be operated by unskilled labor with a short period of training. An outstanding advantage is that the procedure avoids the necessity of terminating the operations to remove accumulations of ice or replenish the absorbent as was necessary in the prior methods.

What I claim is:

1. The process of removing water vapor from a frozen organic water-containing material which is in a closed system, which process comprises evacuating the closed system to a sufficiently low pressure that water vapor is rapidly evolved from the frozen water-containing material, continuously spraying cold aqueous salt solution into the closed system and into contact with the evolved water vapors, but not into contact with the frozen material being dehydrated, continuously withdrawing the spent aqueous salt solution containing the absorbed water vapor from the closed system, introducing the spent solution into a second evacuated dehydrating system containing an organic, water-containing material which is to be less completely dehydrated and withdrawing spent aqueous solution from this second dehydrating system.

2. The process of dehydrating an organic water-containing material which is in the frozen state and which is in a closed system which process comprises evacuating the closed system to a pressure below 4 mm. of mercury, introducing a cold aqueous solution obtained by mixing ice with salt into the closed system but not in contact with the frozen material being dehydrated so that the introduced cold aqueous solution and the evolved water vapors are brought into contact with each other at below 4 mm. of mercury and removing spent aqueous solution containing the absorbed water vapor from the closed evacuated system.

3. The process of dehydrating a biological product which is in the frozen state and which is in a closed system which process comprises evacuating the closed system to a pressure below 4 mm. of mercury, introducing a cold aqueous solution obtained by mixing ice with calcium chloride into the closed system but not in contact with the frozen material being dehydrated so that the introduced cold aqueous solution and the evolved water vapors are brought into contact with each other at below 4 mm. of mercury and removing spent aqueous solution containing the absorbed water vapors from the closed evacuated system.

4. Apparatus for dehydrating water-containing materials comprising in combination a closed system including a dehydrating chamber, means for evacuating the closed system, a receptacle for mixing ice with a salt, means for introducing the resultant cold aqueous solution from the receptacle into the closed system, means for exposing a large surface area of the introduced solution into contact with the vapors evolved from the material being dehydrated, which exposing means is positioned in the closed system so that the cold aqueous solution does not come into contact with the material being dehydrated and so that the evolved water vapors and the cold aqueous solution are brought into contact at about the same pressure as that pressure to which the material to be dehydrated is subjected and means for removing spent aqueous solution from the closed system.

KENNETH C. D. HICKMAN.