

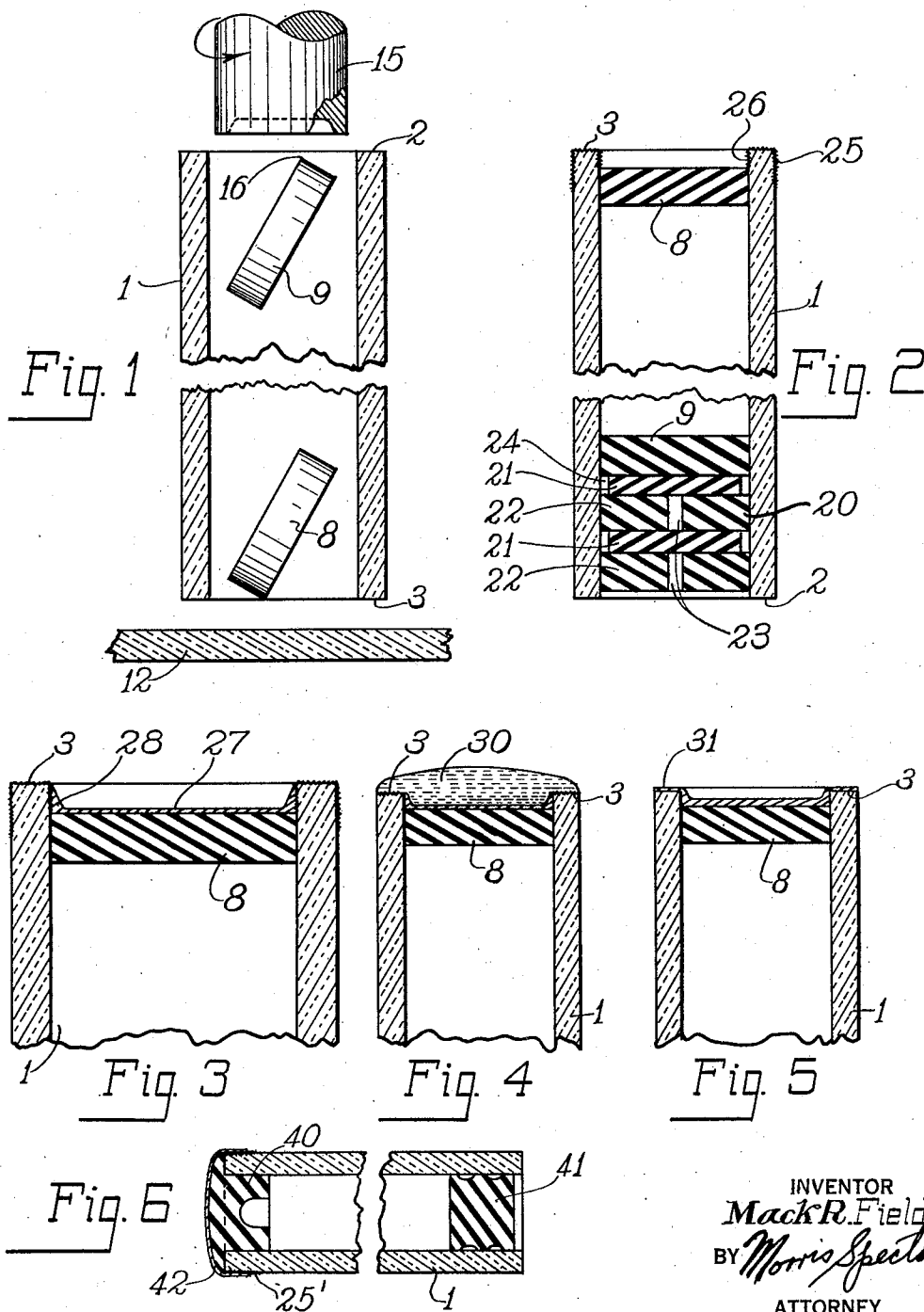
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AMPULE

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AMPOULE

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11 Claims. (Cl. 128—272)

This invention relates to ampoules such as are used with syringes for hypodermic or other injections, particularly dental ampoules.

Ampoules of the type with which the present invention is concerned comprise a tube, generally of glass, which is closed at both ends by closure plugs. One of the closure plugs comprises a piston which may be forced into the tube to eject the liquid contents thereof through a hypodermic needle, which needle pierces either the piston closure or a pierceable plug at the opposite end of the tube.

In dealing with ampoules of the type wherein the hypodermic needle pierces a closure at an end of the tube opposite the piston plug, certain difficulties are frequently encountered due to a plugged hypodermic needle. As pressure is applied to the piston closure to force it into the tube the liquid within the tube is placed under considerable pressure, especially if the contents of the tube cannot escape through the hypodermic needle. Frequently the closure opposite the piston plug is forced out of the tube, thus spilling the contents thereof and frequently, also, spraying the patient with the medicine.

It is one of the objects of the present invention to provide a pierceable closure for ampoules of the above mentioned character, and means for holding the closure in place against expulsion under any pressures that might reasonably be exerted thereagainst by the piston plug, even when the contents of the ampoule are confined against escape.

It has heretofore been proposed to cover the outside of the pierceable closure plug with a thin layer of plastic material which extends over and onto the adjacent part of the glass tube. It has been found that such a film as heretofore applied cannot hold the pierceable plug in place under appreciable pressure. It is one of the objects of the present invention to provide means for causing a sealing film to adhere to the glass of the tube with such tenacity and such form as to positively prevent expulsion of the pierceable piston plug and yet at the same time not interfere with the piercing thereof by a hypodermic needle. In accordance with one preferred embodiment of the present invention the end of the glass tube is etched to provide a roughened surface against which a sealing film will adhere tenaciously. The closure plug or disc is positioned in the ampoule tube with the end of the ampoule tube projecting beyond the end of the closure for a very short distance, a distance of the order of $\frac{1}{2}$ of an inch or more. This provides a cavity within which a

bit of thermo-plastic material may be placed and which will adhere to the closure disc and also adhere to the etched surface of the tube wall and form a thickened portion at the interior surface of the tube wall to prevent shearing of the thermo-plastic layer by an outward force exerted thereagainst by the closure plug.

The preferred plastic film is one which has the property of adhering to glass with great tenacity, one which is easily pierceable by a hypodermic needle and will not dull the needle, one which is not frangible and will not break off when it is pierced by the needle and thus plug the needle opening during piercing, and one which is sufficiently flexible to prevent shearing of the plastic layer at the glass ampoule under pressure. I have found that an acrylic resin, known as "Acryloid B-72," is a suitable material. It is a toluol solution and in its commercial liquid form is 30% solid and 70% solvent. Toluol is a rubber solvent and therefore as the liquid plastic is drying the toluol dissolves some of the rubber of the closure plug with which it is in contact so that upon complete drying of the plastic the plastic is integrally united with the rubber.

Heretofore it has been suggested to coat the end of an ampoule with plastic by dipping the ampoule into the liquid plastic in solution. I have found that the dipping process is not applicable efficiently for coating the end of a tube wherein the closure disc is recessed within the tube. In a recessed structure dipping of the end downward into a viscous liquid plastic traps a bubble of air, preventing the spread of the liquid plastic outwards from the center and upwards. It is essential that the liquid plastic spreads in an unbroken film without any trapping of minute air bubbles along the juncture of the rubber plug and the wall of the tube. In accordance with the principles of the present invention this difficulty is overcome by avoiding the dipping operation entirely. Instead the tube is held vertically with the end to be coated located uppermost, and one or several drops of liquid plastic is deposited in the recess formed by the inset disc in the end of the tube. As the plastic solvent dries, the remaining solid material adheres to the surface of the glass tube and forms a layer which is appreciably thicker around the periphery of the disc than at the center. This acts as a positive stop to prevent forcing of the closure plug outwardly of the tube, since such forcing action would necessitate shearing of a thick mass of thermo-plastic material.

In order to facilitate the formation of a thick-

ened rim of plastic material in proper position around the inner periphery of the glass tube adjacent the outer surface of the closure plug, the glass tube is square ground at its end as distinguished from the usual flamed or rounded end of the glass tubes heretofore used. This facilitates formation of a thick layer of thermoplastic along the periphery of the closure plug and assures holding of the same against outward ejection.

It is a further object of the present invention to use a closure plug of sheet rubber material as distinguished from the usual molded closure plugs. Rubber is inherently porous. When sheet material is used it is possible to treat the surfaces of the rubber to close the pores. This may be done before the sheet material is cut or stamped to form the individual closure plugs. Such treating may comprise calendering, or coating a surface of the sheet with an inert material such as wax, a thermo-plastic, or the like. This provides a non-porous closure plug for the tube. In addition to the porosity of the molded plugs the rubber itself is not entirely chemically inert with the solution in the tube. The use of sheet material makes possible an inexpensive method of treating chemically or applying an inert film to the sheet to render inert the surface of the subsequently cut disc that is to be in contact with the chemicals in the tube.

It is a still further object of the present invention to provide an improved method of filling and sealing an ampoule. In accordance with the preferred method rubber discs or diaphragms, which are ultimately to constitute sealing closures, are positioned in opposite ends of the ampoule at a substantial angle to the axis thereof so as not to prevent the flow of fluid into and through the ampoule tube. This may be done under non-sterile conditions. Thereafter, superheated steam is passed through the tube to sterilize the tube and the closure diaphragms, and then the ampoule may be filled with its sterile solution and the diaphragms righted. The diaphragms then provide temporary seals for maintaining the contents of the tube sterile while further operations for completing the ampoule are performed.

It is another object of the present invention to provide an improved piston closure plug for an ampoule. The piston is preferably made of a plurality of parts which includes a facing diaphragm that is capable of effecting a temporary sealing closure at the end of the ampoule. In the manufacture of the ampoule the diaphragm is positioned in the tube and at an angle to the axis thereof to permit the free passage of fluid past the facing diaphragm. This is done before sterilization. After sterilization the diaphragm is righted to seal the end of the tube. The rest of the piston closure, of which the diaphragm is to constitute the face, may then be assembled. The rest of the piston preferably comprises discs of rubber which form a seal with the tube wall, and discs of fiber or the like between the rubber discs, for assuring that the pressure is transmitted from one rubber disc to the next in an axial direction. This eliminates, or very greatly reduces, the tendency for a long piston to bind or lock with the surface of the ampoule tube when pressure is exerted thereagainst.

The attainment of the above and further objects of the present invention will be apparent from the following specification taken in conjunction with the accompanying drawing forming a part thereof.

In the drawing:

Figure 1 is an enlarged longitudinal sectional view through a partially completed ampoule and illustrating one step in the process of completing the same;

Figure 2 is a longitudinal sectional view through a partially completed ampoule;

Figure 3 is a longitudinal sectional view through one end of the ampoule at a later stage in the process of manufacture;

Figure 4 is a longitudinal sectional view similar to Figure 3 and showing the ampoule in a still later stage in the process of making the same;

Figure 5 is a longitudinal sectional view through the same end of the completed ampoule; and

Figure 6 is a longitudinal sectional view through another type of ampoule embodying the present invention.

Reference may now be had more particularly to Figure 1 of the drawing. The ampoule of the present invention comprises a tube 1 of glass or other suitable material, which tube is circular in cross section and of uniform diameter throughout. The opposite ends 2-3 of the tube are ground to be perfectly flat and at right angles to the longitudinal axis of the tube, thus forming right angle corners between the surfaces 2 and 3 and the inner and outer peripheral walls of the tube 1. Because the tube is ground perfectly flat at the end surfaces 2 and 3 these surfaces can be located a fixed and predetermined distance apart, thus facilitating quantity assembly and filling of the ampoules.

Two end closure discs or diaphragms, indicated at 8 and 9, are then inserted into the tube. The diaphragms 8 and 9 are formed of pure rubber. The diaphragms may be cut from a rubber sheet one or both surfaces of which may have been treated to render the surface coming into contact with the solution non-porous and chemically inert with the solution. Such treatment may consist of a glazing, coating with a wax or plastic or other inert sealing film, or the like. Only one surface of the sheet need be thus treated. Each diaphragm is inserted into the tube 1 so that the treated end will ultimately be the end in contact with the solution within the tube. The diaphragms 8 and 9 are circular in cross section and of a diameter slightly in excess of the internal diameter of the tube. They are inserted into the tube at an acute angle to the longitudinal axis of the tube, as illustrated in Figure 1, so that they do not close the ends of the tube but they are nevertheless held firmly in position within the tube, since they are slightly oversized.

The interior of the tube may then be sterilized. This may be accomplished by passing live superheated steam through the tube. The tube and the closure discs 8 and 9 are thus completely sterilized. The tube 1 may then be placed in firm contact with a sterile sheet or plate 12 of yielding and moisture-proof material so that the end 3 forms a temporary seal with the plate 12. The tube is then filled with the sterile medicine solution, being filled preferably to a height above the top of the disc 9 but certainly over the center of the disc 9. The disc 9 is then righted to a position extending across the tube 1. This may be accomplished by a cylindrical plunger tool 15 of a diameter slightly less than the internal diameter of the tube 1 so that the tool can be inserted freely into the tube 1. The lower face of the plunger 15 may be flat across, or recessed as illustrated.

The tool or plunger 15 is mounted in a suitable mechanism adapted to rotate it axially. The slowly rotating tool 15 is then lowered into the tube 1. The tool first engages the top 16 of the disc 9 and as the tool is further lowered, and rotated, it rotates the disc 9 to a position such that its plane is at right angles to the axis of the tube 1, my pending application, Serial No. 340,329. This, as is pointed out above, is done after the tube has been filled with its solution. The disc or diaphragm 9 is then in a position within the tube such as illustrated in Figure 2. The tool 15 is then retracted from the tube 1 and the tube, with the contents thereof, and the closure sheet or plate 12 are reversed so that the disc or diaphragm 9 is now uppermost. The rotating tool 15 is then again lowered into the tube 1 to right the diaphragm 9 in the same manner that it righted the diaphragm 9. Thereafter the diaphragm 9 may be pushed into the tube 1 to push the diaphragm 9 closer to the edge 3 until ultimately the diaphragm 9 is positioned at a distance from the edge 3 of the tube 1 as illustrated in Figure 2. In this position the outer surface of the diaphragm 9 is at a distance approximately $\frac{1}{2}$ of an inch from the end 3 of the tube. This is approximately half the thickness of the diaphragm 9.

The diaphragms 8 and 9 afford temporary seals to maintain the contents of the tube sterile. It is now necessary to convert these temporary seals into permanent seals. This may be accomplished under ordinary non-sterile conditions since the contents of the tube are maintained sterile and temporarily sealed from the atmosphere. One end of the tube is built up to constitute the diaphragm 8 a facing surface of a slidable piston plunger 20 in the manner described in my pending application Serial No. 340,329, to which reference may be made for a further description. The other end is closed off to form a permanent seal adapted to be pierced by a hypodermic needle. Either of the two ends may be sealed first.

Briefly, the piston plunger assembly, indicated at 20, comprises the diaphragm 9, two serrated rigid fiber discs 21 of a maximum outside diameter slightly less than the inside diameter of the tube 1, and rubber closure plugs 22 making snug fits with the interior wall of the tube and having central holes 23 therein. The serrations 24 in the discs 21, and the outer surfaces of the rubber closure plugs may be coated with glycerine which is medicated with a germicide and bactericide so that the glycerine acts as a lubricant to facilitate insertion of the closure plugs. As each closure plug 22 is assembled within the tube 1 the opening 23 therein constitutes an air passageway for the expulsion of air between the plug 22 and the ends adjacent the serrated disc 21. Since the plugs 22 are never in contact with the medicine within the tube 1 they do not have to be made of a material which is resistant to the action of the medicine and which is chemically inert therewith. The plugs 22 may therefore be made of a cheap grade of rubber.

An explanation will now be given of the manner of converting the temporary closure 8 into a permanent seal. The tube is held with the end 3 lowermost and that end is then dipped into an etching solution to etch the end of the tube. The solution may comprise hydrofluoric acid which etches the glass but is inert with respect to the rubber 8. The glass is etched along its outer periphery indicated in exaggerated form by the roughened part 25 and at its inner periphery as

similarly indicated at 26. The end of the tube is then washed and the tube is then held in a vertical position with the diaphragm 9 uppermost. It is to be understood that the etched part 25—26 is not as rough as illustrated, the same being exaggerated for illustrative purposes. A drop or globule of suitable thermo-plastic material is then dropped on the diaphragm 9. The globule immediately spreads out along the upper surface of the diaphragm 9 to the wall of the tube 1 and rises along the periphery of the tube to the end 3. This may be due to a surface tension or capillary action phenomenon. As the liquid contents of the thermo-plastic globule evaporate there is left a thin film 27 which overlays and is adhered to the upper surface of the rubber diaphragm 9 and which also rises along the edge of the inner surface of the tube 1 to form a ridge or ring 28. This ridge or ring firmly adheres to the roughened or etched surface 26 of the tube and forms a liquid-tight, air-tight seal and also forms an abutment ring to prevent the expulsion of the diaphragm 9 by pressure within the ampoule 1. A preferred thermo-plastic material is an acrylic resin known as "Acryloid B-72." It is a toluol solution and in its commercial liquid form is 30% solid and 70% solvent. Toluol is a rubber solvent and therefore as the liquid plastic is drying, the toluol dissolves some of the rubber of the closure diaphragm with which it is in contact so that upon complete drying of the plastic the dried plastic is integrally united with the rubber. The pierceable closure end of the tube 1 is now in the condition illustrated in Figure 3. Thereafter another drop or globule of the same material as the globule 25, but considerably thinned, as by the addition of toluol, so that it contains less solid and more solvent, is deposited upon the film 27. This substance may be 20% solid and 80% solvent. It assumes the shape illustrated at 30 in Figure 4 overlying the end surface 3 of the tube. Thereafter as the solvent evaporates it forms a thin film 31 which overlies the end surface 3 of the tube 1 and is adhered thereto and extends over and is integrally united with the previously formed film or layer 27. There is thus formed a liquid-tight, air-tight seal over the end of the diaphragm 9, which seal is firmly bonded to the glass 1 and includes the reinforcing ring 28 along the inner periphery of the glass tube, which ring is bonded to the etched surface of the glass to hold the ring firmly in place, and which ring acts as an abutment against expulsion of the diaphragm 9. The thermo-plastic film is non-frangible and will not break off when it is pierced by the hypodermic needle and thus will not plug the needle during piercing. It has sufficient flexibility to prevent shearing of the plastic layer when the contents of the glass ampoule are placed under pressure. If any part of the thermo-plastic globule 30 should run down a short distance along the side of the tube 1 it will adhere to the etched surface 25 thereof, thus helping to hold the thermo-plastic film in place and avoiding the formation of a loose edge which might otherwise cause peeling of the thermo-plastic film. The etched surface 25 also provides a place for placing an identifying mark on the tube.

When the tube 1 is inserted into a hypodermic syringe the hypodermic needle is caused to pierce the diaphragm 9. Thereafter a plunger of the syringe presses against the piston comprising the diaphragm 9, the discs 21—22 and the plugs 22—22 to force the piston into the tube to

eject the liquid contents thereof through the hypodermic needle. At that time the piston is placed under pressure by the plunger of the syringe and the piston places the liquid contents of the cartridge 1 under pressure. The material 9 and 22—22 being compressible, and being placed under axial pressure, will tend to expand radially and thus exert a greater pressure against the inner wall of the tube 1. The spaces between the serrations 24 in the discs 21—21 as well as the spaces 23 in the plugs 22—22 constitute spaces for receiving some of the rubber of the diaphragm 9 and the plugs 22—22 which are being compressed. This reduces the pressure that would otherwise be applied to the wall of the tube 1 by the rubber of the diaphragm 9 and the plugs 22—22, thus reducing the tendency for the rubber to bind to the glass tube. By building up the piston of thin layers 9, 22—22 of rubber separated by non-rubber discs 21—21, the pressure is transmitted through the rubber plugs 22—22 in an axial direction throughout, thereby further reducing the tendency for the rubber to bind against the wall of the tube as the rubber is compressed by the pressure which forces the piston inwardly. Furthermore, because the plugs 22—22 are comparatively thin, they may be formed by stamping the same out of sheet rubber, which is appreciably cheaper than molded rubber.

In Figure 6 I have illustrated another standard type of ampoule to which the present invention is applicable. This ampoule is of the type illustrated, for instance, in United States Patent No. 1,715,771 and includes a glass tube circular in cross section having a pierceable closure 40 at one end adapted to be pierced by a hypodermic needle and having a piston plug 41 at the opposite end adapted to be forced into the tube to expel the liquid contents thereof through a hypodermic needle that pierces the closure 40. In a tube of this type there is considerable danger that the closure plug 40 will be forced out of the tube by the pressure within the tube in the event that the piston 41 is forced in the tube while no needle has pierced the closure 40 or if the canula in a piercing needle is plugged. In order to prevent such expulsion the end of the glass ampoule may be dipped into a glass etching solution such as hydrofluoric acid, to etch the end of the glass, as indicated at 25'. The solution used is one which does not react chemically with the rubber closure 40. The etched surface 25' extends around the entire periphery of the glass tube adjacent the end thereof. Thereafter the outside of the tube is washed clean and may be coated with a film 42 of thermo-plastic material in any desired manner as, for instance, by dipping the tube into a suitable thermo-plastic medium such as above described, or the tube may be held vertically and the thermo-plastic material may be dropped on the outer end of the closure plug. If a thick film is desired repeated dippings may be resorted to alternating with drying periods. The film 42 extends over the etched surface 25' of the ampoule which constitutes a foundation for the film 42 so that the film holds tenaciously to the end of the glass and thus holds the plug 40 against being forced out of the tube.

This application is a continuation in part of my co-pending application, Serial No. 340,329, filed June 13, 1940.

In compliance with the requirements of the patent statutes I have here shown and described a few preferred embodiments of my invention. It is, however, to be understood that the inven-

tion is not limited to the precise constructions here shown, the same being merely illustrative of the principles of the invention. What I consider new and desire to secure by Letters Patents is:

1. A liquid-tight container comprising a tube open at at least one end, the part of the tube adjacent the open end being etched around the entire periphery thereof to provide a roughened surface, and a liquid-tight coherent film of plastic material extending across and sealing the opening and adhered to the roughened surface to hold the film against rupture by internal pressure.

2. A container comprising a tube open at an end, the inner peripheral surface at the open end of the tube being etched to provide a roughened surface, a closure disc closing said one end of the tube with the outer surface of the closure disc located within the tube so that the end of the tube projects beyond the outer surface of the disc, and an air-tight film adhered to the outer surface of the disc and to the adjacent inner periphery of the tube at said roughened surface.

3. A container comprising a tube open at both ends, a piston plug closing one end of the tube, a closure disc closing the opposite end of the tube with the outer surface of the closure disc located within the tube so that the end of the tube projects beyond the outer surface of the disc, and a film of self setting air-tight material adhered to the outer surface of the disc and to the adjacent inner periphery of the tube, said film being thickened at its outer edge to constitute an abutment stop to hold the disc against expulsion by pressure within the tube.

4. An ampoule comprising a tubular body of uniform cross section throughout and having a pierceable closure disc at one end adapted to be pierced by a hypodermic needle, the outermost end of the disc being within the tube, and an abutment ring within the tube and on the outer side of the disc and anchored to the tube for holding said disc against expulsion by pressure within the tube.

5. An ampoule for a hypodermic syringe, said ampoule comprising a tubular body having within one end thereof a pierceable closure diaphragm adapted to be pierced by a hypodermic needle, said diaphragm being of rubber-like material which is self sealing to seal around a hypodermic needle piercing the same and to close the needle opening upon retraction of the needle, and means for sealing the ampoule against leakage around the periphery of the diaphragm, said sealing means comprising a non-porous sealing film over the outside of the diaphragm and sealed and anchored to the adjacent tube wall around the entire periphery of the diaphragm, the end of the tube wall being etched, and the film being anchored along its periphery to the etched part of the tube.

6. The method of making an ampoule which comprises inserting into a tube a diaphragm of compressible sealing material of a diameter slightly in excess of the diameter of the tube and at a substantial angle to the normal to the longitudinal axis of the tube to permit the passage of fluid past that diaphragm, sterilizing the tube and the diaphragm by passing superheated steam through the tube holding the tube vertical with the inclined diaphragm end uppermost, placing a closure over the lower end of the tube, filling the tube, and righting the inclined diaphragm.

7. The method of filling and closing a container having a tubular filling opening which comprises,

Inserting into the opening a disc of compressible sealing material of a size slightly in excess of the size of the tubular opening and at an angle to the axis of the opening so that the disc does not close the opening, then filling the container by passing the filling material through the space between the interior wall of the tubular opening and the inclined disc, then tilting the disc to a position closing the opening.

8. An ampoule comprising a tubular body and a piston closure in the body adapted to be moved therein to eject the contents thereof, said piston closure comprising a facing diaphragm of rubberized material and a backing therefor comprising alternate discs of compressible and incompressible materials, the compressible discs being radially compressed by and in frictional engagement with the wall of the tubular body, certain of said backing discs providing air spaces to allow for yielding movement of the compressible material as the piston closure is forced into the tube.

9. The method of sealing a container having a tubular filling opening which comprises, placing a disc of compressible sealing material of a size slightly in excess of the size of the tubular opening entirely within the container and at a substantial angle to the axis of the opening so that the disc does not close the opening, then tilting the disc to a position sealing the opening whereby the disc is positioned across the opening and substantially spaced from the adjacent end of the container, and then forming a second seal at the other end of said container.

10. The method of sealing a container having a tubular filling opening which comprises, placing

a disc of compressible sealing material of a size slightly in excess of the size of the tubular opening entirely within the container and at a substantial angle to the axis of the opening so that the disc does not close the opening, then tilting the disc to a position sealing the opening by pressing the disc inwardly of the container at an off-center point on the disc above the center thereof, whereby the disc is positioned across the opening and substantially spaced from the adjacent end of the container, and then forming a second seal at said adjacent end of the container thereby sealing the outer end of said disc from the atmosphere.

11. The method of making an ampoule which comprises inserting into each end of a tube a diaphragm of compressible sealing material of a diameter slightly in excess of the diameter of the tube, positioning each diaphragm adjacent its end of the tube and at a substantial angle to the normal to the axis of the tube to permit passage of fluid through the tube past the diaphragm, forcing a sterilizing fluid through the tube, then temporarily closing one end of the tube, filling the ampoule with liquid to a level above that of the diaphragm at the opposite end, then turning that diaphragm in the tube to a position normal to the tube axis to effect a liquid-tight closure of the tube, then reversing the tube with the liquid contents, removing the temporary closure, and then turning the second diaphragm to a position normal to the tube axis to effect a liquid-tight closure of both ends of the tube.

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