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(54) **METHOD OF STERILIZATION OF CONTAINER FOR STERILE PACKING.**

(57) A method of sterilization using a gas or water containing a high concentration of ozone to shorten the sterilization time and eliminate the necessity for washing by sterile water after the sterilization.

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METHOD OF STERILIZATION OF CONTAINERS FOR ASEPTIC PACKING

Technical Field

The present invention relates to a dry-type method of sterilizing containers for aseptic packing, and more specifically to a method of sterilization by using a gas that contains ozone at a high concentration and
 5 ozone water of a high concentration.

Background Art

Air-tightly packed foods have heretofore been widely produced by method according to which a content
 10 is packed in a container and is air-tightly sealed, and the air-tightly sealed package is heated and sterilized in a device called retort, and by a method (hot packing) according to which the content is packed in a container while it is hot and is air-tightly sealed. These methods, however, involve such problems that the composition is broken, vitamins and pigments are destroyed, and quality is deteriorated as represented by browning, due to the excess heating of the contents.

15 In order to solve such problems, there has been proposed a method in which the content is sterilized at a high temperature for a short period of time outside the packing container by means such as a heat exchanger, and the content after sterilization is packed in the container that has been sterilized or pasteurized and is air-tightly sealed.

Water of hydrogen peroxide and aqueous solution containing peracetic acid have been commercially
 20 used for the sterilization of aseptic containers making, however, it necessary to use large amounts of aseptic water for the rinsing.

There have so far been proposed a variety of dry methods of sterilizing containers such as a method which uses the combination of hot air and sterilization lamp (Japanese Utility Model Publication No. 35867/1964), a method using a high-frequency induction heating (Japanese Laid-Open Patent Publication
 25 No. 13183/1975), and a method which uses ultraviolet-ray irradiation and high-frequency induction heating (Japanese Laid-Open Patent Publication No. 15027/1984), which, however, have not yet been put into practical use.

It has further been known that a gas containing ozone and ozone water exhibit sterilizing ability requiring, however, an increased period of time for effecting the sterilization. Therefore, the gas containing
 30 ozone has been used for sterilizing the refrigerators and air-conditioning facilities, and ozone water has simply been used in such fields as city water and sewer systems, raw sewage disposal, and medical equipment. There has not yet been reported the use of ozone-containing gas or ozone water for the sterilization of containers for packing.

In the case of the method that uses ultraviolet rays for sterilization, the sterilization is little effected in
 35 the shaded portions and, besides, it is difficult to uniformly irradiate the interior of the container. The method that uses the high-frequency induction heating, on the other hand, is limited to treating metal containers such as cans, and is not suited to uniformly heating the cubic containers.

The present inventors have studied in an attempt to apply ozone for the sterilization of containers for aseptic packing, and have discovered the fact that the concentration of ozone plays a very important role
 40 for sterilizing the containers for aseptic packing, and that the sterilization is carried out within short periods of time and reliably if a gas or water containing ozone at a concentration greater than a predetermined critical value is brought into contact with at least the inner surfaces of the containers.

Summary of the Invention

45 According to the present invention, there is provided a method of sterilization of containers for aseptic packing which comprises bringing an ozone-containing gas or ozone water into contact with at least the inner surfaces of the containers for aseptic packing, said ozone-containing gas having an ozone concentration of greater than 4000 ppm and said ozone water having an ozone concentration of greater than 10 ppm.

50 According to the present invention, it is preferred to finish the sterilization within five minutes and, especially, within two minutes.

In one aspect of the present invention, furthermore, the air in the containers for aseptic packing is substituted by an ozone-containing gas and then the ozone-containing gas is kept contained in the containers, or the containers are filled with the ozone water which is then kept contained therein, in order to finish the sterilization treatment during the time while the containers are being stored and transported.

Brief Description of Drawings

Fig. 1 is a horizontal section view of a rotary sterilization apparatus to which the method of sterilization of the present invention is adapted; and

Fig. 2 is a vertical partial section view showing a major portion of the sterilization apparatus of Fig. 1.

5 Best Mode for Carrying out the Invention

The present invention is based on a discovery that the sterilization treatment is completed within a very short period of time and, generally, within five minutes if an ozone-containing gas having an ozone concentration higher than 4000 ppm or the ozone water having an ozone concentration higher than 10 ppm
10 is brought into contact with at least the inner surfaces of the containers.

So far, ozone to be contained in a gas is usually generated by the high-voltage electric discharge (voiceless discharge and creeping discharge). However, the concentration of ozone in the gas is of the order of 2000 ppm at the highest, and the time of 30 minutes to one hour or longer is necessary for effecting the sterilization in the container; i.e., the ozone concentration of this order is far from being
15 utilizable for the dry method of sterilization of containers for aseptic packing. The reason is attributed to the fact that microorganisms that are to be sterilized adhere on the surface of the container wall, a very thin air layer (laminar layer) that is difficult to remove exists on the surface, and the microorganisms exist under the condition being protected by the laminar layer, and that an extended period of time is needed to replace the laminar layer by the ozone-containing gas, and the probability and the frequency in which the ozone and
20 the microorganisms come in contact with each other are not so high.

According to the present invention which uses a gas containing ozone at a concentration as high as 4000 ppm or greater, the probability and frequency in which the ozone and the microorganisms to be sterilized come in contact with each other are increased, the air in the container is replaced by the ozone-containing gas of a high concentration which is then kept contained therein to prevent the ozone from being
25 diluted, and the laminar layer of air is thus easily replaced by the ozone-containing gas of a high concentration making it possible to effect the sterilization within a short period of time and reliably.

When the ozone water is used, the ozone water comes into direct contact with the wall surfaces of the container. Therefore, no laminar layer is formed on the wall surface of the container unlike the case of using the gas, and ozone directly acts upon the wall surfaces.

30 The half-life of ozone decomposition is about 10 hours. The air in the containers for aseptic packing is replaced by the ozone-containing gas which is then kept contained therein, or the containers are filled with the ozone water which is then kept contained therein, in order to reliably effect the sterilization in the containers by utilizing the time for storing and transporting the containers and by using the ozone-containing gas in relatively small amounts.

35 Moreover, since ozone turns into harmless oxygen in about 10 hours, safety is maintained even if some ozone happens to remain in the containers.

In the case of the dry method according to the present invention, use is made of a gas having an ozone concentration of greater than 4000 ppm and, particularly higher than 5000 ppm. As the ozone gas generator that maintains such a high concentration, there can be used an ozone generator based on the pulse
40 discharge method, such as an ozone generator of the type of streamer discharge manufactured by Ishikawajima-Harima Heavy Industries Co. The ozone generator of this type has a distinguished advantage in that a gas containing ozone at a high concentration can be generated in large amounts per unit time.

In the case of the wet method, use is made of the ozone water having an ozone concentration of greater than 10 ppm and, particularly, greater than 20 ppm. So far, the ozone concentration in the ozone water was
45 2 to 3 ppm at the greatest. In recent years, however, it has been made possible to produce the ozone water having a concentration as high as 20 to 30 ppm. The ozone water having such a high concentration can be obtained by using an ozone water-producing apparatus disclosed in, for example, Japanese Patent Laid-Open Publication No. 119991/1990.

Examples of containers for aseptic packing include metal cans, glass bottles, plastic containers, various
50 film containers, pouches, caps, can lids, and the like. The present invention is particularly effective in the dry sterilization of plastic containers and a variety of film containers that are subject to be easily deformed by the heat of sterilization. Moreover, the containers should have small ports and, particularly, the shapes of a various kinds of bottles, such that the ozone-containing gas is effectively held in the containers.

It is desired that the containers for aseptic packing are sterilized just prior to aseptically packing the
55 contents. That is, the ozone-containing gas is replaced for the air in the containers and is held therein, or the ozone water is fully poured into the containers and is held therein, continuously or intermittently while the containers are being supplied to the step of aseptic packing using, preferably, an apparatus of the tunnel type or of the rotary type.

The accompanying Figs. 1 and 2 are a horizontal section view and a vertical partial section view of the rotary sterilizing apparatus which can be placed in practice using either the ozone-containing gas or the ozone water.

Described below is the case when use is made of the ozone-containing gas.

5 In the drawings, the sterilization zone 5 is locally partitioned by a fixed outer peripheral wall 4 and a rotary wall having a $\equiv$ -shape in cross section that consists of an inner peripheral wall 1 and an upper wall 2 integrally mounted on a turn table 3. The outer peripheral wall 4 is removable, and a very small gap 6 is provided between the rotary wall and the outer peripheral wall 4 to facilitate the rotation of the rotary wall and to permit the blow-out of the ozone-containing gas in small amounts. On the sterilization zone 5 are
10 arranged a number of container seats 7, 7, --- in the form of a ring, being held by container-support fittings 8. A container 9 is placed on each container seat 7, and a nozzle 10 for feeding the ozone-containing gas is installed thereon penetrating through the upper wall 2. The nozzle 10 is equipped with a nozzle elevation drive mechanism 11 and a switching valve 12 which is connected to an ozone-containing gas storage vessel 14 through a conduit 13. The ozone-containing gas storage vessel 14 is served with the ozone-containing
15 gas of a high concentration from an ozone generator 15 through a conduit 16 and a rotary joint 17.

The rotary sterilizing apparatus is provided along a container feed line 18, and an introduction unit 19 and a delivery unit 20 thereof are partitioned by fixed outer walls 21, an outer upper wall (not shown), tables (not shown) under star wheels 22, and a rotary wall (not shown).

20 The container 9 is placed on the container seat 7 on the turn table as it is introduced from the introduction unit 19, and the elevation drive mechanism 11 is energized to insert the nozzle 10 into the container 9. Then, the switching valve 12 is opened so that the ozone-containing gas of a high concentration is emitted into the container 9 from the ozone-containing gas storage vessel 14. Therefore, the air in the container 9 is replaced by the ozone-containing gas which is then contained therein. The ozone-containing gas has a pressure which is slightly higher than the atmospheric pressure. Therefore, not only the interior of
25 the container 9 but also the interior of the sterilization zone 5 are filled with the ozone-containing gas that gushes from the port of the container, preventing the contaminated air from flowing into the sterilization zone 5.

A very small gap 6 is provided between the rotary wall and the outer peripheral wall in the diagrammed embodiment. In order to prevent the ozone-containing gas from leaking into the working environment,
30 however, this portion may be constructed in a tight-seal structure or a local exhaust facility may be provided in this portion.

The ozone-containing gas is kept contained in the container 9 for a predetermined period of time to complete the sterilization operation. The necessary time for keeping the gas contained generally ranges from 0.5 minutes to 2 minutes. After the sterilization operation is finished, the switching valve 12 is closed
35 and the nozzle 10 is moved upwards by the elevation drive mechanism 11. The container 9 after sterilized is delivered onto the container feed line 18 via the delivery unit 20 and is fed to a step of aseptic packing (not shown).

It is further allowed to continuously flow the ozone-containing gas through the nozzle 10 instead of providing the switching valve 12, as well as to emit the aseptic air into the container 9 after it has been
40 sterilized by switching the valve 12 in order to replace the ozone-containing gas in the container by the aseptic air.

It is preferred that the ozone-containing gas is used in an amount of from 0.5 to 5 times as much (standard condition) with the volume of the container as a reference and, particularly, in an amount of 1 to 3 times as much as the volume of the container.

45 Even when the ozone water is used, it is allowed to use the same apparatus as that of case of ozone-containing gas as described above. In this case, the ozone water is kept poured into the container for aseptic packing to overflow in order to sterilize not only the inner surface of the container but also the outside of the container at the same time.

It is further allowed to hold the container upside down with its port being located at the lower part, and
50 to spray the ozone water into at least the inner surface of the container. In this case, the ozone water does not stay in the container, and no step is required to drain the ozone water.

Even when the ozone water is used, the ozone water is kept contained in the container for a predetermined period of time to complete the sterilization operation. The necessary time for keeping the water contained generally ranges from 0.5 minutes to 5 minutes.

55

Examples

Example 1

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Spores of bacillus subtilis are adhered in a number of 10^4 on the inner surface of a 1000-ml glass bottle.

Ozone generated by the ozone generator A is blown at a concentration of 400 ppm (air-flow rate of 40 liters/min.) into the glass bottle.

5 Ozone generated by the ozone generator B is blown at a concentration of 6000 ppm (air-flow rate of 6 liters/min.) into a glass bottle.

The spores in the glass bottle are washed out with the physiological saline solution and are cultured. The number of the surviving spores are counted.

<u>Number of surviving spores/glass bottle</u>			
	<u>Treatment time</u>	<u>400 ppm (A)</u>	<u>6000 ppm (B)</u>
	(Initial number of spores)	10^4	10^4
15	30 sec.	+ +	0
	1 min.	+ +	0
	2 min.	+ +	0
20	5 min.	+ +	0
	20 min.	243	0
	30 min.	47	0
25	60 min.	3	0

Example 2

30 Spores of bacillus subtilis are adhered in a number of 10^4 on the inner surface of a 1500-ml plastic bottle.

Ozone generated by the ozone generator B is blown at a concentration of 6000 ppm (air-flow rate of 6 liters/min.) into the plastic bottle.

35 The spores in the plastic bottle are washed out with the physiological saline solution and are cultured. The number of the surviving spores are counted.

<u>Number of surviving spores/plastic bottle</u>		
	<u>Treatment time</u>	<u>6000 ppm (B)</u>
	(Initial number of spores)	10^4
45	30 sec.	0
	1 min.	0
	2 min.	0
	5 min.	0
50	20 min.	0
	30 min.	0
	60 min.	0

55

Example 3

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Spores of each of the following indicator bacteria are adhered in a number of 10^8 on the inner surfaces of the 1500-ml plastic bottles.

The plastic bottles are filled with the ozone water of a temperature of 20°C having an ozone concentration of 25 ppm.

5 After the water is kept contained for predetermined period of time, the spores in the plastic bottles are washed out and are cultured to count the number of the surviving spores.

(1) *Bacillus subtilis*

10

<u>Treatment time</u>	<u>Number of surviving spores/ plastic bottle</u>
-----------------------	---

15

(Initial number
of spores)

4×10^8

1 min.

4×10^4

2 min.

5×10^3

20

3 min.

1×10^2

5 min.

0

10 min.

0

25

(2) *Bacillus cereus*

30

Treatment time

Number of surviving spores/
plastic bottle

(Initial number
of spores)

2×10^8

35

1 min.

0

3 min.

0

5 min.

0

40

(3) *Salmonellas*

45

Treatment time

Number of surviving spores/
plastic bottle

(Initial number
of spores)

5×10^8

50

1 min.

0

3 min.

0

5 min.

0

55

(4) *Staphylococci*

	<u>Treatment time</u>	<u>Number of surviving spores/ plastic bottle</u>
5	(Initial number of spores)	3×10^8
	1 min.	0
	3 min.	0
10	5 min.	0

According to the present invention which uses the ozone-containing gas of a concentration which is as high as 4400 ppm or more or which uses the ozone water having an ozone concentration of greater than 10 ppm for the sterilization of containers for aseptic packing as described in the foregoing, the time for sterilization is shortened and ozone decomposes into harmless oxygen with the passage of time. Therefore, either when the ozone-containing gas is used or when the ozone water is used, safety is maintained despite ozone happens to remain in the containers. Moreover, no step is required for washing out the water of sterilization.

Claims

1. A method of sterilization of containers for aseptic packing which comprises bringing an ozone-containing gas or ozone water into contact with at least the inner surfaces of the containers for aseptic packing, said ozone-containing gas having an ozone concentration of greater than 4000 ppm and said ozone water having an ozone concentration of greater than 10 ppm.
2. A method of sterilization of containers for aseptic packing according to claim 1, wherein the sterilization treatment is finished within five minutes.
3. A method of sterilization of containers for aseptic packing according to claim 1, wherein the air in the containers for aseptic packing is replaced by the ozone-containing gas which is then kept contained therein to effect the sterilization treatment.
4. A method of sterilization of containers for aseptic packing according to claim 1, wherein the containers for aseptic packing are filled with the ozone water which is then kept contained therein to effect the sterilization treatment.
5. A method of sterilization of containers for aseptic packing according to claim 4, wherein the ozone water is kept poured into the containers for aseptic packing to overflow in order to effect the sterilization treatment.
6. A method of sterilization of containers for aseptic packing according to claim 1, wherein the containers for aseptic packing are held upside down, and the ozone water is sprayed at least onto the inner surfaces of said containers to effect the sterilization treatment.
7. A method of sterilization of containers for aseptic packing according to claim 1, wherein the containers for aseptic packing after brought into contact with the ozone-containing gas or the ozone water are fed to the step of packing the contents without effecting the after-washing.

FIG. 1

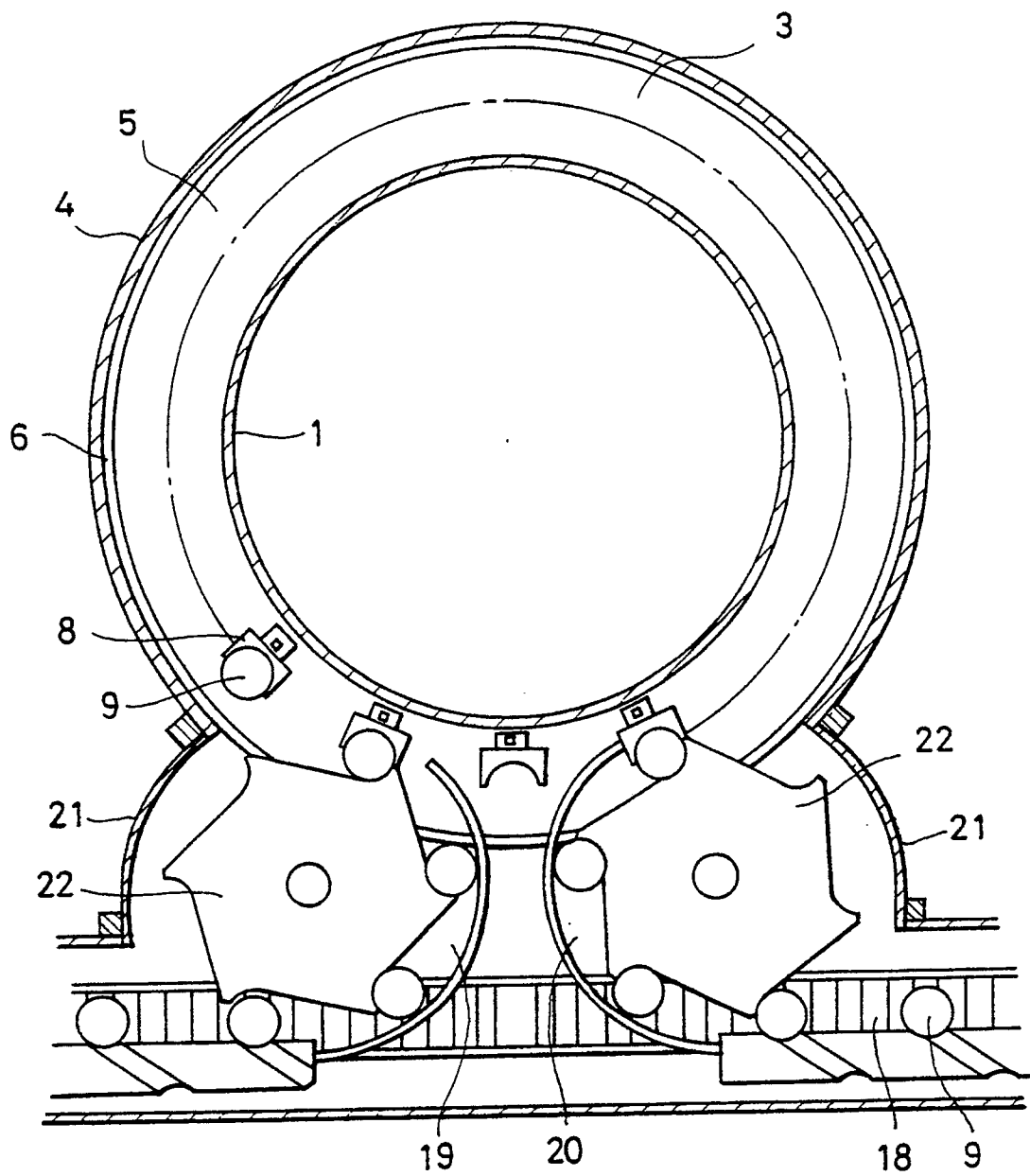
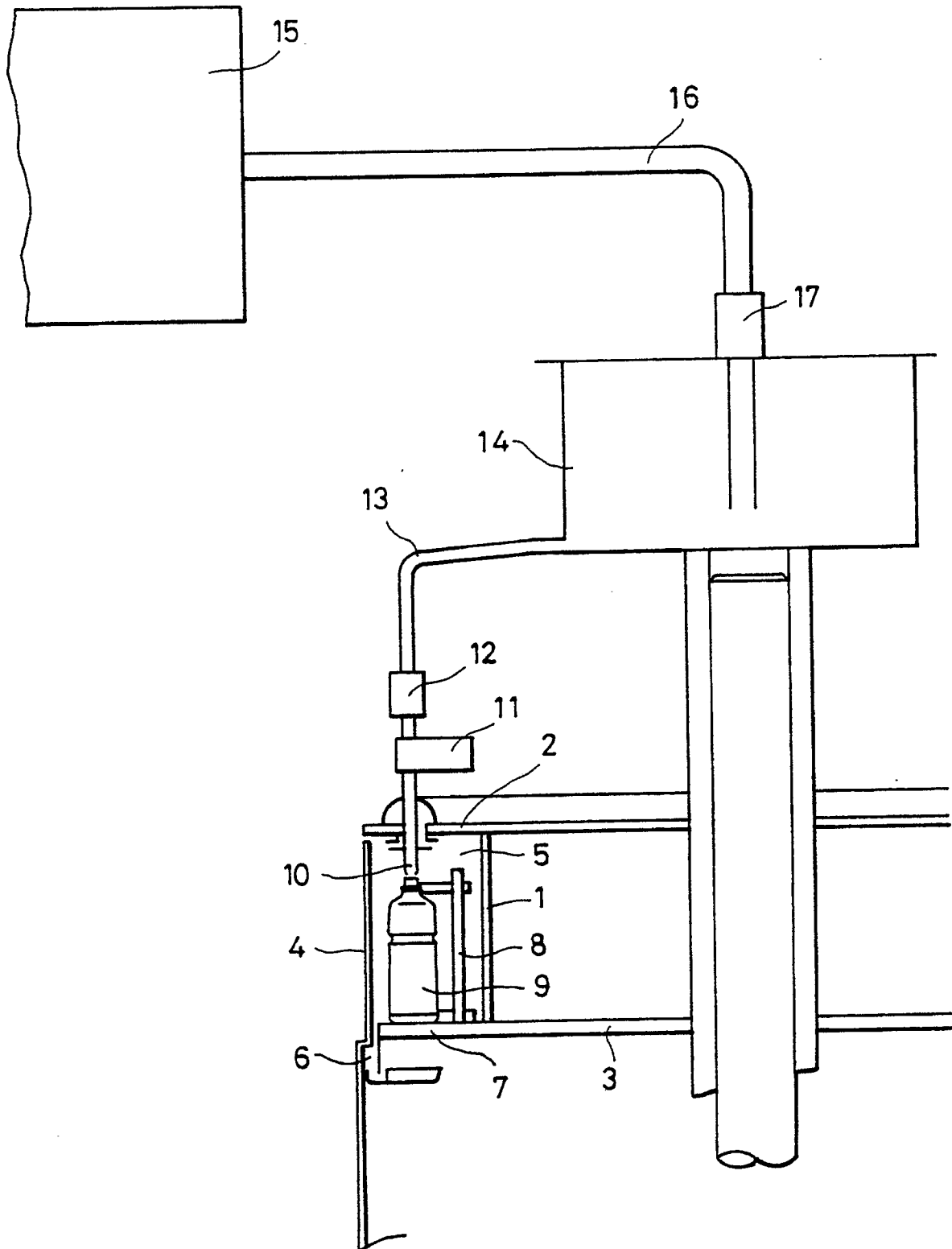


FIG. 2



INTERNATIONAL SEARCH REPORT

International Application No PCT/JP90/00955

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶ According to International Patent Classification (IPC) or to both National Classification and IPC Int. Cl⁵ B65B55/10																						
II. FIELDS SEARCHED Minimum Documentation Searched ⁷ <table border="1" style="width: 100%; border-collapse: collapse; margin-top: 5px;"> <tr> <td style="width: 30%; padding: 5px;">Classification System</td> <td style="padding: 5px;">Classification Symbols</td> </tr> <tr> <td style="text-align: center; padding: 10px;">IPC</td> <td style="text-align: center; padding: 10px;">B65B55/10, 55/18</td> </tr> </table> Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸ <table style="width: 100%; margin-top: 10px;"> <tr> <td style="width: 40%;">Jitsuyo Shinan Koho</td> <td style="width: 60%; text-align: right;">1926 - 1989</td> </tr> <tr> <td>Kokai Jitsuyo Shinan Koho</td> <td style="text-align: right;">1971 - 1989</td> </tr> </table>			Classification System	Classification Symbols	IPC	B65B55/10, 55/18	Jitsuyo Shinan Koho	1926 - 1989	Kokai Jitsuyo Shinan Koho	1971 - 1989												
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