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Muys et al.

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[54] **STERILIZATION OF CONTAINERS**

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[21] Appl. No.: **369,348**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 131,626, April 6, 1971, abandoned.

[52] **U.S. Cl.** ..... **53/21 FC**; 21/58; 53/22 R; 53/111 RC; 53/110

[51] **Int. Cl.** **B65b 55/10**

[58] **Field of Search** ...53/21 FC, 21 R, 22 R, 111 RC, 53/110, 112 R; 21/58

[56] **References Cited**

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Attorney, Agent, or Firm—Arnold Grant, Esq.

[57] **ABSTRACT**

A process and apparatus for continuously sterilising aseptically filling and closing containers, in which each successive container is dosed with a quantity of a sterilising agent which is retained within the container for a sufficient time for sterilisation to take place, is conveyed through a tunnel within which a gaseous environment under slight overpressure is maintained, and is filled and has a sterile lid applied under sterile conditions within the tunnel.

12 Claims, 5 Drawing Figures

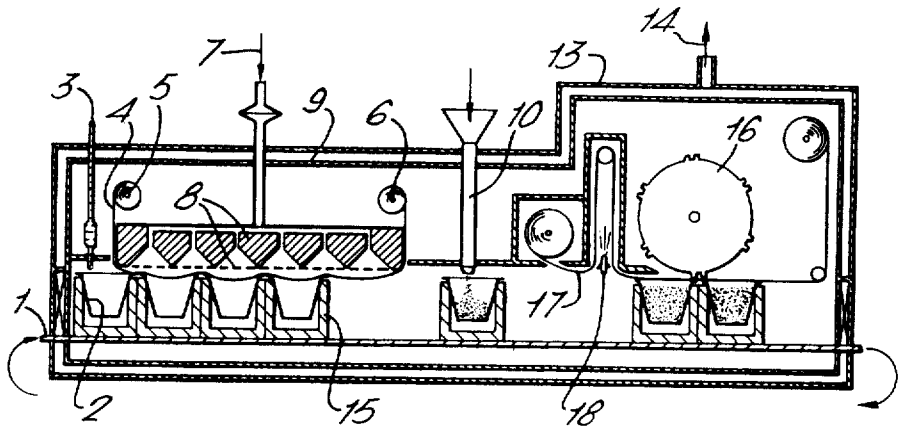


FIG. 1.

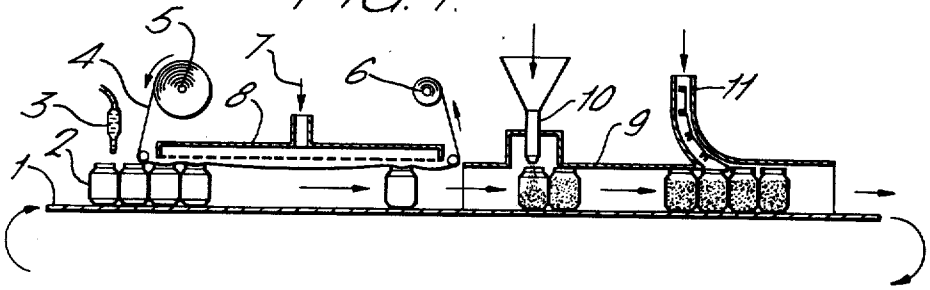


FIG. 2.

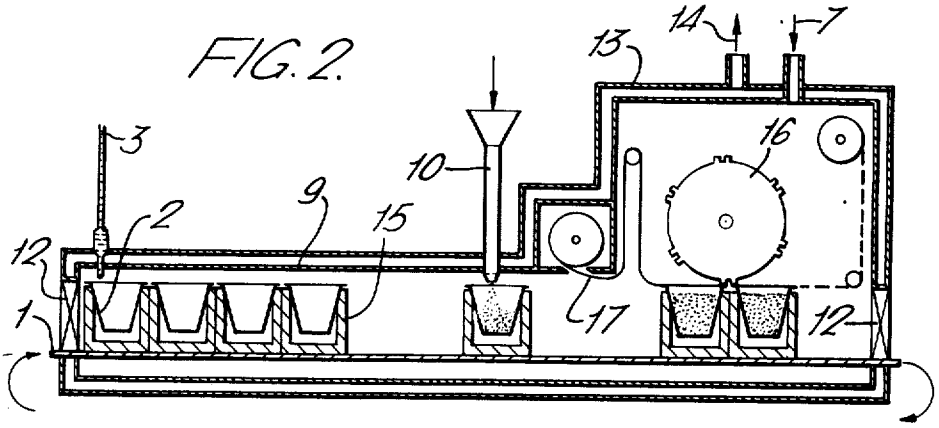
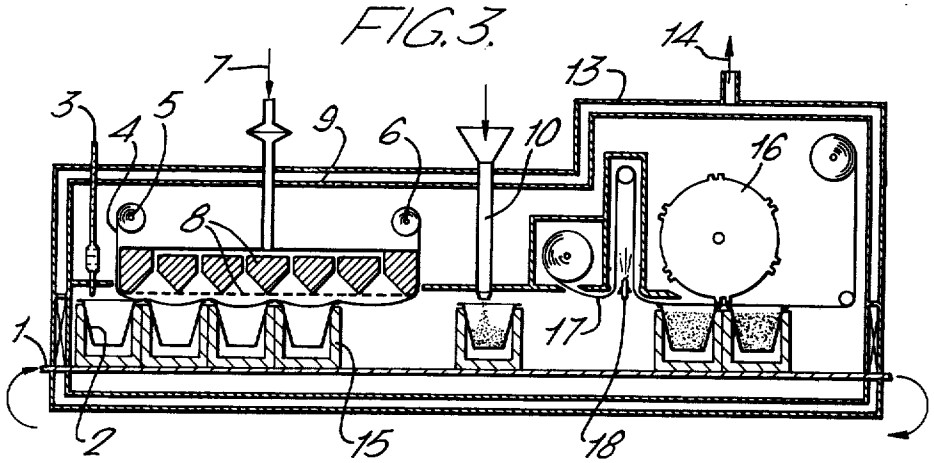


FIG. 3.



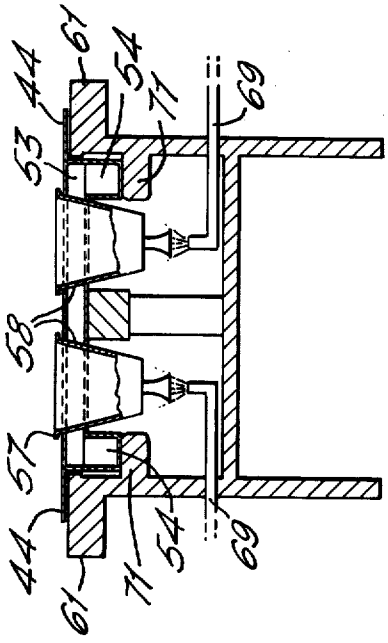

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FIG. 5.

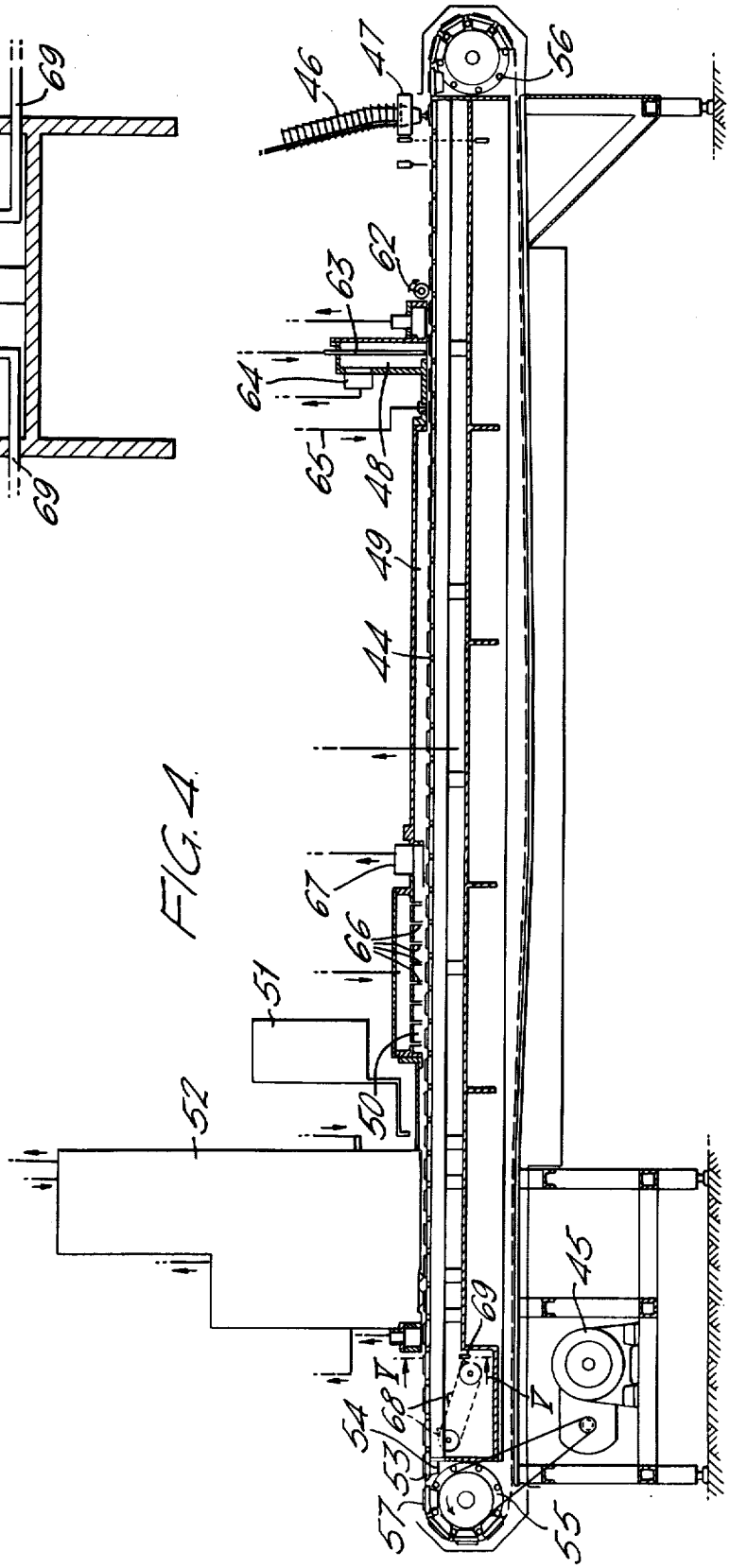
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FIG. 4.

STERILIZATION OF CONTAINERS

This application is a continuation-in-part of our co-pending application Ser. No. 131,626 filed Apr. 6, 1971, now abandoned.

The invention relates to a process and apparatus for sterilising, aseptically filling and closing containers by means of sterilising agents such as gases. By sterilisation of the containers is also understood, besides the killing of e.g. Bacillus and Closteridium spores, the specific killing of certain groups of microorganisms, e.g. osmophilic moulds and yeasts.

According to one aspect of the present invention a process is provided for continuous sterilising, aseptically filling and closing containers, in which each successive container is dosed with a quantity of a sterilising agent which is retained within the container for a sufficient time for sterilisation to take place, is conveyed through a tunnel within which a gaseous environment under slight overpressure is maintained, and is filled and has a sterile lid applied under sterile conditions within the tunnel.

Sterilisation may also take place within the tunnel to reduce any risk of contamination during the sterilising process from the ambient atmosphere.

Advantageously the lid may be formed from a strip of foil which is sterilised within the tunnel.

To accelerate the optimum sterilisation of particularly the inside surfaces of the containers and in order to minimise on the use of sterilising agent, each container may temporarily be sealed subsequent to dosing with the sterilising agent and during sterilisation so as to maintain the sterilising agent within the container during sterilisation, after which the temporary sealing is removed to allow filling.

According to another aspect of the invention an apparatus for carrying out the above mentioned process comprises a tunnel which is arranged to enclose a gaseous environment under slight overpressure, a dosing device for dosing a quantity of a sterilising agent into successive containers, a conveyor passing through said tunnel and arranged to convey successive containers past the dosing device to allow sterilisation and through the tunnel, and means located within the tunnel for the filling and for the application of a sterile lid to each successive container under sterile conditions.

The sterilising means may be arranged within the tunnel, so that sterilisation takes place within this tunnel. The lid applying means may comprise a strip of foil which is located and sterilised within the tunnel. To derive optimum result from the dosing of and to prevent wasting of sterilising agent, the apparatus may be provided with temporary sealing means for the temporary sealing of each successive container after dosing with the sterilising agent to maintain the sterilising agent within the container during sterilisation.

The temporary sealing means may be composed of a strip of foil which is arranged to be pressed over the mouth of each successive container during sterilisation. A blowing means may be provided for generating an air current to press the foil over the mouth of each successive container during sterilisation.

The foil can also be pressed over the mouths of the containers by means of a block of elastic material.

The present apparatus may also be provided with a second conveyor parallel to the first conveyor and arranged to convey an endless strip of material resistant

to the sterilising agent and keeping this strip pressed over the mouth of each successive container during sterilisation, while the speed of the second conveyor being arranged to match that of the containers on the first conveyor.

The sterilising agent may conveniently but not necessarily be hydrogen chloride mixed with air and satisfactory sterilisation times can be achieved if the partial pressure of hydrogen chloride is from about 0.5 mm Hg to about several atmospheres, preferably above 50 mm Hg, and most preferably above 100 mm Hg, at a temperature preferably between −5°C and 40°C. A partial pressure of hydrogen chloride of from 120 mm Hg to 140 mm Hg, especially a partial pressure of about 130 mm Hg, has been found to be particularly satisfactory. At these partial pressures of hydrogen chloride it is generally necessary, in order to avoid condensation on the surface to be sterilised, which condensation is undesirable because it may damage the surface or materials subsequently in contact with the surface, to reduce the partial vapour pressure of the water vapour in the air below that which occurs naturally. These partial water vapour pressures can be determined from the literature, e.g. from the International Critical Tables (F. C. Zeisberg, W. B. van Arsdel, F. C. Blake, C. H. Greenewalt and G. B. Taylor, International Critical Tables of Numerical Data, Physics, Chemistry and Technology, Vol. III, 1st Edn. National Research Council, New York, 1928).

From the foregoing it will be appreciated that dry hydrogen chloride alone could be used for sterilisation and this would generally involve a pressure equal to atmospheric. When more rapid sterilisation is required, hydrogen chloride at superatmospheric pressures may be used e.g. at six atmospheres. In such cases the advantages of rapid sterilisation may outweigh in practice the disadvantages of complicated construction to prevent the escape of hydrogen chloride gas.

It is in practice generally necessary to purge or flush the sterilisation apparatus and the containers, the surfaces of which are to be sterilised, with an inactive dry gas to remove all ambient air which is generally sufficiently humid to produce local condensation on contact with hydrogen chloride gas at the partial pressures under consideration. Practical considerations in some cases require the containers after sterilisation to be flushed with an inactive dry sterile gas to remove traces of hydrogen chloride in and around the containers, which traces of hydrogen chloride could be objectionable during subsequent storage.

When a supply of inactive dry sterile gas has to be available, it is generally more economical to use gaseous hydrogen chloride mixed with this gas, after adjusting the water vapour content of this gas, to provide the sterilising gas mixture, and also to use this gas for flushing the apparatus and containers.

It has also surprisingly been found that for water vapour partial pressures between zero and those which will cause condensation, there is a minimum partial water vapour pressure above which microorganisms are killed more quickly than they are below it. This is a new and surprising discovery since it was generally believed that there was a steady increase in rate of killing microorganisms as the partial water vapour pressure increased from zero to the point where condensation occurs. This critical partial water vapour pressure has been found to be between 1 mm and 1.5 mm mer-

cury, and appears to be substantially independent of the partial vapour pressure of hydrogen chloride.

The partial water vapour pressure of the mixture of hydrogen chloride and inactive dry gas (which conveniently is air and is hereafter referred to as air) used to sterilise containers in accordance with the present invention may be adjusted as follows:

1. The humidity of the ambient air is measured,
2. Ambient air is mixed with substantially dry air, e.g. from the reduction valve of a compressed air bottle, (e.g. internal pressure initially 100 atmospheres), both streams of air passing through bacterial filters being chosen to give the required partial water vapour pressure, i.e. a partial water vapour pressure between about 1 mm mercury and that at which condensation arises at the temperature of operation. The resulting sterile air of predetermined partial water vapour pressure is referred to as "conditioned air"; "air" as indicated above being used herein to include the use of any inert gas besides air.
3. Conditioned air and hydrogen chloride gas are then mixed together to give the desired partial pressure of hydrogen chloride gas.

The air from a normal commercial compressed air bottle, after throttling down to atmospheric pressure has a negligible partial water vapour pressure, since it will be approximately the quotient of the saturated water vapour pressure at any given temperature (compressed air bottles always contain free water), and the pressure, in atmospheres, inside the bottle.

When a larger supply of air is required, it is conveniently supplied by a pump and passes through a drier which is essentially a refrigerating unit where with the compressed air may be cooled to a controllable temperature, thereby condensing and removing excess water. Thereafter the air passes through a microbiological filter and is mixed in metered proportions with hydrogen chloride gas from commercial cylinders.

More specifically, therefore, the present invention comprises a process for the continuous sterilisation of solid surfaces of articles, particularly the surface of foodstuff containers, at a temperature preferably between −5°C and 40°C, wherein the articles undergo continuously and successively a first flushing treatment where ambient air is and around the articles is flushed away with a blast of dry inert gas, a sterilising treatment where the articles are exposed to hydrogen chloride gas, preferably hydrogen chloride mixed with 0.5 conditioned air wherein the hydrogen chloride has a partial pressure from 0.5 mm Hg to one atmosphere, immediately followed by a second flushing treatment where conditioned air is used to flush away from the articles any traces of hydrogen chloride gas.

When employing a mixture of ambient air and air from a compressed air bottle it is generally desirable to have the partial water vapour pressure of the conditioned air some fraction, say 50, 70 or 80 percent, of that at which condensation occurs, so that there is some margin for variation in the water content of the ambient air between ambient air-humidity determinations.

In a preferred manner of operation which is particularly suited to give the best results when sterilising plastic containers for foodstuff, the articles (containers) are subjected to a dust-removal treatment with ionised air prior to the first flushing treatment to accelerate the removal of dust, and the exteriors of the containers are subjected to a neutralising treatment after the second flushing treatment to neutralise any hydrogen chloride adsorbed on the external surfaces of the containers. The object of this neutralising treatment is to prevent desorption of the absorbed hydrogen chloride during storage, since otherwise, in the case of storage of large quantities of containers, this desorption of hydrogen chloride could produce in the atmosphere of the storage space undesirably high concentrations of hydrogen chloride gas. The hydrogen chloride gas absorbed in the interior of the containers is so small that it is generally absorbed by the foodstuff subsequently placed in the container without any noticeable effect on the foodstuff especially if the foodstuff is naturally acid.

In apparatus for carrying out the process of the present invention, the articles are conveniently moved continuously by means of a conveyor disposed substantially horizontally, through a number of zones or enclosures corresponding to the successive treatments indicated above and wherein these treatments are carried out. It is preferred, especially when the articles to be treated are plastic food containers, to provide the conveyor with openings of a shape corresponding closely to the upper part of the containers, so that the latter have a more or less gas-tight fit in the conveyor. In this way the contact of the exterior of the containers with hydrogen chloride gas is reduced to a minimum.

For safety reasons the apparatus may be provided with an outer casing enclosing said tunnel and retaining round the tunnel a surrounding jacket of a gaseous atmosphere which is kept under a slight underpressure.

Containers sterilised, filled and sealed by means of the above mentioned process are included in the invention.

The invention will now be further illustrated with reference to the drawing, in which:

FIG. 1 is a schematic cross-section of an apparatus according to the invention with temporary sealing foil;

FIG. 2 is a cross-section of another construction of the apparatus without temporary sealing means;

FIG. 3 is a cross-section of a third embodiment of the apparatus provided with a double encasing and temporary sealing means;

FIG. 4 is a part longitudinal sectional elevation of a preferred type of apparatus without temporary sealing means.

FIG. 5 is a part transverse sectional elevation on the line V—V of FIG. 4 and shows means for applying an alkaline solution to the exterior of sterilised containers.

The process and apparatus to be further described now are particularly suitable for so-called "cold" sterilisation. The sterilising agents useful in this respect include e.g. gaseous hydrogen halides, in particular hydrogen chloride, ethylene oxide, ethylene imine, Cl_2 and maybe these agents in gaseous form or maybe gases evaporating from the liquid state.

A tunnel is particularly advantageous in this invention since it provides a convenient enclosure in which the environment can be kept under the required conditions for a continuous process.

In the figures, 1 denotes a conveyor, here in the form of an endless conveyor belt (not completely shown) of a material determined by the choice of the sterilising gas, e.g. coated metal or PVC. This conveyor serves to convey the containers 2 to be sterilised and filled, the

containers passing under a dosing device 3 for dosing either a jet of undiluted sterilising gas or a microdroplet of a gas-generating liquid. The dosing device is connected to a photo-electric cell by means of an auxiliary circuit, which assembly permits the dosing of a small, reproducible amount of the sterilising agent, e.g. hydrogen chloride gas or a concentrated hydrochloric acid solution, into each open container at the moment that it passes under the nozzle 3. In a reliability test of more than 40,000 dosings, which was faultlessly performed in glass jars having a neck opening of 60 mm, speeds were obtained of 70 jars/min or 4,200 jars/h.

After dosing the required amount of the sterilising agent, the containers should preferably be kept closed by a temporary sealing means for a certain time, in order to effect optimum sterilisation of the inside surfaces and in order to prevent wasting of sterilising agent.

The temporary sealing operation may be effected in various ways. A foil 4 of material resistant to the gas used can be made to extend over the container openings. For this purpose a number of materials can be used. For the present application polyethylene foil, polypropylene foil or PTFE (Teflon) were used. In order to enable renewal of this material, in case of possible wear owing to contact with the containers passing under it, the foil is led from a supply roller 5 to an uptake-roller 6.

The foil can be kept pressed over the mouths of the containers during the sterilisation period by means of a blowing device 7, generating a sterile air current, which is divided by a divider 8 over the foil surface to be blown as shown in FIG. 1. The divider 8 is shown in more detail in FIG. 3.

The temporary sealing operation can also be achieved by pressing the foil over the mouths of the containers during sterilisation by means of a block of elastic material (not shown in drawing). Finally, this is also possible by means of a second conveyor which conveys an endless strip of material resistant to the sterilising agent, e.g. impermeable PVC-foam. The rotating speed of the strip should in this case match, i.e. be substantially equal to that of the containers on the first conveyor belt. This embodiment is not illustrated in the drawing either.

When leaving the strip of covering material as shown in FIG. 1, the containers on the conveyor should immediately come into a tunnel 9, in order to prevent infection. It is preferred to accommodate the whole plant in a tunnel, also indicated by 9 in FIGS. 2 and 3, which by means of a predetermined mixture of air, and a sterilising gas is kept under a slight excess pressure. In this case the blowing device 7 can as well be used to supply the mixture which keeps the whole space under the slight excess pressure (FIG. 2), as retain its function of blowing the foil (FIG. 3). The enclosed tunnel 9 preferably is provided with an entrance and exit device 12 (FIGS. 2 and 3), which may have any desired construction. The air used for the mixture in the sterilising space should have a relative moisture content such that no condensation of the sterilising agent can occur. When no temporary sealing means are used, as shown in FIG. 2, it is preferred to have blowing device 7 near the exit of the apparatus. This is done in order to avoid turbulence around the containers at the beginning of the sterilisation process, which turbulence could cause still live spores to be blown out of the containers e.g. towards the filling device. For reasons of safety the apparatus should additionally be surrounded by an outer casing 13 within which a slight under-pressure is permanently maintained, so that any escaping sterilising gas is exhausted at 14 to a safe place.

The tunnel may be subdivided into separated compartments, e.g. where different compartments need different gaseous environments.

Dependent on the kind of product to be filled in the container and its compatibility with the sterilising agent it may be desirable to wash out each successive container after sterilising and prior to filling, e.g. by means of a jet of sterile air.

Before the dosing of the filling mass begins, the whole tunnel 9 and the device 10 are to be sterilised, preferably with hydrogen chloride gas. For the sterilisation of the sealing material of the containers 2 use can be made of steam or hydrogen peroxide, HCl gas also being preferred for this purpose.

The containers 2, shown in FIG. 1 as glass jars, are sealed with sterilised lids, as indicated schematically at 11, and subsequently discharged from the apparatus by the conveyor belt.

The containers 2 indicated in FIGS. 2 and 3 may e.g. be cups of synthetic material, which may have to be placed into supporting moulds 15. It is schematically shown how these cups are provided with closure from a foil 17 and sealed by means of sealing device 16. As indicated at 18, the foil may be additionally sterilised with a jet of hydrogen chloride gas before the dosing operation.

Turning now to FIG. 4, a conveyor belt 44 driven by a motor 45 conveys plastic beakers 46 of the type used for yoghurt from a delivery point 47 through a first flushing zone 48, a sterilisation zone 49, a second flushing zone 50 to filling and closing devices 51 and 52, which devices are indicated diagrammatically since their detailed construction does not form part of the present invention. The conveyor belt 44 is an endless flexible belt of material resistant to hydrogen chloride, e.g. PVC reinforced with polyester gauze, or a glass fibre fabric impregnated with PFTE. At regular intervals along this belt are fixed plates 53 of hard plastic material, e.g. hard PVC or polyester. These plates have projections or teeth 54 engaging respectively driving and idling sprockets 55, 56 and also have openings 57 registering with openings 58 in the belt 44, which openings have an internal profile adapted to fit closely round the upper parts of the beakers 46 as shown in FIG. 5. By having openings of varying internal profiles, the conveyor belt 44 may be made to handle beakers of varying shapes and sizes. By having the beakers fitting closely in the openings 58 and edges of the flexible belt 44 having rubbing contact with outwardly projecting flanges 60 forming part of a machine frame 61 (see FIG. 5), the penetration of hydrogen chloride gas to the lower side of the upper run of the belt 44 is reduced to a minimum. Inwardly projecting flanges 71 support the teeth 54.

At the delivery point 47 the beakers are released from a stack of nested beakers and fall into the openings 58. Known means, such as photoelectric means, are provided to ensure that each ring receives a properly positioned beaker, or to give a signal, or stop the conveyor 44 is a beaker if missing from an opening or improperly placed therein. A roller 62 ensures that the beakers are securely seated in the openings 58 with

flanges at the tops of the beakers overlapping the upper edges of the openings.

In a first flushing zone **48**, a jet of dry air is directed through pipe **63** into the interior of a beaker **46** to remove the ambient air therein. A vent **64** allows the outflow of air from this zone and the arrangement is preferably such that a gauge pressure of 0.05 mm H_2O is maintained in this zone. Thereafter the beakers pass through the sterilisation zone **49** which is fed via supply line **65** with the required mixture of hydrogen chloride gas and air and a water gauge pressure of **0.32** mm is maintained on this zone. It will be noted that the beakers **46** move in counter-current to the flow of sterilising mixtures.

The beakers next pass to the second flushing zone **50** wherein a plurality of nozzles **66**, supplied with dry sterile air from a chamber fed by supply line. A vent line **67** allows the escape of a mixture of flushing-air and sterilising mixture.

The beakers are finally filled at **51** and closed at **52**, e.g. with sterilised foil lids, and ejected by an ejection device **68**. whereupon they pass to storage.

Under the conditions indicated above it has been found satisfactory for the beakers to be for **30** seconds in the sterilising zone **49** and **12** seconds in the second flushing zone **50**.

FIG. **5**, also shows inlet pipes **69** whereby an alkaline fluid, e.g. an aqueous solution of sodium bicarbonate, may be sprayed around the external surface of the beakers **46** after they have left the closing device **52**.

We claim:

1. A process for continuously sterilizing, aseptically filling and closing containers in a low-moisture environment maintained at substantially ambient conditions comprising the steps of subjecting the containers to a first flushing treatment wherein dry gas moves relatively to said containers to flush away ambient air in and around said containers, subsequently subjecting the containers to a sterilizing treatment wherein said containers are sterilized by the action of hydrogen chloride gas, subjecting the containers to a second flushing treatment wherein conditioned air moves relatively to said containers to remove free hydrogen chloride from the surface thereof, filling the container, effecting a sterile closure of said containers and substantially removing from the external surface hydrogen chloride absorbed thereon.

2. A process as claimed in claim **1**, in which the external surfaces of the containers are subjected to a treatment with an agent which neutralizes the hydrogen chloride absorbed thereon.

3. A process according to claim **1**, in which the external surfaces of the containers are sprayed with an alkaline solution.

4. A process according to claim **1**, in which the external surfaces of the containers are flushed with an inactive gas.

5. A process as claimed in claim **1**, in which a mixture of hydrogen chloride gas and conditioned air is used in the sterilising treatment and the partial pressure of the hydrogen chloride gas is at least 100 mm Hg.

6. A process as claimed in claim **1**, in which the sterilising treatment is carried out in 30 seconds.

7. A process as claimed in claim **1**, in which the dry gas used in the first flushing treatment is conditioned air.

8. A process as claimed in claim **1**, in which prior to the first flushing treatment, the containers are subjected to a stream of ionised air to remove dust from the surfaces of

9. A process as claimed in claim **1**, in which after the second flushing treatment, the containers are sprayed with an alkaline solution to neutralise hydrogen chloride adsorbed on the exterior surfaces of said containers.

10. A process for continuously sterilising, aseptically filling and closing containers in a low moisture environment maintained at substantially ambient conditions comprising the steps of placing the containers on a moving conveyor; moving the containers past a sterilising agent dosing device; dosing the containers with a sterilising agent which does not substantially increase the temperature of the container; temporarily sealing the container; retaining the sterilising agent in the container for a time sufficient for sterilisation to take place; moving the containers into a sterile tunnel within which a gaseous environment is maintained at slight over pressure; removing the temporary seal; filling the container; applying a sterile lid to the container; and moving the closed container out of the tunnel.

11. An apparatus for continuously sterilising, aseptically filling and closing containers in a low moisture environment maintained at substantially ambient conditions comprising a sterilising agent dosing device which delivers a measured amount of a sterilising agent into the containers; means to temporarily seal the container after dosing with the sterilising agent; the sterilising agent being retained in the container for a time sufficient to sterilise them without substantially increasing the temperature of the containers; a sterile tunnel within which a low moisture gaseous environment is maintained at slight overpressure, said tunnel containing means to remove the temporary seal, to aseptically fill the containers and means to emplace a sterile lid on the containers; and, a conveyor for receiving the containers and moving them serially past the sterilising agent dosing device, the filling means, the lid emplacing means and out of the tunnel.

12. An apparatus according to claim **1**, in which the temporary sealing means comprises a strip of foil which is arranged to be pressed over the mouth of each successive container during sterilisation.

* * * * *