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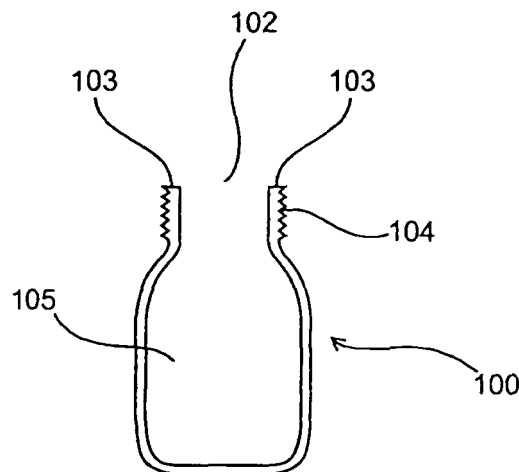
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(54) **Sealing devices**

(57) Devices and methods are provided. Embodiments include devices (2) for sealing an opening of a container, where such container sealing devices (2) may include a flexible sealing element (4) dimensioned to cover the opening of a container, a compliant channel (6) in

the flexible sealing element, and a compression member (20) for maintaining the compliant channel in a sealed configuration, regardless of whether a fluid transfer member is present in the compliant channel. Also provided are containers sealed by the subject sealing devices, and methods of using the same.



**FIG. 1**

## Description

### BACKGROUND OF THE INVENTION

[0001] A variety of different devices have been developed for sealing a container opening, for example to seal a sample inside a container. Many of these seals are designed so that a sample may be sealed inside a container and subsequently removed through the seal for use.

[0002] For example, a commonly used container seal includes a disc or cylindrically shaped seal made of an elastomeric material, such as a rubber or the like. To remove sample from the container without removing the seal from the container, the seal may be pierced with a syringe needle or the like and the contents of the container may be contacted with, and withdrawn into, the needle. The needle and contents therein may then be removed back through the seal to remove the needle with the container contents from the container.

[0003] However, current container seals suffer from a number of disadvantages. For example, once pierced by a syringe needle, the integrity of the seal is broken and the container contents are exposed to the atmosphere. This exposure may result in evaporation of the contents and/or oxidation of the contents due to exposure to atmospheric oxygen. Evaporation may change the concentration of the contents and oxidation can change the identity of the contents either of which may adversely effect the results of an experiment using the thus-exposed contents. In many cases, a container seal is designed to be pierced multiple times, which multiple piercing results in the acceleration of evaporation and/or oxidation of the container contents.

[0004] Another disadvantage of current container seals is that a syringe needle may abrade a seal's material during penetration of the seal producing loose particles of seal material. These abraded particles can clog the syringe needle and/or fall into the container contents. Over time, the container contents contacted with these abraded particles may leach components from, or otherwise become contaminated by, the abraded particles and may adversely effect experimental results.

[0005] In view of the continued need to seal containers, there continues to be an interest in the development of new device and methods for sealing container.

### SUMMARY OF THE INVENTION

[0006] Devices and methods are provided. Embodiments include devices for sealing an opening of a container, where such container sealing devices may include a flexible sealing element dimensioned to cover the opening of a container, a compliant channel in the flexible sealing element, and a compression member for maintaining the compliant channel in a sealed configuration, regardless of whether a fluid transfer member is present in the compliant channel. Also provided are containers

sealed by the subject sealing devices, and methods of using the same.

[0007] Embodiments also include methods of sealing a container, where methods may include introducing a fluid into a container and sealing the fluid within the container with a subject container sealing device. Also provided are methods of removing fluid from within a container sealed with a subject container sealing device, where embodiments include introducing a fluid transfer member through the channel of the container sealing device, urging the walls of the channel against the introduced fluid transfer member with the compression member of the sealing device, and removing a volume of fluid from the sealed container with the fluid transfer member.

[0008] The subject invention also provides kits for use in sealing a container, where embodiments of the subject kits may include a subject container sealing device and other components such as a container for sealing a fluid inside the container using the container sealing device.

### BRIEF DESCRIPTIONS OF THE DRAWINGS

[0009] Fig. 1 shows an embodiment of a container that may be sealed with a container sealing device of the subject invention.

[0010] Fig. 2 shows an embodiment of a flexible sealing element of the subject invention having a substantially constant outer diameter.

[0011] Fig. 3 shows an embodiment of a flexible sealing element of the subject invention having barrel shape.

[0012] Fig. 4 shows an embodiment of a flexible sealing element of the subject invention having a container-contacting lip.

[0013] Fig. 5A shows an embodiment of a container sealing device of the subject invention which includes the flexible sealing element of Fig. 4 and a compression member and Fig. 5B shows the device of Fig. 5A positioned in the opening of the container of Fig. 1 to seal the opening.

[0014] Fig. 6A shows the container sealing device of Fig. 5A having a fluid transfer member present in the channel of the device wherein the walls of the channel are sealingly contacted with the walls of the fluid transfer member providing a fluid tight seal therebetween; Fig. 6B shows the device of Fig. 6A positioned in the opening of the container of Fig. 1 to seal the opening and the fluid transfer member present within the channel and partially within the container; and Fig. 6C shows the fluid transfer member contacting the contents of the container.

### DEFINITIONS

[0015] The term "container" is used in its ordinary sense to refer to any object that can be used to hold one or more items, such as a volume of a fluid, e.g., liquid or gas.

[0016] The phrase "flexible sealing element" refers to any structure that can cover an opening of a container in

a manner such that the contents of the container are sealed from the outside environment of the container.

**[0017]** The term "channel" refers to any type of fluid conduit, such as a tubular passageway.

**[0018]** By "compression member" is meant a structure that can apply a compressive force to another structure.

**[0019]** By "fluid transfer member" is meant a device that serves to transfer a volume of fluid from a first to a second location.

**[0020]** By liquid seal is meant a "closure" that is substantially, if not completely, impermeable to liquids.

**[0021]** By "spring" is meant an elastic device, such as a coil of wire or an elastomeric band, that regains its original shape after being compressed or extended.

**[0022]** By "transparent" is referenced permitting light to pass therethrough without substantial attenuation or distortion.

**[0023]** By "without substantial attenuation" may include, for example, without a loss of more than about 40% of light, e.g., without a loss of more than about 30%, without a loss of more than about 20%, without a loss of more than about 10%, without a loss of more than about 5% or less.

**[0024]** "Opaque" is meant broadly to refer to the absorbance of rays of a particular wavelength/ Opaque with respect to a shield of the subject invention (or other element as indicated) is referenced that it may permit less than about 20 %, e.g., less than about 10 %, e.g., less than about 5 %, e.g., less than about 2 %, e.g., less than about 1% or less of ambient light from reaching enclosed microvolume space.

**[0025]** "Light returning" is meant broadly to refer to the change in direction which occurs when an electromagnetic wave strikes a surface and is thrown back. A light returning may reflect about 2% or more of light incident thereon, e.g., about 5 % or more, e.g., about 10% or more in certain embodiments.

**[0026]** "Optional" or "optionally" means that the subsequently described circumstance may or may not occur, so that the description includes instances where the circumstance occurs and instances where it does not.

**[0027]** A "plastic" is any synthetic organic polymer of high molecular weight (for example at least 1,000 grams/mole, or even at least 10,000 or 100,000 grams/mole).

**[0028]** "Flexible" with references that the flexible item can be bent 180 degrees around a roller of less than 1.25 cm in radius. The item can be so bent and straightened repeatedly in either direction at least 100 times without failure (for example, cracking) or plastic deformation. This bending must be within the elastic limits of the material. The foregoing test for flexibility is performed at a temperature of 20 °C.

**[0029]** "Rigid" with refers to an item which is not flexible, and is constructed such that a segment about 2.5 by 7.5 cm retains its shape and cannot be bent along any direction more than 60 degrees (and often not more than 40, 20, 10, or 5 degrees) without breaking.

**[0030]** "Fluid tight" is used herein to describe the spa-

tial relationship between two solid surfaces in physical contact, such that fluid (liquid and/or gas) is prevented from flowing into the interface between the surfaces.

**[0031]** The terms "Compliant" and "Deformable" are employed interchangeably and refers to a material that is able to be compressed e.g., to conform to a contacted surface.

**[0032]** "Chromatographic" processes generally include preferential separations of components, and include reverse-phase, hydrophobic interaction, ion exchange, molecular sieve chromatography, affinity chromatography and like methods.

**[0033]** The term "surface treatment" is used to refer to preparation or modification of the surface of a substrate such as a container surface. Accordingly, "surface treatment" as used herein includes: physical surface adsorptions; covalent bonding of selected moieties to functional groups on the surface of treated substrates (such as to amine, hydroxyl or carboxylic acid groups on condensation polymers); methods of coating surfaces, including dynamic deactivation of treated surfaces (such as by adding surfactants to media), polymer grafting to the surface of treated substrates (such as polystyrene or divinylbenzene) and thin-film deposition of materials such as diamond or sapphire to treated substrates.

**[0034]** One item is considered to be "larger" than a second item with respect a given measurement parameter if the first item is at last about 1% greater, such as at least about 5% greater, including at least about 10% greater than the second item with respect to the given measurement parameter.

**[0035]** "Remote location," means a location other than the location at which the array is present and hybridization occurs. For example, a remote location could be another location (e.g., office, lab, etc.) in the same city, another location in a different city, another location in a different state, another location in a different country, etc. As such, when one item is indicated as being "remote" from another, what is meant is that the two items are at least in different rooms or different buildings, and may be at least one mile, ten miles, or at least one hundred miles apart.

**[0036]** The term "assessing" and "evaluating" are used interchangeably to refer to any form of measurement, and includes determining if an element is present or not. The terms "determining," "measuring," "assessing," and "assaying" are used interchangeably and include both quantitative and qualitative determinations. Assessing may be relative or absolute. "Assessing the presence of" includes determining the amount of something present, as well as determining whether it is present or absent.

**[0037]** "Communicating" information references transmitting the data representing that information as signals (e.g., electrical, radio, optical, etc) over a suitable communication channel (e.g., a private or public network).

**[0038]** "Forwarding" an item refers to any means of getting that item from one location to the next, whether by physically transporting that item or otherwise (where

that is possible) and includes, at least in the case of data, physically transporting a medium carrying the data or communicating the data.

**[0039]** By "remote location" it is meant a location other than the location at which the optical measurement have been made. For example, a remote location could be another location (e.g. office, lab, etc.) in the same city, another location in a different city, another location in a different state, another location in a different country, etc. As such, when one item is indicated as being "remote" from another, what is meant is that the two items are at least in different buildings, and may be at least one mile, ten miles, or at least one hundred miles apart.

**[0040]** A "computer", "processor" or "processing unit" are used interchangeably and each references any hardware or hardware/software combination which can control components as required to execute recited steps. For example a computer, processor, or processor unit includes a general purpose digital microprocessor suitably programmed to perform all of the steps required of it, or any hardware or hardware/software combination which will perform those or equivalent steps. Programming may be accomplished, for example, from a computer readable medium carrying necessary program code (such as a portable storage medium) or by communication from a remote location (such as through a communication channel).

**[0041]** A "memory" or "memory unit" refers to any device which can store information for retrieval as signals by a processor, and may include magnetic or optical devices (such as a hard disk, floppy disk, CD, or DVD), or solid state memory devices (such as volatile or non-volatile RAM). A memory or memory unit may have more than one physical memory device of the same or different types (for example, a memory may have multiple memory devices such as multiple hard drives or multiple solid state memory devices or some combination of hard drives and solid state memory devices).

**[0042]** To "record" data, programming or other information on a computer readable medium refers to a process for storing information, using any such methods as known in the art. Any convenient data storage structure may be chosen, based on the means used to access the stored information. A variety of data processor programs and formats can be used for storage, e.g. word processing text file, database format, etc.

## DETAILED DESCRIPTION OF THE INVENTION

**[0043]** Devices and methods are provided. Embodiments include devices for sealing an opening of a container, where such container sealing devices may include a flexible sealing element dimensioned to cover the opening of a container, a compliant channel in the flexible sealing element, and a compression member for maintaining the compliant channel in a scaled configuration, regardless of whether a fluid transfer member is present in the compliant channel. Also provided are containers

sealed by the subject sealing devices, and methods of using the same.

**[0044]** Embodiments also include methods of sealing a container, where methods may include introducing a fluid into a container and sealing the fluid within the container with a subject container sealing device. Also provided are methods of transferring fluid from within a container sealed with a subject container sealing device, where embodiments include introducing a fluid transfer member through the channel of the container sealing device, urging the walls of the channel against the introduced fluid transfer member with the compression member of the sealing device, and removing a volume of fluid from the sealed container with the fluid transfer member.

**[0045]** The subject invention also provides kits for use in sealing a container, where embodiments of the subject kits may include a subject container sealing device and other components such as a container for sealing a fluid inside the container using the container sealing device.

**[0046]** Before the present invention is described in greater detail, it is to be understood that this invention is not limited to particular embodiments described, as such may, of course, vary. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments only, and is not intended to be limiting, since the scope of the present invention will be limited only by the appended claims.

**[0047]** Where a range of values is provided, it is understood that each intervening value, to the tenth of the unit of the lower limit unless the context clearly dictates otherwise, between the upper and lower limit of that range and any other stated or intervening value in that stated range is encompassed within the invention. The upper and lower limits of these smaller ranges may independently be included in the smaller ranges is also encompassed within the invention, subject to any specifically excluded limit in the stated range. Where the stated range includes one or both of the limits, ranges excluding either or both of those included limits are also included in the invention.

**[0048]** Unless defined otherwise, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. Although any methods and materials similar or equivalent to those described herein can also be used in the practice or testing of the present invention, the preferred methods and materials are now described.

**[0049]** All patents and publications mentioned herein are incorporated herein by reference to disclose and describe the methods and/or materials in connection with which the publications are cited. The citation of any patent or publication is for its disclosure prior to the filing date and should not be construed as an admission that the present invention is not entitled to antedate such publication by virtue of prior invention. Further, the dates of publication provided may be different from the actual publication dates which may need to be independently con-

firmed.

**[0050]** It must be noted that as used herein and in the appended claims, the singular forms "a", "an", and "the" include plural referents unless the context clearly dictates otherwise. It is further noted that the claims may be drafted to exclude any optional element. As such, this statement is intended to serve as antecedent basis for use of such exclusive terminology as "solely," "only" and the like in connection with the recitation of claim elements, or use of a "negative" limitation.

**[0051]** As will be apparent to those of skill in the art upon reading this disclosure, each of the individual embodiments described and illustrated herein has discrete components and features which may be readily separated from or combined with the features of any of the other several embodiments without departing from the scope or spirit of the present invention.

**[0052]** The figures shown herein are not necessarily drawn to scale, with some components and features being exaggerated for clarity.

## DEVICES

**[0053]** As summarized above, the subject invention provides container sealing devices having compliant channels for receiving fluid transfer members and which devices are configured to maintain a seal prior to introduction of a fluid transfer member into the compliant channel, while a fluid transfer member occupies the compliant channel of the device, and following removal of the fluid transfer member from the compliant channel of the device. In this regard, embodiments of the subject sealing devices may be characterized as fluid leak-free, self-sealing devices.

**[0054]** As will be described in greater detail below, the container sealing devices of the subject invention include a compression member positioned about at least a portion of the compliant channel so as to provide a compression force to the walls of the channel in a manner suitable to produce a closed channel, i.e., a channel that does not allow passage of a fluid (e.g., gas or liquid) there-through. When a fluid transfer member is present in the channel, the compression member urges the channel walls against the walls of the fluid transfer member in a fluid tight arrangement to prevent exposure of the container contents to the atmosphere outside of the container even when a fluid transfer member is present in the channel. In this regard, the subject invention minimizes evaporation of container contents and exposure to atmospheric factors. As such, the subject container sealing devices have a single sealing mechanism, as opposed to two or more sealing mechanisms as in the case of some conventional seals which can increase manufacturing costs and increasing the number of possible failure modes.

**[0055]** The subject container sealing devices may be employed to seal a wide variety of containers and are not intended to be limited to any particular type or size

of container as it will be apparent that the subject devices may be used to seal any container having a periphery that defines an opening, for example an opening into any chamber such as that of a vessel or conduit. In certain embodiments, the subject devices may be used to seal containers used in gas or liquid chromatography applications. For example, a container sealed with a subject device may include a gas or liquid chromatography sample, a gas or liquid chromatography control or standard or the like to be evaluated by gas or liquid chromatography, a gas or liquid solvent, etc. In certain embodiments, the sealing devices may be adapted to seal an opening of a fluid pathway, e.g., an opening to a chromatograph column.

**[0056]** As noted above, in certain embodiments a subject sealing device may be used in a gas chromatography application. In certain embodiments, a sealing device may be adapted to isolate the carrier gas of gas chromatography apparatus from the atmosphere and through which samples may be injected into the gas chromatography system by traversing the channel of the sealing device with a fluid transfer member such as a syringe needle or the like. That is, aspects include sealing devices configured for use for an injection port of a gas chromatograph or the like.

**[0057]** A cross-sectional view of an exemplary embodiment of a container 100 that may be used with the subject sealing devices is shown in Fig. 1 and includes opening 102 and interior space or cavity 105. In this particular embodiment, the neck of the container includes threads 104 for threadable engagement of an overcap cap (see, e.g., Fig. 6B) which may be positioned over a sealing device, e.g., bonded or otherwise affixed thereto and/or to the container.

**[0058]** Container 100 may be of any suitable size, where in certain applications cavity 105 of container 100 may have a volume that ranges from about 100  $\mu$ l to about 20 liters or more, e.g., from about 100  $\mu$ l to about 125 ml. The dimension of opening 102 will of course vary, where in certain embodiments opening 102 may have an area that ranges from about 0.75 cm<sup>2</sup> to about 80 cm<sup>2</sup> or more e.g., from about 0.75 cm<sup>2</sup> to about 5 cm<sup>2</sup>.

**[0059]** A container sealable with a subject sealing device may be made of any suitable material and in certain embodiments in which the container is used to contain light-sensitive contents, the container may be opaque or made of light returning material. A container may be made of, e.g., a plastic, glass, ceramic, metal, etc. In certain embodiments, a container may be made of polypropylene or USP Type 1 Borosilicate Glass, or the like. For example, for containing fluid that may stick to glass or chemically react with glass, a polypropylene container may be used.

**[0060]** The subject sealing devices include a flexible sealing element 4 having at least one deformable bore or channel 6 therein, as shown in Figs. 2, 3 and 4. Fig. 2 shows an exemplary embodiment of flexible sealing element 4 having compliant channel 6 therethrough. In

this particular embodiment, the outer diameter (OD) of the flexible sealing element is substantially constant along its entire length L such that the exterior of the flexible sealing element is substantially straight along its length dimension.

**[0061]** Compliant channel 6 extends from a first end 4a of the flexible sealing element or layer all the way through the flexible sealing element to a second end 4b so that a fluid transfer member may be inserted through the flexible sealing element via the channel to access the contents of a container sealed by device 4. First end 4a may include a tapered lead-in or depression (see, e.g., Figs. 4, 5A, 5B, 6A and 6B) for guiding a fluid transfer member into the channel. The lead-in may be formed in the shape of the flexible sealing element or may be produced when a compression member compresses the flexible sealing element as described in greater detail below. The lead-in assists in guiding a fluid transfer member to the same point of the sealing device each time so that the fluid transfer member enters the channel in precisely the same manner each time, further reducing the possibility of abrading the flexible sealing element even in those instances in which a sharp tipped fluid transfer member is used. In certain embodiments, first and second ends are annular ends. Compliant channel 6 may be defined by channel walls 7. The inner diameter of channel 6 is substantially constant along its entire length in certain embodiments, i.e., the channel walls are substantially straight.

**[0062]** Fig. 3 shows another exemplary embodiment in which the OD of flexible sealing element 4 varies and thus flexible sealing element 4 is barrel-shaped. Flexible sealing element 4 has a smaller outer diameter dimension near ends 4a and 4b relative to the area of the flexible sealing element between the ends, however the diameter of the compliant channel is substantially constant along its length. For example, embodiments include flexible sealing elements having variable or rather a plurality of different wall thicknesses WT, where the wall thickness is defined as the dimension from a channel wall to an exterior surface of the flexible sealing element, such that the thickness of the wall may vary along its length and may be thicker about the mid section of the flexible sealing element than nearer the ends of the flexible sealing element.

**[0063]** As shown in Fig. 4, certain embodiments may include a lip 8 at an end of the flexible sealing element. Lip 8 may be configured to contact a surface of a container that defines an opening to be sealed by a subject device. For example, lip 8 may extend over and engages the top edge 103 of a container in a fluid-tight manner. It will be appreciated that the fluid-tight attachment of the lip to a container may be made by any one of a variety of suitable methods, such as any suitable chemical and/or mechanical method, e.g., welding, bonding, adhesive, overcap, etc. While lip 8 is shown with the barrel shaped flexible sealing element embodiment of Fig. 3, the lip may be used with any shaped flexible sealing el-

ement.

**[0064]** In constructing the flexible sealing element/channel component, flexible sealing element 4 may be made of any suitable flexible material. For example, flexible sealing element 4 may be made of a resilient polymer such as natural and synthetic polymers, for example butadiene polymers and copolymers, neoprene, chloroprene and the like. For example, a flexible sealing element may be made from rubber (natural/butyl); PTFE/natural or butyl rubber; silicone/silicone rubber; PTFE/silicone, PTFE/Silicone/PTFE, VITON®.

**[0065]** The material of flexible sealing element 4 may be selected to be compatible with any chemicals and conditions to which the flexible sealing element may be exposed in the intended use, however this need not be case if, for example, the flexible sealing element is surrounded by a barrier (e.g., coating or the like) which prevents direct contact of any chemicals with the flexible sealing element. In certain embodiments, a septum may be made of a material that is capable of withstanding high temperatures, e.g., temperatures as great as about 150°C or more, e.g., about 200°C or more, e.g., about 300°C or more, e.g., about 400°C or more.

**[0066]** For example, in certain embodiments a flexible sealing element material of rubber (natural/butyl) may be chosen for compatibility with ACN, acetone, DMF, alcohols, diethylamine, DMSO and phenols; in certain embodiments a flexible sealing element of material of Silicone/Silicone rubber may be chosen for compatibility with alcohol, acetone, ether, DMF and DMSO; in certain embodiments a flexible sealing element of material of VITON may be chosen for compatibility with chlorinated solvents, benzene, toluene, alcohols, hexane, and heptane.

**[0067]** Materials that may be used are usually gas-impermeable, or hardly have permeability to water, which makes the container contents isolated from the outside air or any other reactive gas source, thereby providing a long shelf life.

**[0068]** Flexible sealing element may be fabricated from a "composite," i.e., a composition made up of different or unlike materials. The composite may be a block composite, e.g., an A-B-A block composite, an A-B-C block composite, or the like. Alternatively, the composite may be a heterogeneous combination of materials, i.e., in which the materials are distinct or are in separate phases, or a homogeneous combination of unlike materials. As used herein, the term "composite" is used to include a "laminated" composite. A "laminated" refers to a composite material formed from several different bonded layers of the same or different materials.

**[0069]** In certain embodiments, at least a portion of the flexible sealing element may be hydrophobic, where it may be inherently hydrophobic or may be made to be hydrophobic, e.g., by a hydrophobic agent, chemical manipulation, etc. By "hydrophobic" it is meant that at least a portion of a flexible sealing element is substantially if not completely unwettable and substantially if not com-

pletely liquid repellant for the sample contacted thereto, even if the sample is not an aqueous solution. For example, in the case of an oily-based sample, it should therefore correspondingly be a lipophobic surface. In certain embodiments, at least a portion of a flexible sealing element may be hydrophilic, where the material of the flexible sealing element may be inherently hydrophilic or be made hydrophilic, e.g., by a hydrophilic agent, chemical manipulation, etc. By "hydrophilic" it is meant that at least a portion of a flexible sealing element is easily wettable for the type of sample contacted thereto, even if the sample is not an aqueous solution. For example, in the case of an oily-based sample, it should therefore correspondingly be a lipophilic surface. In certain embodiments, a flexible sealing element may have one or more areas that are hydrophobic and one or more areas that are hydrophilic. For example, in certain embodiments flexible sealing element 4 may include a surface modification or treatment such as a surface coating which may render the flexible sealing element hydrophilic, hydrophobic, lipophilic, lipophobic, etc. The coating may cover the some or all of surface area of the exterior of the flexible sealing element and/or some or all of the surface area of the walls of compliant channel 6. A variety of surface coatings may be used and include, but are not limited to, polytetrafluoroethylene (e.g., Teflon®) coatings, and the like.

**[0070]** Flexible sealing element 4 is dimensioned to cover an opening of a container and thus the particular dimensions of the flexible sealing element will of course depend at least on the dimension of the opening it is designed to cover. For example, for a container having dimensions that fall within the ranges described above, in certain embodiments the flexible sealing element may have a length that ranges from about 5 mm to about 25 mm, e.g., from about 5 mm to about 10 mm and a width that may range from about 2 mm to about 15 mm, e.g., from about 5 mm to about 8 mm and a channel having an inner diameter that may range from about 0.5 mm to about 5 mm, e.g., from about 0.5 mm to about 1 mm. The wall thickness may range from about 0.75 mm to about 5 mm, where in certain embodiments a given flexible sealing element may have a constant wall thickness or the wall thickness may vary along the length dimension of a given flexible sealing element, e.g., the wall thickness may change, e.g., gradually change or otherwise.

**[0071]** The dimensions of the compliant channel will depend at least in part on the dimensions of the flexible sealing element and in certain embodiments on the dimensions or range of dimensions of a fluid transfer member the channel is designed to accommodate therein. In certain embodiments, the inner diameter of a compliant channel, when not compressed by a compression member, may range from about 0.5 mm to about 5 mm, e.g., from about 0.5 mm to about 1 mm. For example, in certain application a needle such as 26 gauge needle may be used to access contents of a container sealed by a subject device. In such embodiments, the inner diameter of a compliant channel may be dimensioned to receive such

a needle and may have an inner diameter that ranges from about 0.5 mm to about 1 mm in certain embodiments.

**[0072]** The container sealing device may be adapted to be inserted into openings of existing containers acting as a direct replacement sealing device of the original or currently installed equipment. Embodiments include sealing devices adapted for replacing seals currently integrated with an analytical system such as a septum of a GC inlet system, e.g., a septum or septum nut on a capillary inlet system. In this manner, existing equipment may be easily retrofitted to incorporate the sealing devices of the present invention.

**[0073]** The subject container sealing devices include a compression member positioned at least about the walls of the compliant channel, and in certain embodiments about the OD of the flexible sealing element, to maintain the compliant channel in a sealed configuration by urging the walls of the channel inward (e.g., towards each other), e.g., by applying a radial compression force to the channel walls. In this manner, when a fluid transfer member is positioned within the compliant channel, the compression member urges the walls of the channel against the surface of the fluid transfer member so that the channel walls conform to the surface of the fluid transfer member in a fluid tight arrangement, thereby protecting the contents of the container from the atmosphere by preventing contact of the container contents with the atmosphere.

**[0074]** Fig. 5A shows an exemplary embodiment of a subject sealing device 2 that includes flexible sealing element 4 having compliant channel 6 therethrough and Fig 5B shows the device of Fig. 5A as it may be positioned in an opening of a container 100 to seal the opening and the contents 200 therein. The device includes compression member 20 which, as shown, urges the walls of the channel together so as to close-off the channel in those instances when a fluid transfer member is not present within the channel and urges the walls against a fluid transfer member in those instances when a fluid transfer member is present within the channel. Accordingly, compression member 20 seals the container contents from the atmosphere when there is no fluid transfer member in the channel and also seals the container contents from the atmosphere when a fluid transfer member is in the channel by urging the walls of the channel against the walls of the fluid transfer member in a sealing arrangement.

**[0075]** When the channel is squeezed shut by the compression member, such squeezing may produce a tapered lead-in as described above assist in guiding a fluid transfer member into the channel. Due to the configuration of the compliant channel, a variety of fluid transfer members may be employed including tapered and blunt tipped fluid transfer members. For example, sharp or blunt tipped needles (e.g., stainless steel needles) may be used, tubing such as rubber tubing may be used, etc.

**[0076]** While compression member 20 seals the chan-

nel by applying a force to the channel walls, the device is configured to permit a fluid transfer member through the channel, past the closure in the channel, and into the container sealed by the device. However, even with the fluid transfer member in the channel, the compression member is configured to continue to provide an urging force to the walls of channel to urge them against the exterior surface of the fluid transfer member in a conforming and fluid tight relationship. In this manner, the container contents remain sealed from the environment, a shown in Fig. 6A which shows a fluid transfer member 50 present in channel 6 of device 2 and Fig. 6B shows the device of Fig. 6A as it may be positioned in an opening of a container 100 to seal the opening and the contents 200 therein. More specifically, as a fluid transfer member is inserted into the channel, the sealing device is configured to allow the fluid transfer member to force the compliant channel open causing the channel to expand slightly, but just enough to accommodate the member as the member slides past the closure made by the compression member. The compression member causes the channel walls to sealingly contact the member to provide a leak-free seal. As the member is withdrawn from the channel, the compression member causes the channel to close up. At all times, the container contents are sealed from the atmosphere.

**[0077]** The compression member may be in any suitable form, so long as it is able to apply enough force to the channel walls to produce a closed channel and to permit a fluid transfer member through the closure in the channel and urge the channel walls sealingly against the member to provide a fluid tight sealed configuration. For example, the compression member may be a band or ring (metal, plastic, rubber or the like), a spring, clamp, clip, etc. In certain embodiments, the compression member is a metal, rubber or plastic band or tube.

**[0078]** The compression member may apply force to the compliant channel in any number of ways. In certain embodiments, the dimensions of a compression member may at least in part provide the requisite compression to the compliant channel. For example, the inner diameter of the compression member may be slightly smaller than the area it is positioned (or may be adapted to be caused to be slightly smaller such as for example by tightening or otherwise reducing the inner diameter of a compression member from a first diameter to a second, smaller diameter), e.g., the outer diameter of the channel or the outer diameter of the flexible sealing element, such that once operatively positioned, the channel walls are urged closed due to the dimensional fit of the compression member. In addition or instead of a dimensional fit, a compression member may be crimped or otherwise tightened or cinched about the channel (or the exterior of the flexible sealing element) to apply a force to the channel walls to close the channel.

**[0079]** Aspects may include an overcap 125 positionable over a sealing device of a container (see for example Fig. 5B). An overcap may be maintained in a fixed posi-

tion by any suitable chemical and/or mechanical methods, e.g., welding, bonding, adhesive, threads, snap fit, press fit, friction fit, crimping, etc., to the sealing device and/or the container. For example, a sealing device may be bonded to an overcap. The overcap may be in a variety of forms, e.g., may be in the form of a screw cap, crimp cap, snap cap, or the like. An overcap may be a solid piece such that it may cover the entry to the compliant channel and thus may require removal (or penetration) prior to introducing a fluid transfer member into the channel or may include an opening at least about the opening to the channel so that it need not be removed prior to removal prior to introducing a fluid transfer member into the channel. An overcap may also serve to prevent the inadvertent movement such as inadvertent removal of the sealing device from the container.

**[0080]** A sealing device may be fabricated in any convenient manner. In one aspect, fabrications may be generally described as follows. A septum may be formed in a suitable shape from, e.g., an elastomer, that includes a preformed cylindrical channel. The sealing device may be formed using any suitable process, e.g., injection molding or the like. Methods for fabricating a septum and which may be adapted for use in fabricating a flexible sealing element are described, e.g., in US 6,648,853. Once a flexible sealing element is formed, a compression member may be applied at least about a portion of the flexible sealing element that includes at least a portion of the channel. The compression member is caused (dimensionally, crimped, etc.) to urge the walls of the channel closed. When the channel is urged closed by the compression member, a fluid transfer member lead-in (e.g., a tapered lead-in) may be provided.

**[0081]** Embodiments include sealing devices that may be re-used. In certain other embodiments, sealing devices may be single-use.

## METHODS

**[0082]** The subject invention also provides methods of sealing a container, including sealing a fluid inside a container, with a subject container sealing device. Embodiments also include removing at least a portion of the fluid from the sealed container without removing the sealing device from the container or otherwise compromising the seal.

**[0083]** Aspects include introducing fluid into a container, where any suitable container may be used, e.g., a container as described above, e.g., as shown in Fig. 1. A container may be in the form of a rigid, impermeable sample container such as a glass vial or the like having an open neck. The neck may open into a cavity which receives the sample. In certain embodiments, the container may be a container used in gas chromatography (GC), GC/mass spectrometry (MS), or liquid chromatography (LC) applications. For example, a container may be a housing attached to an injection port or inlet system of a gas chromatograph or the like. Aspects may include



a sealed container having a sample for gas or liquid chromatography analysis sealed therein.

**[0084]** Embodiments include introducing fluid into the container, where any suitable fluid may be introduced, including liquids and gases. As noted above, in certain embodiments the fluid may be a sample such as a liquid sample or the like for evaluation using gas or liquid chromatography. In certain embodiments, both a liquid and a gas, e.g., an inert gas, may be introduced into the same container and sealed therein. Fluids include, but are not limited to, solvents (e.g., for use in gas chromatography), test controls or standards, test samples such as biological samples or the like which may be solubilized in a suitable solvent, etc. For example, a container may be filled with a gas chromatography solvent or sample which may then be overlaid with a layer of inert gas to preserve the gas chromatography solvent or sample.

**[0085]** In certain other embodiments, fluid is introduced subsequent to sealing the container opening with a sealing device and thus may be introduced through the compliant channel of the sealing device. In such instances, a fluid transfer member may be inserted through the compliant channel and fluids may be either injected into or removed from the container through the channel. In certain other embodiments, fluid may be introduced into the container prior to sealing the container opening with a sealing device. In such instances, once the fluid of interest is introduced into the cavity of the container, the container may then be sealed with a sealing device. The fluid may be introduced into the container using any suitable means, which include manual and automated means such as manually actuated pipettors and automated auto samplers or pipettors associated with a robotic arm, all under the control of a processor. For example, an automated filling machine may fill a container with a predetermined amount of fluid.

**[0086]** Regardless of when a container is sealed (prior or subsequent to introducing fluid into the container), a subject sealing device is positioned about the opening of the container, the compliant channel of the sealing device being compressed to produce a sealed, but penetrable channel and thus a sealed container. A subject sealing device extends over the opening of the container to enclose the interior space of the container in a fluid-tight seal.

**[0087]** Once fluid is within the container and the container sealed with a subject sealing device, an overcap may be positioned over the sealing device and maintained in a fixed position by chemical and/or mechanical methods, e.g., welding, bonding, adhesive, threads, snap fit, pres fit, friction fit, crimping, etc., to the sealing device and/or the container. The overcap may be a solid piece such that it may cover the entry to the compliant channel and thus may require removal (or piercing) prior to introducing a fluid transfer member into the channel or may include an opening at least about the opening to the channel so that it need not be removed prior to removal prior to introducing a fluid transfer member into

the channel. An overcap may also serve to prevent the inadvertent removal of the sealing device from the container.

**[0088]** To withdraw some or the entire sample from the container without removing the sealing device from the opening of the container, a fluid transfer member is introduced through the sealing device by parting the compliant channel instead of coring the device (see for example Figs. 6A and 6B). As described above, when a fluid transfer member is present within the compliant channel, the container contents remained fluidly sealed from the atmosphere due to the sealing contact of the channel walls against the fluid transfer member surface.

**[0089]** For example, as shown in Fig. 6B, a fluid transfer member 50, which may be in the form of a hollow needle 50 or the like, may be positioned so as to enter the compliant channel, e.g., via a lead-in portion. An automated motion enabler may support the fluid transfer member 50 such that reciprocal longitudinal motion of the needle may be enabled, e.g., automatically by an automated system under the control of a suitably programmed processor. Fluid transfer member 50 has a fluid contacting end or needle tip 52, a longitudinal tubular passage 54, and a circular cylindrical wall defining the passage. End 52 may be a blunt tip or a pointed tip. The fluid transfer member may be a fixed needle syringe or a removable needle syringe. For example, a fluid transfer member may be about a 23 gauge fixed or removable needle syringe having a volume of about 5  $\mu$ l to about 10  $\mu$ l.

**[0090]** Fluid transfer member 50 may be aligned perpendicular to the sealing device so that a longitudinal motion of the needle towards the sealing device allows the needle tip to enter the compliant channel and part the channel a sufficient distance to allow passage of the needle through the channel and into the container. While the fluid transfer member is present within the channel, the channel walls (i.e., the material of the sealing device that forms the channel walls) surround tightly outside of the fluid transfer member wall for sealing, thereby producing a fluid tight seal about the fluid transfer member. Part of the length of the needle is inserted through the compliant channel to contact the contents of the container as shown in Fig. 6C. An amount of the contents of the container may then be withdrawn into the fluid transfer member, where the remainder of the contents may be removed at a later time in an analogous manner.

**[0091]** By a longitudinal reverse motion of the inserted fluid transfer member, the fluid transfer member may be removed. During removal of the fluid transfer member, the channel is urged closed by the compression force applied to the channel walls by the compression member so that the container contents remain sealed from the environment. As the fluid transfer member is removed from the channel, the outwardly displaced material of the channel walls closes the channel, eliminating fluid passage and maintaining a fluid-tight seal between the atmosphere and the interior of the container. In this man-

ner, the passage through the seal is self-sealed, re-forming a fluid-tight seal.

**[0092]** The fluid transfer member may be repeatedly re-inserted through the sealing device and removed from the sealing device again, where this may be repeated a number of times, e.g., 20 or more times, e.g., 5 or more times, e.g., 10 or more times, e.g., 20 or more times, e.g., 30 or more times, e.g., 40 or more times, e.g., 50 or more times, e.g., hundreds or even thousands of time of times, while maintaining a seal of the container contents from the atmosphere. Accordingly, the subject methods provide a leak-free manner of removing fluid from a sealed container, regardless of the number of fluid transfer member entries/removals. The container contents are isolated from the outside environmental factors so that the contents may have a long shelf life when in the container, e.g., during storage and are not otherwise contaminated. This may be particularly important when the container contents are internal standards or controls in which volumes of such may be repeatedly accessed and removed from the sealed container, e.g., in automated systems in which large numbers of samples are analyzed.

**[0093]** As noted above, certain containers may include a liquid such as a sample liquid for analysis which may or may not be dissolved in a suitable solvent or may just include a liquid solvent. A gas such as an inert gas (e.g., N, Ar, and the like) may be introduced into the container to blanket the contained liquid to help preserve the integrity of the liquid prior to use. A problem encountered in certain conventional seals is that unintentional leakage of the gas may occur when a volume of the liquid is removed from the container using a fluid transfer member due to leakage of the seal. If a fluid transfer member is introduced into and removed from a seal multiple times, the amount of the gas unintentionally leaked from the container due to the compromised seal may be significant enough to compromise the integrity of the liquid. The subject sealing devices, which provide a seal even when a fluid transfer member is present in the channel of the device, enables a volume of sample to be removed from the container without unintentional leakage of gas from the container. As such, the gas may be retained in the container even after repeated introductions and removals of sample from the container

**[0094]** As noted above, in certain embodiments a subject sealing device may be used in gas and liquid chromatography applications, e.g., to retain a sample in a container for analysis (e.g., dissolved in a solvent), to retain a control or standard in a container, to retain a solvent in a container and the like, in a fluid tight sealed manner so that the sample, control, standard, etc., is not exposed to the atmosphere. Aspects include sealing devices adapted to fit in the injection port of a chromatographic device (not shown) such that samples may be injected by inserting a fluid transfer member such as a syringe needle or the like through the channel of the sealing device. For example, a sealing device may be associated with a sample injector for an analytical instrument

such as a gas chromatograph or the like. Embodiments include replacing a septum of a GC inlet system such as a septum or septum nut on a capillary inlet system.

**[0095]** In such injection port applications, a subject sealing device seals the entrance port of an injector block. A quantity of a sample to be analyzed is introduced through the sealing device using a fluids transfer element and into a flowing stream of gas by injecting it from a fluid transfer member, e.g., a hypodermic syringe or the like, in a manner analogous to that described above for introducing a fluid transfer member through a channel of a sealing device. As noted above, the sample is introduced by introducing the fluid transfer member into the channel of the sealing device, the sealing device sealing around the fluid transfer member when present therein and sealing itself when the fluid transfer member is withdrawn. That is, when the fluid transfer member is present in the channel of the sealing device, the walls of the channel are urged against the fluid transfer member in a fluid-tight sealing relationship to provide for a sealed configuration. Once the sample is introduced into the gas, the gas carrying the injected sample then passes through the chromatographic separation column and the effluent gas is passed to a detector all in a manner well known in the art.

#### Kits

**[0096]** Finally, kits are also provided. The subject kits may include one or more container sealing devices. Kits may also include one or more containers sealable with the sealing devices. The containers may be provided in the kit sealed with a sealing device, e.g., may be provided having fluid sealed therein. In instances in which a fluid is sealed in the container, the fluid may be any suitable fluid as described above, e.g., a fluid for use in a gas or liquid chromatography protocol. Kits may also include a fluid transfer member, e.g., a syringe or the like.

**[0097]** The subject kits may also include written instructions for using the sealing devices to seal a container and to introduce and/or remove fluid from a sealed container without removing the sealing device. Instructions of a kit may be printed on a substrate, such as paper or plastic, etc. As such, the instructions may be present in the kits as a package insert, in the labeling of the container of the kit or components thereof (i.e., associated with the packaging or sub-packaging) etc. In other embodiments, the instructions are present as an electronic storage data file present on a suitable computer readable storage medium, e.g., CD-ROM, diskette, etc. In yet other embodiments, the actual instructions are not present in the kit, but means for obtaining the instructions from a remote source, e.g. via the Internet, are provided. An example of this embodiment is a kit that includes a web address where the instructions can be viewed and/or from which the instructions can be downloaded. As with the instructions, this means for obtaining the instructions is recorded on a suitable substrate.

**[0098]** In many embodiments of the subject kits, the components of the kit are packaged in a kit containment element to make a single, easily handled unit, where the kit containment element, e.g., box or analogous structure, may or may not be an airtight container, e.g., to further preserve the one or more chemical arrays and reagents, if present, until use.

**[0099]** While the present invention has been described with reference to the specific embodiments thereof, it should be understood by those skilled in the art that various changes may be made and equivalents may be substituted without departing from the true spirit and scope of the invention. In addition, many modifications may be made to adapt a particular situation, material, composition of matter, process, process step or steps, to the objective, spirit and scope of the present invention. All such modifications are intended to be within the scope of the claims appended hereto.

## Claims

### 1. A container sealing device comprising:

- (a) a flexible sealing element dimensioned to cover an opening of a container and having a compliant channel passing therethrough; and
- (b) a compression member that applies a compressive force to said compliant channel such that said compliant channel remains in a sealed configuration, regardless of whether a fluid transfer member is present in said compliant channel.

2. The device of Claim 1, wherein said compliant channel has a first end having a first outer diameter and a second end having a second outer diameter, wherein said first outer diameter is larger than said second outer diameter.

3. The device of Claim 1, wherein said flexible sealing element is a one-piece structure.

4. The device of Claim 4, wherein said first and second ends of said compliant channel are annular.

5. The device of Claim 1, wherein said flexible sealing element comprises a hydrophobic coating.

6. A sealed container comprising a container sealing device according to any of Claims 1 to 5.

7. The sealed container of Claim 6, further comprising a fluid scaled inside said container.

8. The sealed container of Claim 7, wherein said container further includes a gas.

9. A method of sealing a container, said method comprising: positioning a container sealing device over an opening of said container, wherein said sealing device is a device according to any of Claims 1 to 5; to seal said container.

10. The method according to Claim 9, wherein said method further comprises introducing a fluid into said container.

11. The method according to Claim 10, wherein said fluid is introduced into said container prior to sealing said container.

12. A method comprising:

- (a) positioning a first end of a fluid transfer member into a fluid present in a sealed container according to Claims 6 to 8, wherein said positioning comprises introducing said fluid transfer member into said container through said compliant channel; and
- (b) removing a volume of fluid from a sealed container through said positioned fluid transfer member.

13. A kit comprising:

- (a) a container; and
- (b) a container sealing device according to any of Claims 1 to 5, wherein said sealing device is configured to seal an opening of said container.

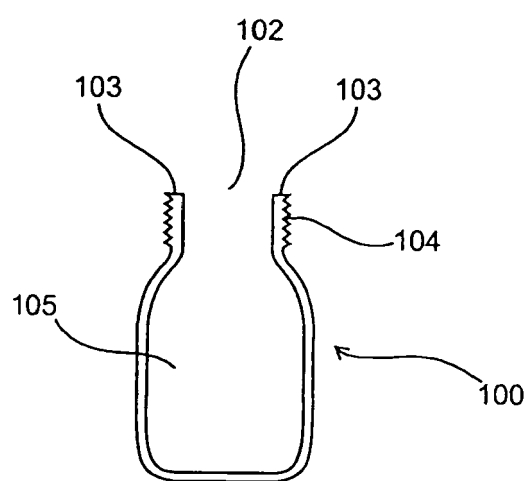


FIG. 1

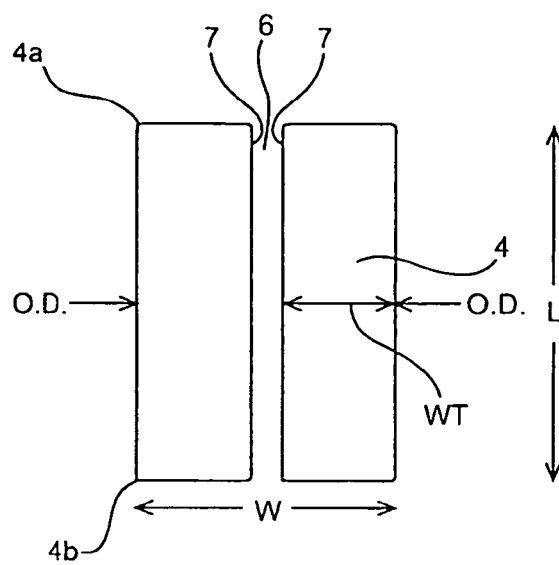


FIG. 2

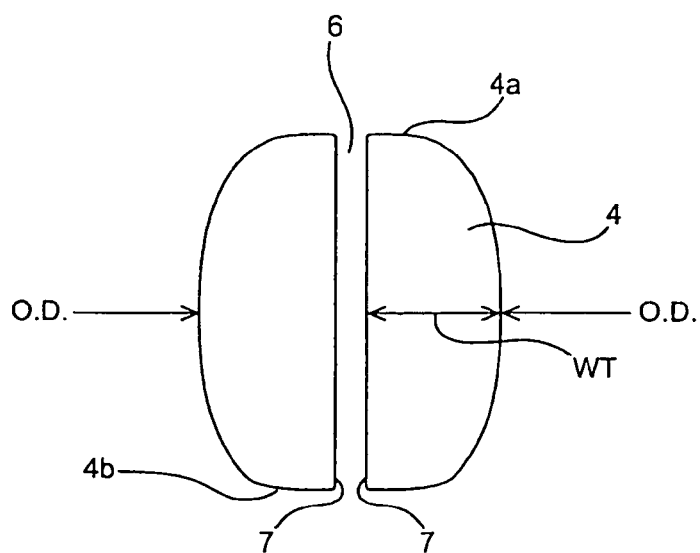


FIG. 3

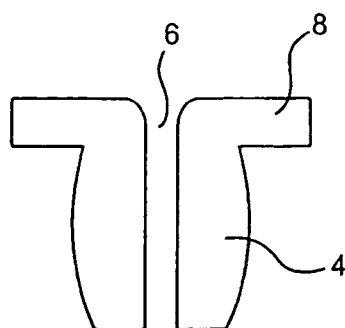


FIG. 4

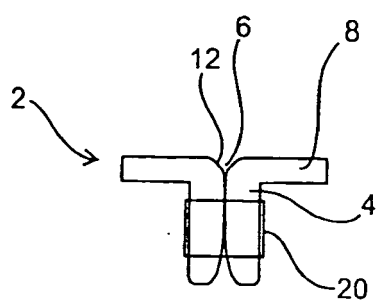


FIG. 5A

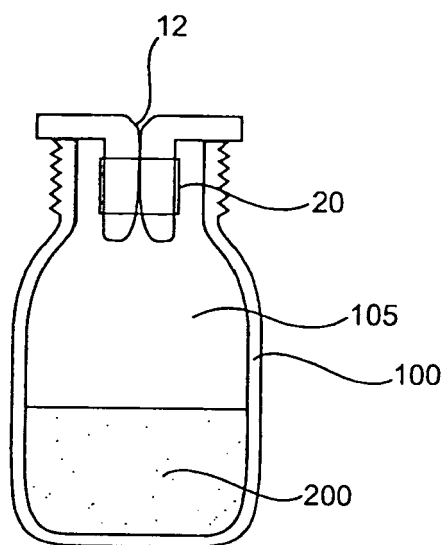


FIG. 5B

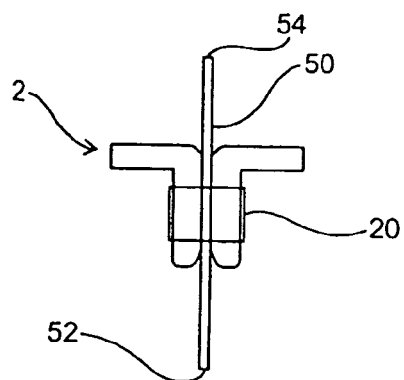


FIG. 6A

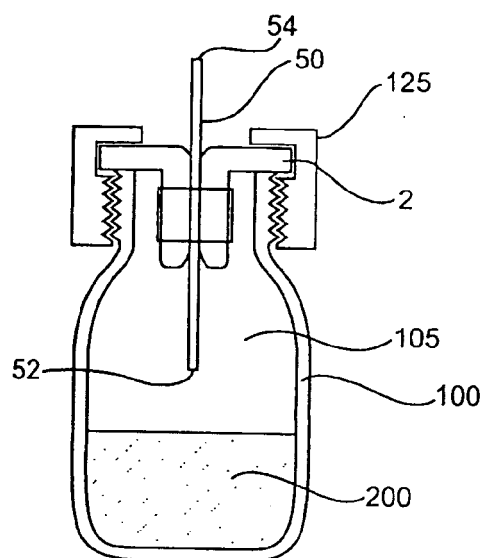


FIG. 6B

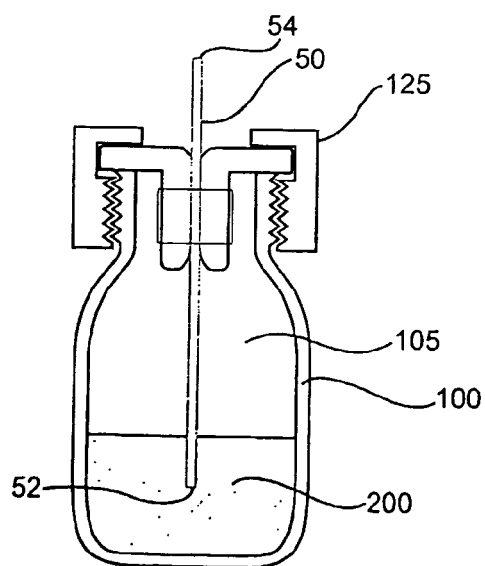


FIG. 6C



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Application Number  
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| DOCUMENTS CONSIDERED TO BE RELEVANT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                                                                              |                                                           |                                              |
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| Place of search<br><b>Munich</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                                                                                                                                                              | Date of completion of the search<br><b>26 August 2005</b> | Examiner<br><b>Marti, P</b>                  |
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26-08-2005

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