APPARATUS FOR FREEZE-DRYING

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The invention relates to an apparatus for freeze-drying products containing water in vacuum, said apparatus being of the type that has a cabinet with built-in cooling surfaces for freezing the water vapour out as ice, and means to thaw the formed ice off.

In connection with freeze-drying in vacuum of products containing water it is necessary to remove very large volumes of vapour from the products. At a pressure of 1 torr it is thus necessary to remove approx. 1000 m³ of vapour pr. kg. of evaporated water. It is known to precipitate the large volumes of vapour as ice on the cooling surfaces at a temperature of, for instance, −35° C.

There is known an apparatus in which the cooling surfaces are placed in the cabinet in which the drying takes place. This is a space saving and cheap solution which, though, has the very essential drawback, that the ice must be removed from the cooling surfaces after each drying operation so that said surfaces can become effective again for the subsequent drying operation, and this removal of the ice, which as a rule takes place by thawing, requires a certain amount of time which thereby is lost as production time for the apparatus.

Furthermore there is known an apparatus in which the mentioned drawback has been remedied by placing the cooling surfaces outside the drying cabinet itself in a special closeable chamber. In the last part of the drying period, the water vapour emission from the material is, as a rule, much less than in the preceding part of the drying period. The smaller amount of water in the last part of the drying period can often be removed by means of the evacuation plant alone or possibly by means of a small auxiliary cooling surface, and said closeable chamber with cooling surfaces can then be closed off from the drying cabinet in this period, and it can be defrosted, so that the next drying period can be initiated immediately after the termination of the preceding drying period. This known apparatus has the drawback that it is rather bulky and expensive. It is thus necessary to use a very large pipe for the connection between the cabinet and the special chamber, and this fact in connection with the fact that the space in the cabinet is not utilized as intensively as in the above mentioned construction with built-in cooling surfaces, entails that the total volume to be evacuated will be relatively large so that a rather large evacuation plant must be employed. Furthermore, the separation into a cabinet and a special chamber involves increased mounting expenses.

The object of the invention is to devise an apparatus of the type indicated in which the drawbacks mentioned above are remedied so that a good utilization of the production time is obtained by means of a relatively cheap apparatus that does not require much space. The characteristic features of the apparatus according to the invention are that the cooling surfaces are separated from the rest of the cabinet by means of partition walls which have an aperture with a closing member that can be opened and closed. The function of the apparatus is substantially the same as the function of the known apparatus with the special closeable chamber for the cooling surfaces, but as, in connection with the thawing of the ice off the cooling surfaces, there only needs to appear a relatively small pressure in the chamber bounded by the partition walls, e.g. 10–20 torr, and as a still smaller pressure exists in the rest of the cabinet, e.g. 1 torr, the stress on the partition walls will be slight so that said walls may be made with small strength. As there only exists a small pressure difference it is not necessary either to have a large degree of tightness, and the construction can therefore be made relatively cheaply. Especially, there may be employed quite simple and cheap closing members. When the closing member is closed the ice may be thawed off and the cooling surfaces while the drying continues in the rest of the cabinet.

According to the invention the apparatus may have partition walls to form two or more chambers, each having its own cooling surface and its own aperture, and closing members to open and close said apertures. In this manner ice can be thawed off from the cooling surfaces in one or more chambers that are closed off from the rest of the cabinet while the drying continues by means of the cooling surfaces in one or more other chambers that are open in relation to the cabinet so that a suitable cooling surface is at disposal at all times. In one embodiment there may be two chambers having cooling surfaces of equal sizes, but other embodiments are conceivable, e.g. a larger or said chambers having large cooling surfaces and being employed in the first part of the drying, the other chamber having a small cooling surface and being employed in the last part of the drying while thawing off is taking place in the first mentioned chamber. An embodiment with three or more chambers with cooling surfaces of equal size is also conceivable, it being possible in this embodiment to interchange in a suitable manner, so that in the beginning of the drying there is a large cooling surface at disposal, while later a change-over takes place between the chambers so that thawing off at all times is taking place in one of the chambers whereas in the final stage thawing off takes place in all chambers except one.

The closing members for two chambers may, according to the invention, be adapted to open one of the chambers while the other chamber simultaneously is closed and vice versa. By these means a simple and practical construction is obtained where a change-over between the cooling surfaces in the two chambers suitably may take place during each drying.

The apparatus may, according to the invention, have time program control members to control the opening and closing of the closing members for each of the chambers and the de-frosting of the cooling surfaces in question. In this manner a simple and advantageous control is obtained. However, it will be possible to provide for the control in another manner, e.g. in dependency of a sensing of the ice thickness on the cooling surfaces, a summation of the cooling machine calories, a summation of the heat supply calories or the pressure in the drying cabinet.

The chamber, or each of the chambers, may, according to the invention, be connected to a water vapour generator that has a member to maintain the water at a temperature below a certain maximum temperature. This maximum temperature is determined by the strength of the partition walls. Supposedly it would be sufficient to dimension the partition walls for a pressure difference of 10–20 torr, as the maximum temperature corresponding hereto will be sufficient for a suitably rapid thawing of the ice on the cooling surfaces. It is possible to ensure in different manners that the water temperature does not become too high, and likewise it is possible to dimension the thermal security valves or like security means in the partition walls so as to avoid too large pressure differences. The water vapour generator may, according to the invention, be made like a heat exchanger that receives heat from cooling water.
or cooling medium from the components of the apparatus with the purpose of evacuation or of cooling the cooling surfaces. In this manner a heating medium with a suitable temperature is obtained in a simple manner.

In the accompanying drawings there is shown an apparatus according to the invention, the figure showing a vertical cross sectional view of a freeze-drying cabinet in an apparatus according to the invention. A cylindrical freeze-drying cabinet is made of steel plate 1 with reinforcement rings 2 and is placed on legs 3. In the top part of the cabinet a rail 4 is suspended in which a supporting frame 5 is suspended, said frame 5 carrying trays 6 for the material to be freeze-dried. The trays 6 are inserted between heating plates 7 which form part of a heating medium system not illustrated in the drawings.

In the bottom of the cabinet there are cooling surfaces 11 and 12 in the form of pipes in which a cold cooling medium can be circulated from a not shown cooling plant. The cooling surfaces are separated from the rest of the cabinet and from each other by means of partition walls 8 and 9 and by means of a closing member in the shape of a plate 10 that may be set to close an aperture in one or the other of the chambers formed by the partition walls. These chambers can be connected to an evacuation plant, not shown, by means of pipes 13 and 14, in which closing members 15 and 16 are arranged. Pipes 17 and 18 are connected to the bottom of the two chambers, closing valve 19 and 20 being arranged in said pipes, the latter forming a connection to a water vapour generator in the shape of a container 21 that is filled with water up to a level 22. The water in this container can receive heat from pipe 23, through which a medium warmer than the water can be circulated by means of the connection pipes 24 and 25. A temperature control member 29 is provided inside the container 21 to maintain temperature of the water below a certain maximum value which is determined by the strength of the partition walls. The container 21 has a spillover pipe 26 with a valve 27 through which the water above the level 22 can be removed in the pause between the freeze-drying periods, atmospheric pressure existing in the cabinet and parts connected thereto in said pause, that is, while a new batch of material to be freeze-dried is being inserted in the apparatus.

The freeze-drying process takes place in the following manner: Fresh goods in the trays 6 are placed in position in the cabinet between the heating plates 7. The cabinet is then sealed and is evacuated, for instance through the pipe 13 with the valve 15 open, until the pressure has been reduced to approx. 1 torr. The necessary conditions for freeze-drying the goods are then present. Heating medium is then supplied to the heating plates 7 and cooling medium is supplied to the cooling surfaces 12. The closing valve 19 is in its right hand position, shown with full lines. The valve 19 is closed. So as to avoid rupture of the partition walls evacuation of the space around the cooling surfaces 11 must be provided for. This can possibly be done by not closing the valve 10 tightly or by keeping the valve 16 open during the first evacuation period in each freeze-drying process. As a result, the stress on the partition walls 8 and 9 will be reduced so that said walls may have substantially smaller strength than the steel plate 1.

The goods in the trays 6 now emit water vapour which is precipitated as ice on the cooling surfaces 12. When the ice on the cooling surfaces 12 has grown so much that the latter's effectiveness has been reduced considerably the valve 10 is moved to the left hand position shown with dotted lines. The valve 16 is opened whereas the valve 15 is closed. The pressure in the cabinet will then be kept down through the pipe 14. Furthermore, the cooling medium of tray 6 now supports the cooling medium, whereas this is no longer the case for the cooling surfaces 12. The valve 20 is closed and the valve 19 is opened. The water vapour from the goods will now be precipitated as ice on the cooling surfaces 11. The cooling surfaces 12, which are separated from the rest of the cabinet, are connected to the container 21 by means of the pipe 17 and the valve 19. Water vapour is developed from the water in the container 21, said vapour flowing up through the pipe 17 and condensing on the ice on the cooling surfaces 12. Hereby heat is transmitted to the ice and the pressure in the chamber in question increases so that the ice melts successively. The water from the melting process flows through the pipe 17 down into the container 21.

When so much ice has accumulated on the cooling surfaces 11 that the latter's effectiveness has been considerably reduced, and when the cooling surfaces are completely free of ice, a change-over of the valves 15, 16, 19 and 20 and the hinged valve 10 is carried out so that cooling surfaces 11 and 12 exchange functions. In this manner a change-over between two groups of cooling surfaces is carried out until the drying is terminated. At this point the water will be removed to a desired degree from the goods in the trays 6 and will be collected in the container 21 that must have a correspondingly large volume. When the drying is terminated the pressure in the cabinet and the container 21 is increased to atmospheric pressure. The dried goods are taken out and packed and the water is let out of the container 21 until level 22 is attained. To control automatically the operation of respective closing members, the apparatus may have a control means which is adapted to control operatively the valves 10, 15, 16, 18, 19 and 27 according to a predetermined time program, or in dependency of a sensing member (not shown), such as for instance a member for sensing ice thickness on the cooling surfaces 11 and 12, or a pressure sensing member in the drying cabinet.

The shown and described embodiment is only to be considered by way of example as different variations are conceivable within the scope of the invention.
2. Apparatus for the freeze drying in vacuum of products containing water, comprising a principal chamber for containing products to be dried, heating means in said principal chamber for heating said products, at least one cooling chamber, said cooling chamber being separated from said principal chamber by vacuum-tight partition means, for condensing water vapor out as ice, valve means in said vacuum-tight partition means for interconnecting said principal chamber and said cooling chamber, evacuating means for evacuating non-condensible gases from said principal chamber, melting means for melting ice formed in said cooling chamber, pressure control means for maintaining a pressure substantially lower than atmospheric pressure in said cooling chamber during the melting of ice in said cooling chamber, a water vapor chamber, vacuum-tight partition means separating said water vapor chamber from said cooling chamber, valve means in said last mentioned vacuum-tight partition means for interconnecting said cooling chamber and said water vapor chamber, said melting means comprising water vapor generating means in said water vapor chamber, temperature control means for controlling the temperature of water vapor generated by said water vapor generating means, and time control means for controlling said valve means to selectively interconnect said cooling chamber and said water vapor chamber.

3. Apparatus as claimed in claim 1, wherein said water vapor generating means comprises a container for water, heating means for heating water in said container and overflow means for removing excess water above a determined level from said container.

4. Apparatus as claimed in claim 1, wherein said water vapor generating means comprises heat exchanging means.

5. Apparatus as claimed in claim 1, further comprising time control means coupled to and controlling said valve means and each of said plurality of valves for controlling the selection of a cooling chamber for freezing water vapor from said principal chamber out as ice and for controlling the selection of a cooling chamber for melting ice formed on the cooling surface thereof.

6. Apparatus as claimed in claim 2, wherein said valve means is positioned to open said water vapor chamber into the lowest point of said cooling chamber to drain water formed by melting the ice in said cooling chamber into said water vapor chamber, and wherein said water vapor chamber includes discharge means for discharging part of the water therein.

References Cited

UNITED STATES PATENTS
2,994,132 8/1961 Neumann 34—5
3,116,122 12/1963 Otten 34—92
3,132,930 5/1964 Abbott 34—5
3,178,289 4/1965 Cox 34—5
3,233,333 2/1966 Oppenheimer 34—5
3,255,534 6/1966 Kan 34—5
3,262,212 7/1966 De Buhr 34—5
3,273,259 9/1966 Hackenberg 34—5

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