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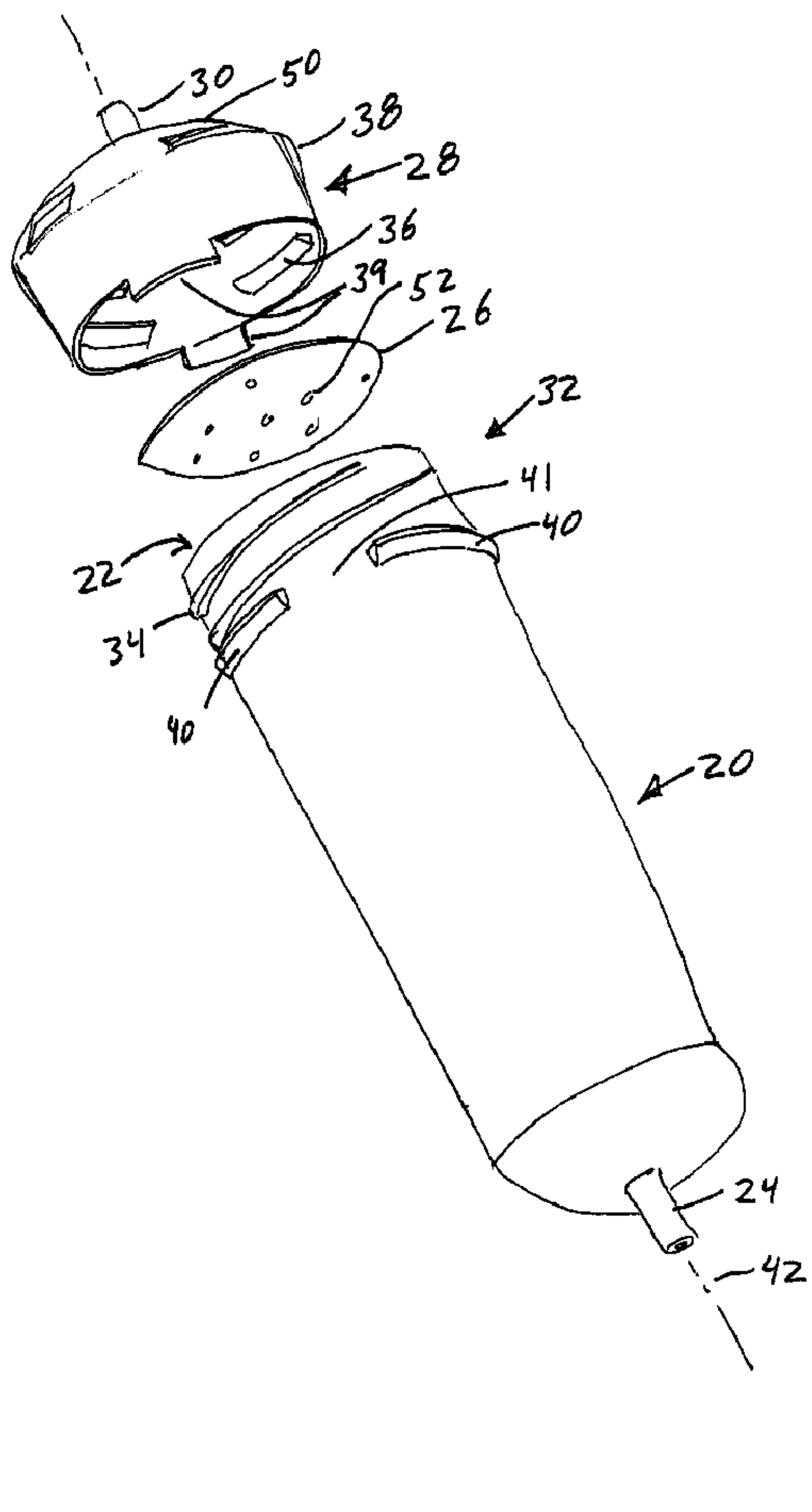


(57) Abrégé/Abstract:
A low pressure liquid chromatographic cartridge is provided having a tubular polymer container adapted to receive a chromatographic packing material. The container has an outlet port located at a downstream end of the container and container

(57) Abrégé(suite)/Abstract(continued):
threads formed on an upstream end of the container. A polymer cap having cap threads located on the cap threadingly engage the container threads. An inlet port is located on an upstream end of the cap. A flange depends from the cap and mates with the lip of the container to form a fluid tight seal between the polymer cap and container suitable for use in low pressure liquid chromatography. A locking tab on a skirt of the cap engages a recess on the container when the seal engages the cap and container to lock the cap in position relative to the container. Alternatively, continuous screw threads on the cap and container may hold the parts together.

ABSTRACT OF THE INVENTION

A low pressure liquid chromatographic cartridge is provided having a tubular polymer container adapted to receive a chromatographic packing material. The container has an outlet port located at a downstream end of the container and container threads formed on an upstream end of the container. A polymer cap having cap threads located on the cap threadingly engage the container threads. An inlet port is located on an upstream end of the cap. A flange depends from the cap and mates with the lip of the container to form a fluid tight seal between the polymer cap and container suitable for use in low pressure liquid chromatography. A locking tab on a skirt of the cap engages a recess on the container when the seal engages the cap and container to lock the cap in position relative to the container. Alternatively, continuous screw threads on the cap and container may hold the parts together.

FLASH CHROMATOGRAPHY CARTRIDGE

BACKGROUND OF THE INVENTION

This invention relates to method and apparatus involving cartridges for use in flash chromatography and low pressure liquid chromatography equipment.

Chromatographic analysis passes fluids through columns containing specially treated sorbent which allows the chemicals in the fluid to be eluted at different times and thus form separated peaks on a chromatogram. In order to prepare or clean up the fluid being analyzed the fluid is often passed through a sorbent under pressure. Further, for low pressure liquid chromatography (LPLC) or flash chromatography the fluid may be passed through a sorbent at a pressure of 20-100 psi. This operating pressure is sufficiently high that these cartridges, which have relative large diameter bodies leak at the seams. Threaded connections are thus not used to form the body when the body is made of polymers. Thus, these cartridges are traditionally made of plastic and have sonically welded ends. But even that welded construction will leak if there are defects in the welds. That welded construction and the accompanying manufacturing and material costs cause in undesirably high costs, especially as the cartridges must be either discarded, or must under go extensive and thorough cleaning after a single use, or at most after a few uses with similar fluids. There is thus a need for a low cost, disposable cartridge.

Further, the welded construction requires the chromatographic packing material be placed in the cartridge before it is welded, or it requires careful packing of the column under pressure, both of which limit the usefulness of the cartridge and increase its cost.

Recently one company has introduced a disposable cartridge made of molded polypropylene having an end fitting that uses openings in a number of cantilever members to engage detent members which fit into the openings to create an interference fit to snap-lock the end fitting onto the cartridge. This is described in U.S. Patent 6,565,745. But this interference fit is created at the factory and again creates a cartridge that does not allow the user to easily vary the contents of the cartridge. There is thus a need for a cartridge that allows a user to easily vary the contents.

Secondary cartridges are sometimes tied into the system for use, but these secondary cartridges are limited in size to 70ml (or between 20-25g of material). These secondary cartridges lie with tubing 1 to 2 feet away from the sample. The use of secondary cartridges increases the amount of run time and expensive solvent, due to elution of the sample. It causes dilution of sample because the cartridge is 1 to 2 feet away from the sample. The tubing and secondary cartridge also allows the addition of air which may or may not affect the chemical composition or performance. Also secondary cartridges have a capacity limit of 20-25g which does need meet the needs of all users since at times up to 60g is needed to be loaded. This forces an end user to separate the chemicals into several separate cartridges. The secondary cartridge is also an additional expense, and requires additional time for loading.

There is thus a need for a cartridge that can be sealed to function under LPLC pressures but which allows the user to access the inside of the cartridge before it is sealed.

Moreover, welded cartridges are limited by being pressure rated to only about 45 psi, due to leaking and instability at higher pressures. This pressure limits the end user, because high pressures are desirable for separating chemical compositions that are thick and viscous. Without these higher pressures these thick, viscous chemicals can not be distinguished. There is thus also a need for a larger capacity cartridge that can be used at higher pressures, especially for viscous fluids.

In LPLC the fluid sample is sometimes prepared by passing it through one or more cartridges of different material, each of which has a different sorbent to clean the fluid of particular undesirable materials or chemicals. Because the fluid sample can vary, a wide variety of cartridges with different sorbents sealed in the cartridges must be maintained. Further, the removal and reconnection of these various cartridges is cumbersome and time consuming, and the cost of each cartridge is expensive. There is thus a need for a way to reduce the complexity and cost of using different sorbents.

Sometimes a Y fitting is used to inject one or more fluids into the LPLC cartridge. The connection and use of these Y fittings is cumbersome. Further, the fitting must be either discarded or cleaned after each use. There is thus a need for a better and less expensive way to introduce fluid or materials into the cartridge.

SUMMARY

A low pressure liquid chromatographic cartridge is provided having a tubular polymer container adapted to receive a chromatographic packing material. The container has an outlet port located at a downstream end of the container and configured for connecting to chromatographic equipment during use of the cartridge. Container threads are formed on an upstream end of the container. A polymer cap has cap threads located on the cap to threadingly engage the container threads. The cap also has an inlet port located on an upstream end of the container. The port is configured for connecting to chromatographic equipment during use of the cartridge. A resilient fluid tight seal is interposed between the cap and container suitable for use in low pressure liquid chromatography. A locking tab is provided on a skirt of the cap and is located and configured to engage a recess on the container when the seal engages the cap and container. The locking tab locks the cap in position relative to the container.

In further variations the seal comprises a resilient ring extending from a top of the cap with the seal being located and sized to engage a lip of the container. Preferably a fluid dispenser is interposed between the container and the cap. The dispenser has a plurality of fluid outlets located across a substantial portion of a cross-section of the container to dispense fluid from the inlet of the cap over the cross-section. The fluid dispenser preferably takes the form of a dish having a plurality of holes extending through the dish, so as to place the inlet and the outlet in fluid communication. Moreover, the dish preferably, but optionally has a rim placed between the cap and the container. In further embodiments the locking tab extends parallel to a longitudinal axis of the container and extends from a distal end of a skirt of the cap. Further, the inlet can take the form of a tube threadingly engaging one of the cap or container, with the tube having a threaded exterior distal end located on an exterior of the engaged one of the cap or container. Advantageously the seal and lip abut at an inclined angle with the seal extending inward toward a longitudinal axis of the container and cap. Moreover, the seal preferably joins the top of the cap at a corner which encircles a longitudinal axis of the container. Still further, the fluid dispenser has a periphery located in that corner. Preferably chromatographic packing material is placed in the cartridge by the user before the cap is locked onto the container. Preferably, but optionally, the material to be analyzed is also placed in the

cartridge by the user before the cap is locked onto the container. This allows the user to custom select and place any of a plurality of different chromatographic packing materials in the container.

In a further embodiment there is provided a low pressure liquid chromatography cartridge having a tubular container adapted to receive a chromatographic packing material. The container has an outlet port located at a downstream end of the container and configured for use with chromatographic equipment during use of the cartridge. The container also has container threads formed on an upstream end of the container. A cap is provided with an inlet port located on an upstream end of the cap, with port being configured for use with chromatographic equipment during use of the cartridge. The cap also has cap threads located on the cap to threadingly engage the container threads. Locking means on the container and cap prevent manual removal of the cap. Resilient sealing means are provided for sealing the cap to the container when a user places the cap on the container and engages the locking means.

In still further variations, the cartridge has means for distributing fluid from the inlet port over a cross-section area of the container during use of the cartridge. Moreover, chromatographic packing material and materials to be analyzed can be placed in the container by the user of the cartridge before the locking means are locked.

There is also provided a method for a user to perform low pressure liquid chromatography. The method includes placing at least one chromatographic packing material in a tubular container which has an outlet port located at a downstream end of the container. The outlet is again configured for use with chromatographic equipment. Container threads are formed on an upstream end of the container. The method includes threadingly engaging threads on a cap with the container threads. The cap is also provided with an inlet port on an upstream end of the cap. The method further includes sealing the cap to the container by tightening the threads and engaging a seal between the cap and the container. The seal provides a fluid tight seal below about 100 psi suitable for LPLC use.

In further variations the method includes locking the cap to the container. A still further variation includes connecting the inlet to a source of fluid for chromatographic analysis; and distributing the fluid from the inlet over a cross-section area of the container. Moreover, distributing step preferably, but optionally includes collecting the fluid in a fluid dispenser having a wall with a plurality of holes spread across the cross-section and passing the fluid through those holes. Inclining the surface with the holes toward a central longitudinal axis of the fluid dispenser which also passes through the fluid dispenser is also a preferred variation. In a still further variation the distributing step is performed by a fluid dispenser with a periphery that is interposed between the cap and the container.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features and advantages of the invention will be better understood by reference to the following drawings in which like numbers refer to like parts throughout, and in which:

Figure 1 is a perspective view of a first embodiment of a cartridge with a screw cap;

Figure 2 is a partial sectional view of the juncture of the cap and cartridge of Figure 1;

Figure 3 is a schematic view of the cartridge of Figure 1 connected to chromatographic equipment;

Figure 4a-b are top and side views, respectively, of the fluid dispenser shown in Figure 1;

Figure 5 is a partial sectional view of a further embodiment of inlet and outlet fittings for use with the cartridge of Figure 1;

Figure 6 is a plan view showing a further embodiment of a container and locking mechanism;

Figure 7 is a partial sectional view of a further embodiment of the connection of the cap and the container of Figure 1;

Figure 8 is a further embodiment of a fluid dispenser;

Figure 9 is a section of the fluid dispenser of Figure 8 taken along section 9-9 of Figure 8;

Figure 10 is a front perspective view of a further embodiment of a cap;

Figure 11 is a rear perspective view of the cap of Figure 10;

Figure 12 is a top plan view of the cap of Figure 10;

Figure 13 is a sectional view taken along section 13-13 of Figure 12;

Figure 14 is a cross-sectional view of a further embodiment of a container for use with the cap of Figure 10;

Figure 15 is a partial sectional view taken along circular section 15-15 of Figure 14;

Figure 16 is a sectional view of a further embodiment of a cap taken along section 16-16 of Figure 19;

Figure 17 is a sectional view of the cap of Figure 16, taken along section 17-17 of Figure 19;

Figure 18 is a circular sectional view taken along section 18-18 of Figure 16; and

Figure 19 is a top plan view of the further embodiment of the cap of Figure 16.

DETAILED DESCRIPTION

Referring to Figures 1-2 and 7, a cartridge is provided comprising tubular container 20 suitable for flash chromatography. The container has a cap 28 with inlet and outlet ports 24, and 30, respectively. The container 20 has an open end 22 at and upstream or proximal end, and an outlet port 24 at a downstream or distal end. A fluid dispenser 26 is placed in or upstream of the open end 22 and a cap 28 is fastened over the open end 22 and fluid dispenser. An inlet port 30 is provided on the cap 28. A locking mechanism 32 is placed on one or both of the container 20 and cap 28 to hold the cap to the container. A seal 48 between the cap 28 and container 20 is held in fluid tight compression by mating threads 32, 34 and the locking mechanism 32.

In use, the inlet 30 is placed in fluid communication with a source of fluid to be processed in a low pressure liquid chromatography (LPLC) or flash chromatography process. Processing or filtering media is placed in the container 20. The sample fluid to be tested is passed through the media in the container, and the resulting fluid is removed from the outlet port 24 for further processing or other treatment or analysis. Preferably, but optionally, the outlet port 24 is placed in fluid communication with the LPLC equipment or other chromatographic equipment for the processing or treatment. Advantageously the downstream or distal end of the container 20 is slightly curved or domed or inclined so the fluid being processed is funneled toward the outlet 24.

In more detail, the locking mechanism 32 can advantageously, but optionally take the form of mating threads on the container 20 and the cap 28. Figure 1 shows external threads 34 on the container mating with internal threads 36 on a skirt 38 of the cap 28. But the container threads 24 could be internal threads and the cap threads 36 could be external threads. The threads can 34, 36 be single lead, or multiple lead. The threads 34, 36 can be continuous or segmented.

Preferably, but optionally, a lip or flange 40 extends outward around the outer circumference of the container 20 adjacent the trailing end of the threads. Preferably, one or more gaps or spaces or recesses 41 are formed in the flange 40. As used herein, the leading end of the threads refers to the ends that first engage the mating threads, and the trailing end refers to the last to engage end of the threads. The outward direction means away from the longitudinal axis 42 of the container 20.

Referring to Figure 2, the polymer cap 28 is sealed to the polymer container 20 sufficiently to allow flash chromatography up to about 100 psi. A lip 44 is formed on the distal edge of the skirt 38 of the cap 28 so the lip 38 abuts the flange 40 on the container to limit the tightening of the cap on the container 20, and to help seal the cap to the container. A locking tab 39 extends from the skirt 28 along the direction of axis 42. The locking tab 39 is sized and shaped to fit into one of the recesses 41 on the flange 40. Thus, when the cap 28 is threaded onto the container 20 by threads 34, 36, the tabs 39 advance axially along axis 42 and fit into the recesses 41 to lock the cap from further rotation, and to lock the cap from unscrewing and the accompanying leaking. The tabs could be located on the container and the recesses on the cap.

Advantageously the locking tabs 39 are configured so the shape matches that of the flange 40, making it difficult to manually grab the tabs 39 and manipulate them to unscrew the cap 28. Advantageously, but optionally, the distal or downstream edge of the locking tab 39 tapers toward the axis 42 and that helps remove defined edges of sufficient size that the edge can be manually grabbed, and that helps avoid unlocking the cap. The locking tabs 39 thus provide means for preventing manual removal of the cap.

A shaped lip 46 is also preferably, but optionally placed around the opening 22 on the proximal or upstream end of the container 20. The shaped lip 46 is shown as inclined outward at an angle of about 30° from a line parallel to axis 42. The container lip 46 abuts a sealing surface 48 on the cap 28 to provide a fluid tight seal.

Different angles and lip shapes could be used, especially if different types of seals are used.

The cap sealing surface 48 is shown as comprising an annular seal depending from the inside of the cap 28. The sealing surface 48 is shown as connected to a top wall 50 adjacent the juncture of the top wall 50 with the side walls or skirt 38 of the cap 28. The top wall 50 is preferably, but optionally slightly domed or slightly curved outward. The sealing surface 46 is advantageously a thin walled ring, and preferably, but optionally has a slight conical shape narrowing toward the downstream end of the cap 28 and inward toward the axis 42. The cap sealing surface 48 thus preferably has a larger diameter at the upstream or proximal end where it fastens to the cap 28, and has a smaller diameter, open end located downstream and inward of the upper end to form a cone with the smaller end facing downstream.

Preferably, but optionally, the cap sealing surface 48 is integrally molded with the cap 28, although a two part assembly is also believed suitable. Referring to Figures 1-2, a series of rectangular openings appear near the juncture of the skirt 38 and the top of the cap 28 and these openings allow mold slides to pass through the cap to integrally mold the seal 48 with the cap. Referring to Figure 2, at the periphery where the cap sealing surface 48 extends downstream and inward toward axis 42, a corner is formed and the outer edge of the fluid dispenser 26 is placed in this corner.

As the cap 28 is threaded onto the container 20 using threads 34, 36, the downstream side of the cap sealing surface 48 abuts the upstream side of the cap lip 44 to form a fluid tight seal. The cap lip 44 on the distal or downstream edge of the cap 28 abuts the flange 40 on the container to prevent over-tightening and preferably, but optionally also form a redundant seal. As the cap 28 is threadingly tightened on the container 28 the conical cap sealing surface 48 is resiliently urged toward the container lip 46, squeezing and further sealing the periphery of the fluid dispenser 26 between the seal 48 and the cap 28. The fluid dispenser is thus held between the container and the cap, and distributes fluid along axis 42 across at least a substantial portion of a cross section of the container.

Referring to Figures 1, 2 and 4, the fluid dispenser 26 has a containing volume within which the fluid being processed collects and spreads over the cross-section of the container 20 in order to more evenly distribute the fluid over the material in the container. A plurality of holes 52 in the downstream surface of the fluid dispersing device allow the fluid to exit the fluid dispenser26. Various shaped fluid dispensers 26 could be used, including a container with a flat bottom, or inclined bottoms. As used herein, inclined surfaces include curved surfaces. As the container 20 is preferably, but optionally cylindrical in shape, these shapes result in a cylinder with a flat bottom, or a shallow conical surface or a downwardly curved surface. A flat surface on the dispensing device risks some fluid collecting in the device, and is thus not preferred. Various shaped and sized holes and hole patterns could also be used, with the holes 52 being preferably arranged to distribute the fluid being processed evenly over the cross-sectional area of the container 20. If a curved fluid dispenser 26 is used then the holes 52 may advantageously be larger in diameter as the holes get further from the longitudinal axis.

Still referring to Figures 1-2 and 4 the fluid dispenser 26 comprises a circular, domed dish having a plurality of holes 52 extending through the dish. The dish shaped fluid dispenser 26 is preferably, but optionally curved toward the downstream direction so that fluid entering the cap 28 through the inlet port 30 collects in the dish and passes through the holes 52. One hole is preferably located at the lowermost or most downstream portion of the surface to avoid fluid collecting in the fluid dispenser 26. Preferably the lowest opening 52 is on the longitudinal axis 42. A lip or rim of the dispenser is held between the container 20 and the cap 28, and in the preferred embodiment is held by the lip of the container resiliently urging the seal 48 against the rim of the dispensing device 26 against the top 50 of the cap.

The fluid entering the cap 28 through inlet port 30 enters at pressures of about 20-100 psi, and preferably about 50 psi, and at a flow rate of about 10-100 ml/min, although the pressure and flow rate can vary. The pressure and flow rate of the fluid entering the cap 28 and collecting on the fluid dispenser 26 is sufficient that the fluid spreads across the upstream surface of the dish shaped fluid dispenser 26 and squirts through the holes 52 like a showerhead to more evenly distribute the fluid over the cross-section of the container.

Referring to Figures 1-3, the container 20 is at lest partially filled with a chromatographic packing material 60 selected to suit the fluids being analyzed and the operating pressures and conditions. This is advantageously done by the user just before the cap 28 is locked onto the container 20. Various silica based sorbents are commonly used, and various sorbents 60a, 60b (Fig. 2) or other chromatographically useful materials can be layered by the user to achieve different effects on the fluid being processed. The level of the chromatographic packing material 60 can be varied by the user to leave a predetermined volume inside the container 20, with fluids or other materials being added to fill that predetermined volume.

The dish shaped dispensing device 26 is preferably thin, with a thickness of about 1/16 inch (16 mm) is believed suitable when the dish is made of polypropylene. The thickness and material will vary with the operating pressures and fluids being used. A radius of curvature of about 1-2 inches for the dish shaped dispensing device is believed suitable, and 1.5 inch curvature is used in one embodiment, but other curvatures could be used. The holes 52 are preferably, but optionally all the same diameter and are equally spaced. A diameter of about .03 to .04 inches (about 7-10 mm) for the holes 52 is believed suitable. The spacing and size of the holes 52 can vary to suit the fluids and pressures being used, and are preferably varied to ensure uniform flow through the dispensing device 26 across the entire cross-section of the container. The dispensing device 26 can be made of materials suitable for the processing of the desired fluid. The fluid dispenser 26 is preferably made of a polymer, such as polyethylene or polypropylene, and preferably of high density polypropylene. Other polymers can be used, although are preferably used that are low cost and suitable for injection molding to form disposable containers and caps. But metal dispensing devices are also believed suitable, such as stainless steel.

Referring to Figures 1 and 3, in use a desired amount of filtering media or chromatographic packing material 60, such as a silica sorbent, is placed in the downstream end of the container 20 by the user. Removing a partially secured, and unlocked screw cap 28 allows easy access to place the chromatographic packing material 60 in the container, to adjust the amount of material in the container, to add a different material or sorbent to the container or to adjust the amount of free volume in the container to receive the sample fluid or material or sorbent. A frit 62 can optionally be placed on the upstream and/or downstream end of the chromatographic packing material 60 as desired. The cap 28 and fluid dispenser 26 are then fastened to the container 20 to seal the media 60 inside the container 20. The inlet port 24 can then be connected to a chromatographic fluid source or fluid pressurizing source 64 and the outlet 26 connected to chromatographic processing equipment 66 using tubing 68 which tubing is typically flexible. The cap 28 is preferably not removable from the container 20 once it is installed by the end user and the locks 29 engage the recesses 41. Thus, any adjustment of the chromatographic packing material 60 or other contents of the container 20 is done before the cap 28 is sealingly fastened to the container 28. The fluid to be processed is then passed through the inlet 30, through the fluid dispersing device 26, through the contents of the container 20 (e.g., through chromatographic packing material 60) and out the outlet 24. After use the container 20 and cap 28 can be discarded.

This user access and easy modification of the contents of the cartridge was not previously possible as the containers were welded shut at the factory to ensure they didn't leak under the operating pressures. There is thus advantageously provided a low cost, disposable cartridge made of a polymer which has a threaded, sealed cap on the container. The locking tabs 39 and mating recesses 41 provide locking means on the container and cap for preventing manual removal of the cap. The locking tab 39 forms a member resiliently urged into a recess, and various arrangements of such resiliently engaging parts, such as various forms of spring loaded detents and spring loaded mating members can be devised to form the locking means, especially given the disclosures herein. The seal 48 and lip 46 provide resilient sealing means for sealing the cap to the container when a user places the cap on the container. The sealing means includes numerous other seal types, including one or more O ring seals interposed between abutting portions of the cap 28 and container. 20

There is also provided a method in which a chromatographic packing material 60 is placed in a container either by the manufacturer, or the user, but with the cap not being locked to the container, as by partially threading the cap onto the container but not engaging the locking tabs 39 with the recesses 41. Alternatively, the cap is not placed on the container. The user removes the cap 28 and either alters the prior amount of chromatographic packing material 60, or adds chromatographic packing materials of a different type, or adds further materials or chemicals to affect the fluid being processed by the user in the cartridge, or even adds analyte or fluid to be analyzed. The user then places the cap 28 on the container and seals and locks the cap to the container 20. The desired processing is then performed using the cartridge and modified sorbent contained in the cartridge. Given the ability to remove the cap 28 and access the inside of the container 20 immediately before fluid is passed through the container, a variety of process variations can be devised.

The inlet and outlet ports 30, 24, respectively preferably comprise fittings adapted for use in chromatographic applications, and Luer fittings are commonly used. Advantageously the desired fittings at ports 24, 30 are integrally molded with the container 20 and cap 28 to form a unitary construction.

Referring to Figure 5, in a further embodiment the fittings can comprise metal or plastic tubes 72 having external engaging threads 74 adapted for use with chromatographic equipment. A ¼-28 threaded fitting is believed suitable for the engaging threads 74. The tubes 72 can have an opposing end with sealing threads 76 configured to sealingly engage mating threads formed at the location of one or more of the ports 24, 30. The threaded portion of the cap 28 and container 20 may need to be thickened to provide sufficient threaded engagement. The sealing threads 76 preferably form a seal suitable for use up to about 100 psi or higher. Using slightly different thread dimensions or lead angles on the mating threads of the fitting 72 and container or cap can help achieve the desired leak proof seal.

Referring to Figure 7 a slightly different cap and container are shown in which there are continuous threads 34, 36. There is no flange 40 on the container 20, and the lip on the container is only slightly inclined away from the longitudinal axis 42. The fluid dispenser 26 is held in a corner formed by a slight inward projection of the cap which projection extends toward the axis 42. The fluid dispenser 26 can be snapped into position in the cap 28, and tightening the cap onto the container preferably, but optionally helps further squeeze the periphery of the dispenser 26 between abutting portions of the cap. Figure 7 shows the top 59 with an annular recess 51 which allows the thickness of the top 50 to remain fairly constant which helps molding of the cap 28. Further, the recess 51 adds flexibility to the sealing surface 48 on the cap 50 and that is believed to enhance the performance of the fluid tight seal which must maintain the seal under flash chromatography and LPLC conditions.

Referring to Figures 8-9, a further embodiment of the fluid dispenser 26 is shown which has a generally a disk shaped support for a plurality of radially extending flow channels 84 having openings 86 in fluid communication with inlet 88. Fluid to be analyzed enters through central inlet 88 that is advantageously located on the longitudinal axis 42, and flows across the cross-section of container 20 (Fig. 1) through channels 86 and then out openings 86 onto the packing material. The openings 86 are preferably at the distal end of each channel 86, but could be located at one or more locations along the length of channel 86. This configuration is more difficult to mold than the fluid dispenser of Figure 1. This embodiment of the fluid dispenser 26 is held between the cap 28 and container 34 as is the prior embodiment of the fluid dispenser. Various ways of holding the fluid dispenser 26 in the desired position will be apparent to one skilled in the art given the disclosures herein, including various clamps, ledges, snap-fits. The various forms of the fluid dispenser 26 comprise means for distributing the fluid to be analyzed over the packing material and over the cross-section of the container 20.

The threads 24, 36 provide means for fastening the cap 28 to the container 20. But the threads represent one specific form of inclined mating surfaces, and other means for fastening the cap to the container include the broader use of inclined mating surfaces. Thus, a lug 78 on one of the cap 28 or container 20 can mate with a bayonet mount 80 on the other of the cap or container to fasten the cap to the container. Placing the recess 41 on a trailing end of an inclined surface on the bayonet could allow the bayonet to also lock the lug into position so as to combine the locking means and the fastening means.

A further embodiment is shown in Figures 10-14. The cap 28 has a continuous skirt 38 that is preferably, but optionally, stiffened with increased thickness areas, such as by using ribs 90. The ribs 90 are preferably, but optionally aligned parallel to axis 42. Several groups with 3-5 ribs in each group are believed suitable. The ribs and any spaces between the ribs provide for an improved manual griping surface to tighten the cap 28. The spacing between groups of ribs is thus advantageously equal.

The inside of the skirt 38 has threads 34 which mate with threads 36 (Fig. 14) on the upstream end of the container 20. The threads are preferably continuous rather than intermittent or segmented threads. For containers having diameters over about 1 inch (about 2.5 cm) the continuous threads provide a more uniform gripping and holding force better suited to resist pressure variations occurring in the container 20 during use in flash chromatography. For containers having threads 36 about 2 inches (about 5 cm) in diameter or greater the threads are preferably pipe threads to provide better holding and sealing.

The sealing surface is modified in this alternative embodiment. The sealing surface is located on an annular sealing flange 48' having a base on its upstream end that is wider than the downstream, distal end. The sealing flange 48' has a conical shape, preferably with a flat distal downstream end. The sealing flange 48' has two inclined surfaces, one inner surface 48a faces radially inward toward axis 42, and the other outer face 48b faces away from axis 42. Inner face 48a abuts a mating face on the container to form a fluid seal as described in more detail later.

The inner face 48a is inclined at an angle α of about 7.5° relative to an axis parallel to axis 42. The opposing surface 48b has a similar angle of inclination, but in the opposing direction, and the angle of surface 48b can vary.

The base of the sealing flange 48' has a groove 92 encircling the upstream inner periphery of the flange. A slightly raised boss 94 is located on the downstream edge of the groove 92. The top wall 50 of the cap 28 joins the upstream side of the groove 92 to provide a generally flat wall joining the upstream wall of groove 92. The juncture with the top wall could be offset a desired distance, but the tangential connection is preferred.

The groove 92 is sized to receive the peripheral edge of the fluid dispensing device 26. The dispensing device is preferably a thin, curved surface with holes 52 (Fig. 7). The diameter of groove 92 is sized to receive the periphery of the dispensing device 26, and is preferably slightly smaller to slightly compress the dispensing device 26. The boss 96 narrows the opening into which the dispensing device 26 is placed, so the circular periphery of the dispensing device 26 forms a snap-fit with the groove 92. The flat portion where the upper wall 50 joins the groove 90 helps guide the peripheral edge of the fluid dispensing device 26 into the groove 90. The inclined inner face 48a also helps guide the fluid dispensing device toward the groove 90. The tight fit between the circular dispensing device 26 and the circular groove 92 allows the dispensing device to resist deformation of the cap 28 by abutting the walls of groove 92 to maintain those walls in a circular configuration.

The annular sealing flange 48' is offset radially inward from the skirt 38 of the cap 28. A shaped annular groove 96 separates the flange 48 from the upstream end of the skirt 38. The groove 96 has a conical cross-section that is narrower at its upstream end and wider at its downstream end. The radially inward side of the groove 96 is formed by the sealing surface 48b and the radially outward side of the groove 96 is formed by inclined surface 98 formed as an inward facing surface on the inside of the skirt 38, at the upstream end of the skirt. The inclined surface 98 is inclined relative to axis 42 at an angle β of about 10°. The groove 96 has an upstream end 100 which is shaped to conform to the lip of the container 20 defining the opening 22. In the illustrated embodiment the end 100 is flat, located in a plane orthogonal to axis 42.

Referring to Figure 14, the container lip 46 is shown as flat, and joins two inclined sealing surfaces comprising inward facing sealing surface 102a and outward facing sealing surface 102b. The sealing surfaces 102a, 102b are inclined toward each other in an upstream direction to form a generally conical cross section having a top surface comprising lip 46.

The sealing surfaces are about .2 inches (.5 cm) long measured along axis 42. Inward facing sealing surface 102a is inclined at an angle Δ of about 15° relative to axis 42, and sealing surface 102b is inclined at an angle θ of about 10° relative to axis 42. Threads 36 extend from the downstream end of the surfaces 102a, 102b to the outwardly extending flange 40. Shaped groove 96 is formed to receive the lip 46, with the surface 98 having a length about the same as or slightly longer than abutting surface 102b. The threads 36 in the cap 28 preferably stop before inclined sealing surface 98, but could be formed in the inclined surface 98.

The lip 46 defines the upstream opening 22 to the container 20. The container has a downstream end that is domed to better withstand increased operating pressures and to accommodate larger diameter containers 20.

Referring to Figures 13-15, the seal between the cap 28 and container 20 is described. The shaped groove 96 is configured to receive and seals both sides of the container adjacent the lip 46. The inward facing surface 98 on the skirt 38 of the cap abuts and seals against outward facing surface 102b of the container lip. The outward facing surface 48b of the annular seal 48' abuts and seals against the inward facing surface 102a of the container's lip. The surfaces 102a, 102b are inclined at 15° and 10°, respectively, and they abut surfaces 48b and 98 respectively, which are inclined at angles of about 7.5° and 10°, respectively. The abutting surfaces 102a, 48b are inclined so there is an angle of about 7.5° of interference. The abutting surfaces 102b, 98 are inclined so there is little or no interference. The interference fit, abutting surfaces 48b, 102a are radially inward of abutting surfaces 98, 102b. The skirt 38 and mating surfaces 102b, 98 thus form a relatively rigid support to prevent radially outward movement of inclined surface 48b, so that surface 102a is tightly forced against mating surface 48b to form a seal.

Advantageously the lip 46 is advanced into shaped groove 96 until the lip 46 abuts the end 100 to form a further sealing surface. Outwardly extending flange 40 abuts the lip 44 on the cap to prevent over-tightening and stripping of the threads 34, 36.

Viewed in cross-section, the lip 46 of the container 20 forms a trapezoidal surface and the shaped channel has a similar shaped trapezoidal cross-sectional shape in which the outward located inclined walls are inclined at about the same angle, while the inward located walls are inclined for an interference, sealing fit. During use the pressure in low pressure liquid chromatography container 20 can reach 100 psi or more. The radially outward pressure on inner surface 48a urges the flange 48' against the container interposed between the flange 48' and skirt 38 to provide further sealing pressure between the abutting surfaces 48b and 102a, and between abutting surfaces 102b and 98.

Further, the circular groove 92 is located downstream of the end 100 of the shaped groove 96, and on opposing sides of the annular flange 48'. The circular groove 92 forms a narrower cross-section at the base of the flange 48' so that the flange tends to flex locally at or by the location of the groove. That localized bending allows a greater contact surface between abutting surfaces 48b and 102a. The localized bending at circular groove 92 also causes the walls forming the groove 92 to more tightly grip the periphery of the fluid dispensing device 26 located in the groove, and that provides greater resistance to deformation of the circular shape of the groove that may be caused by increased pressure in the low pressure liquid chromatographic container 20.

The angles α and Δ can vary, but advantageously create an interference on the mating surfaces. Interference angles of 5-15° are believed suitable. The angle α can be from about 7° to 15°. Larger angles up to about 30° are believed possible, but are not as suitable. Angles of 6° or less are not desirable because they can leak at higher pressures and/or with larger diameters of the container 20. The angles β and θ are preferably the same, and can vary from the 10° angle of the preferred embodiment. Angles from 7-15° are believed suitable for β and θ. Slight interference angles can be used, but are not preferred. The above angles and dimensions are believed suitable when the cap 28 and container 20 are made of polypropylene, such as PP9074 med polypropylene. The dimensions can vary with different materials and operating conditions for the low pressure liquid chromatographic container 20.

Referring to Figures 16-19, a further embodiment is disclosed that is particularly suited for larger diameter containers 20, or higher operating pressures, or both. In this embodiment, the skirt 38 is thickened in the radial dimension, and the top wall 50 is also thickened. The continuous internal threads 34 comprise power threads having lower angles of inclination on the leading thread face and an even lower angle of inclination on the trailing face of the thread. A flat crest and an increased thickness is typically provided between the thread root and crest. A NPT Thread is preferred. These NPT Threads require more force to unscrew the threads and that prevents loosening of the cap under pressure variations that may occur during chromatographic use. The thickened threads also carry more force along the direction of axis 42 so as to accommodate larger forces on the cap 28. The thickened flange provides radial stiffness and further ensures a sliding interference fit between surfaces 48b and 102a (Fig. 14).

The threads 34 can stop at about the location on axis 42 of the distal end of flange 48' and the beginning of the shaped recess 96, or the threads can continue into the shaped recess 96 as is shown. When the threads 34 extend into the inclined surface 98, the surface becomes intermittent and the seal is not as good. The container 20 will have correspondingly shaped external or male NPT Threads 36, but those are not shown.

Preferably, but optionally, the cap 28 also has a number of external ribs 104 to strengthen the cap. Preferably, but optionally, some of ribs 104 extend only over the top wall 50, while others continue along the exterior of the flange 38. The depicted embodiment has two ribs 104a extending only over the top wall 50, and two ribs extending over the top wall 50 and the flange 38. In addition to strengthening the cap 50, the rigs also provide a manual gripping surface.

In the embodiments of Figures 10-19, it is believed possible to omit the inclined surfaces 98, 102b and provide the seal between inclined surfaces 102a and 48b. The surfaces 48b and 102a are generally aligned, as are the inclined surfaces 98 and 102a. While the surfaces may be inclined to form an interference fit as they slide along each other as the cap is advanced along axis 42 as it is screwed onto the container, the surfaces are aligned sufficiently to abut and form a sealing surface.

In the embodiments of Figures 10-19, the fluid inlet 30 is integrally molded with the cap 28. But it could be an inserted fitting as described herein. Likewise the outlet is shown as integrally molded, but it could be an inserted fitting as described herein.

As required, detailed embodiments of the present invention are disclosed herein; however, it is to be understood that the disclosed embodiments are merely exemplary of the invention, which may be embodied in various forms. Therefore, specific structural and functional details disclosed herein are not to be interpreted as limiting, but merely as a basis for the claims and as a representative basis for teaching one skilled in the art to variously employ the present invention in virtually any appropriately detailed structure.

The above description is given by way of example, and not limitation. Given the above disclosure, one skilled in the art could devise variations that are within the scope and spirit of the invention, including various ways of sealing the cap to the container and various process steps that alter the material in the container through which the fluid being analyzed is passed. Further, the various features of this invention can be used alone, or in varying combinations with each other and are not intended to be limited to the specific combination described herein. Thus, the invention is not to be limited by the illustrated embodiments but is to be defined by the following claims when read in the broadest reasonable manner to preserve the validity of the claims.

WHAT IS CLAIMED IS:

1. A low pressure liquid chromatographic cartridge having a longitudinal axis, comprising:

a tubular polymer container adapted to receive a chromatographic packing material, the container having an outlet port located at a downstream end of the container and configured for connecting to chromatographic equipment during use of the cartridge, the container having container threads formed on an upstream end of the container and a lip defining an opening to the container with a inward facing inclined sealing surface adjacent the lip and facing the longitudinal axis;

a polymer cap having an inlet port located on an upstream end of the container, the port being configured for connecting to chromatographic equipment during use of the cartridge, the cap having cap threads located on a skirt of the cap to threadingly engage the container threads;

an annular sealing flange depending from the cap and located radially closer to the longitudinal axis than the skirt, the flange having an outward facing inclined sealing surface facing away from the axis with an upstream end of the flange being further from the axis than a downstream distal end of the flange; the outward facing inclined surface on the flange generally aligned with and located to abut the inward facing inclined surface on the container to form a resilient fluid tight seal between the cap and container suitable for use in low pressure liquid chromatography when the cap is screwed onto the container.

2. The cartridge of Claim 1, wherein the abutting inclined surfaces abut at an interference angle of about 7-8°.

3. The cartridge of Claim 1, wherein the container further has an outward facing sealing surface facing away from the longitudinal axis and located opposite the container's inward facing sealing surface, the container's inward and outward facing surfaces being inclined in opposite directions, and wherein the skirt has an inward facing sealing surface located and inclined to generally align with the outward facing sealing surface of the container when the cap is fastened to the container.

4. The cartridge of Claim 3, wherein the outward facing surface on the flange is inclined at an angle of about 7°, the inward facing surface on the skirt is

inclined at about 10°, the outward facing surface on the container is inclined at an angle of about 15°, all angles being relative to an axis parallel to the longitudinal axis.

5. The cartridge of Claim 3, further comprising a groove at a base of the flange forming a localized bending location of the flange when the cap is screwed onto the container.

6. The cartridge of Claim 3, further comprising a fluid dispenser interposed between the container and the cap and having at least several of fluid outlets located across a substantial portion of a cross section of the container.

7. The cartridge of Claim 3, further comprising a groove at a base of the flange, and

a fluid dispenser having a periphery located in the groove, the dispenser having at least several fluid outlets located across a substantial portion of a cross section of the container.

8. The cartridge of Claim 1, wherein the threads are power threads.

9. The cartridge of Claim 1, wherein there are external ribs on the cap.

10. The cartridge of Claim 1, wherein the cap has a domed top in which is located the inlet.

11. The cartridge of Claim 1, wherein the container has a downstream end that is domed with the outlet located in the domed portion.

12. The cartridge of Claim 1, wherein there is a shaped space between the annular flange on the cap and the skirt, the shaped space having a trapezoidal shaped sides and top, and wherein the lip of the container has a generally corresponding trapezoidal shape with an interference fit occurring on at least one side of the trapezoid.

13. The cartridge of Claim 1, wherein the inlet comprises a tube threadingly engaging one of the cap or container, the tube having a threaded exterior distal end located on an exterior of the engaged one of the cap or container.

14. The cartridge of Claim 1, wherein the outward facing surface on the flange and the lip of the container abut at an inclined angle with the outward facing surface extending inward toward the longitudinal axis of the container and cap.

15. The cartridge of Claim 1, further comprising a chromatographic packing material placed in the cartridge by the user before the cap is screwed onto the container.

16. The cartridge of Claim 1, further comprising a plurality of different chromatographic packing materials in the container.

17. The cartridge of Claim 1, further comprising a lock on the container mating with a lock on the cap.

18. A low pressure liquid chromatography cartridge, comprising:

a tubular polymer container adapted to receive a chromatographic packing material, the container having an outlet port located at a downstream end of the container and configured for use with chromatographic equipment during use of the cartridge, the container having container threads formed on an upstream end of the container;

a polymer cap having an inlet port located on an upstream end of the cap, the port being configured for use with chromatographic equipment during use of the cartridge, the cap having continuous cap threads located on a skirt of the cap to threadingly engage the container threads;

resilient sealing means for sealing the cap to the container sufficiently to maintain a seal suitable for low pressure chromatographic use when a user screws the cap onto the container.

19. The cartridge of Claim 18, further comprising fluid distribution means for distributing fluid from the inlet port over a cross-section area of the container during use of the cartridge.

20. The cartridge of Claim 18, further comprising chromatographic packing material placed in the container by the user of the cartridge before the locking means are locked.

21. The cartridge of Claim 18, wherein the resilient sealing means comprises a cap with two inclined surfaces each mating with a corresponding surface on the container that are generally inclined to mate with the inclined surfaces on the cap.

22. The cartridge of Claim 18, wherein the resilient sealing means comprises a container with an outwardly inclined lip abutting the flange on the cap to force the flange inward toward the longitudinal axis.

23. A method for a user to perform low pressure liquid chromatography, comprising:

placing at least one of a material to be analyzed or a chromatographic packing material in a tubular polymer container having an outlet port located at a downstream end of the container and configured for use with chromatographic equipment, the container having container threads formed on an upstream end of the container;

threadingly engaging threads on a polymer cap with the container threads, the cap having an inlet port on an upstream end of the cap; and

sealing the cap to the container by tightening the threads and engaging a seal between the cap and the container, the seal providing a fluid tight seal below about 100 psi suitable for LPLC use.

24. The method of Claim 23, further comprising locking the cap to the container.

25. The method of Claim 23, wherein the user changes one of the sorbent type, sorbent volume, empty volume of the container, or adds a fluid before sealing the cap to the container.

26. The method of Claim 23, further comprising connecting the inlet to a source of fluid for chromatographic analysis; and

distributing the fluid from the inlet over a cross-section area of the container.

27. The method of Claim 26, wherein the distributing step comprises collecting the fluid in a fluid dispenser having a wall with a plurality of holes spread across the cross-section and passing the fluid through those holes.

28. The method of Claim 27, further comprising inclining the surface with the holes toward a central longitudinal axis of the fluid dispenser which also passes through the fluid dispenser.

29. The method of Claim 26, wherein the distributing step is performed by a fluid dispenser with a periphery that is interposed between the cap and the container.

30. The method of Claim 23, wherein the placing step comprises placing a chromatographic packing material in the container or removing chromatographic packing material from the container.

31. The method of Claim 23, wherein the sealing step comprises abutting first and second oppositely inclined surfaces on the cap with first and second oppositely inclined surfaces on opposing sides of a lip of the container, the abutting surfaces being generally aligned.

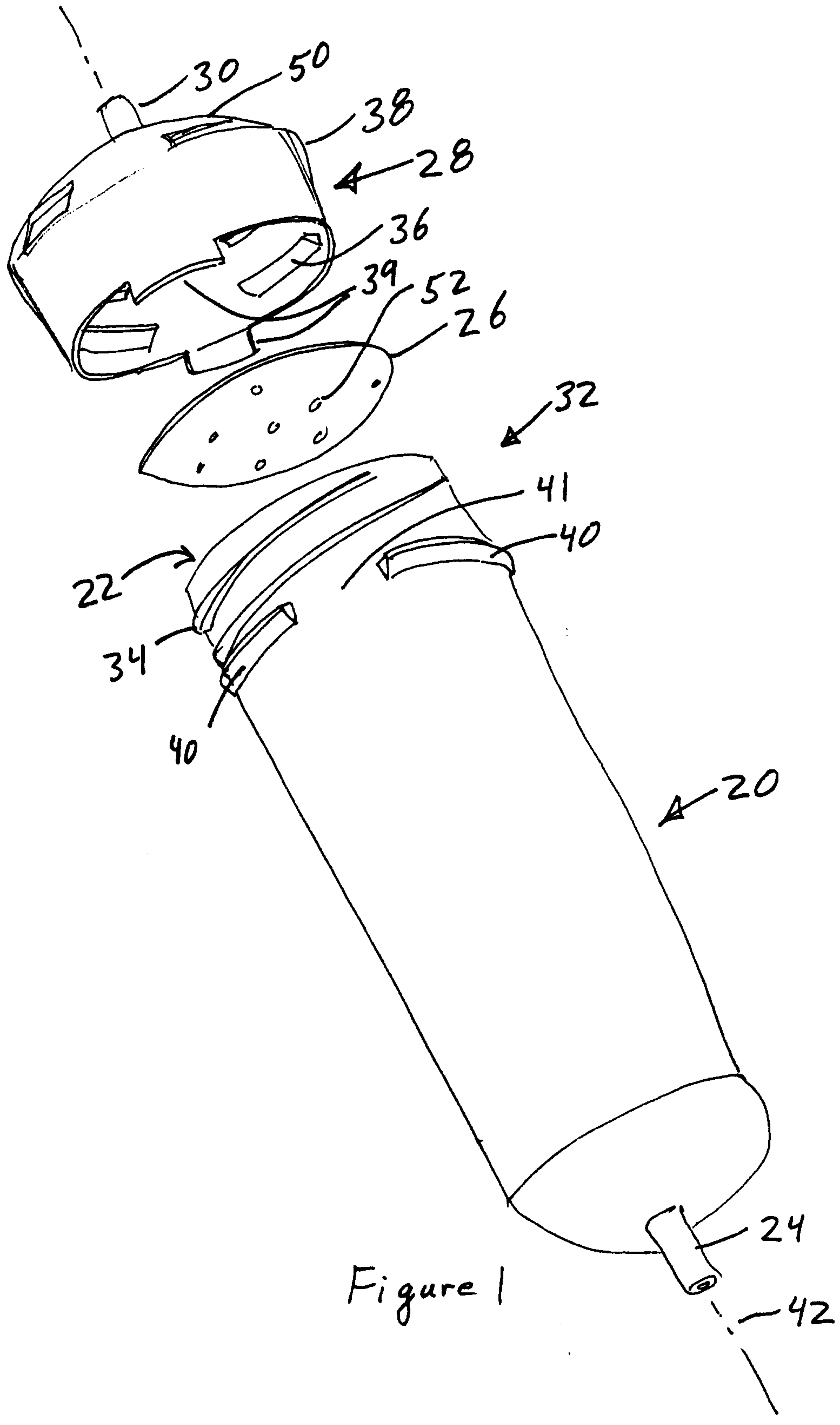


Figure 1

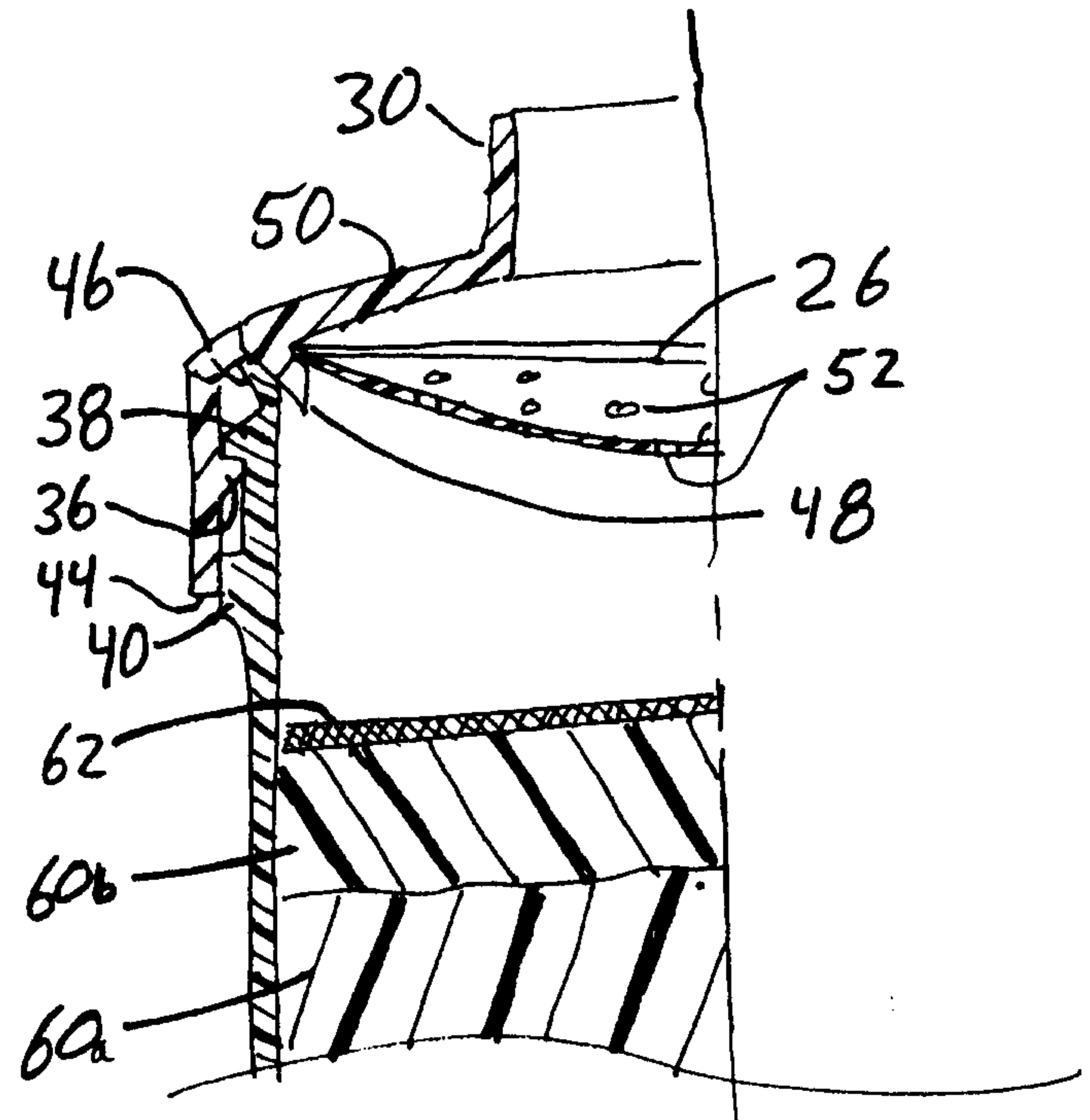


Figure 2

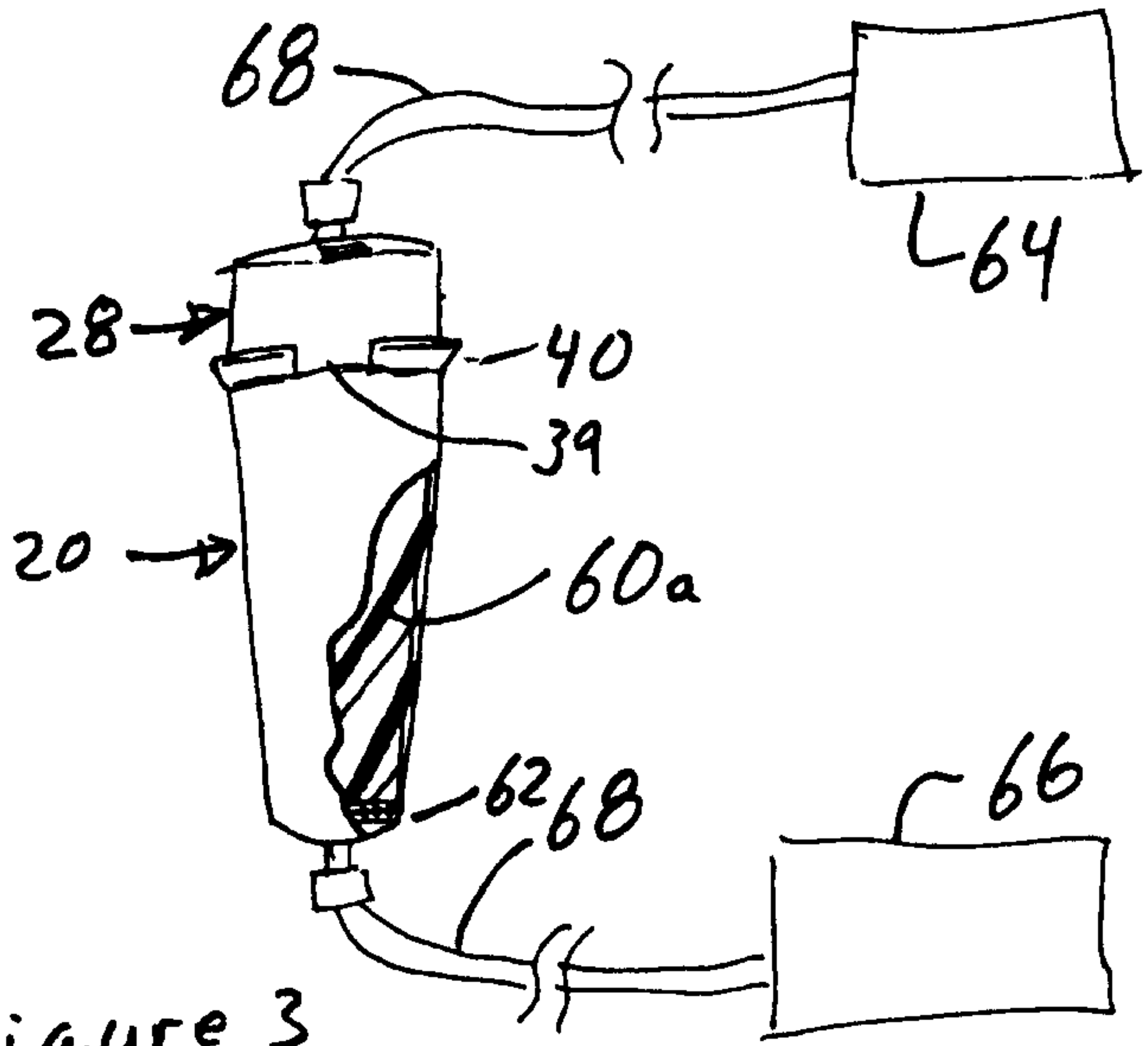


Figure 3

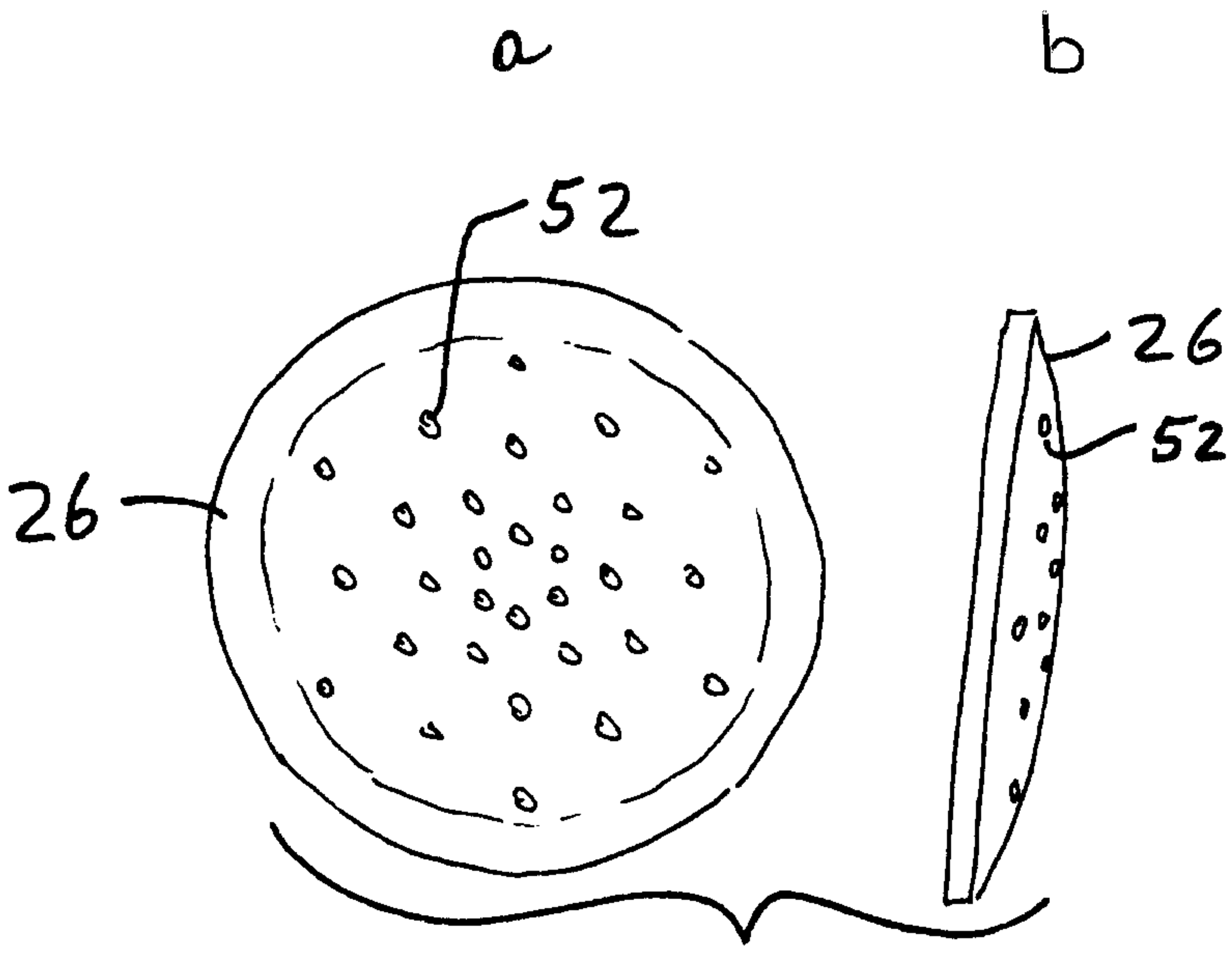


Figure 4

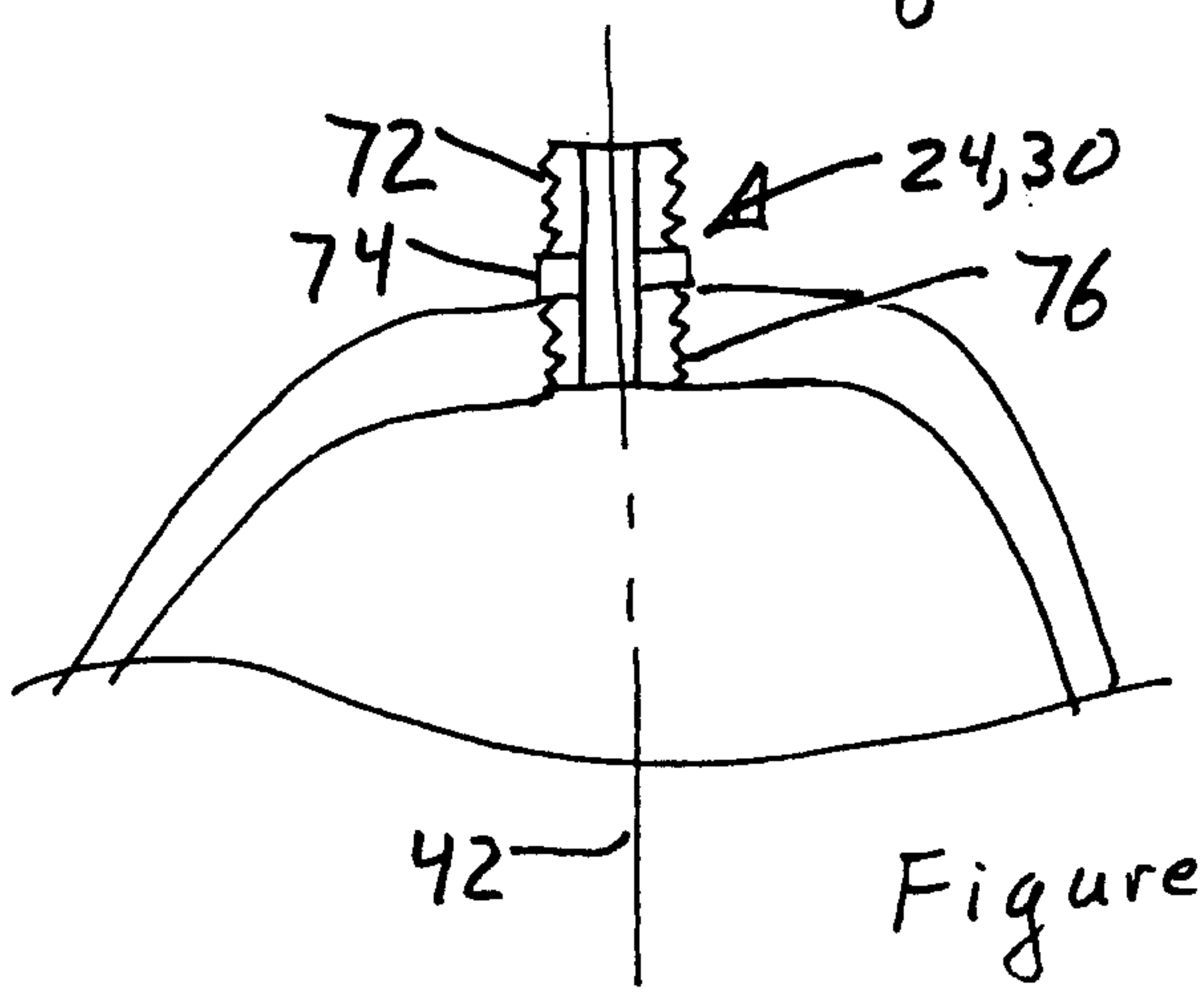


Figure 5

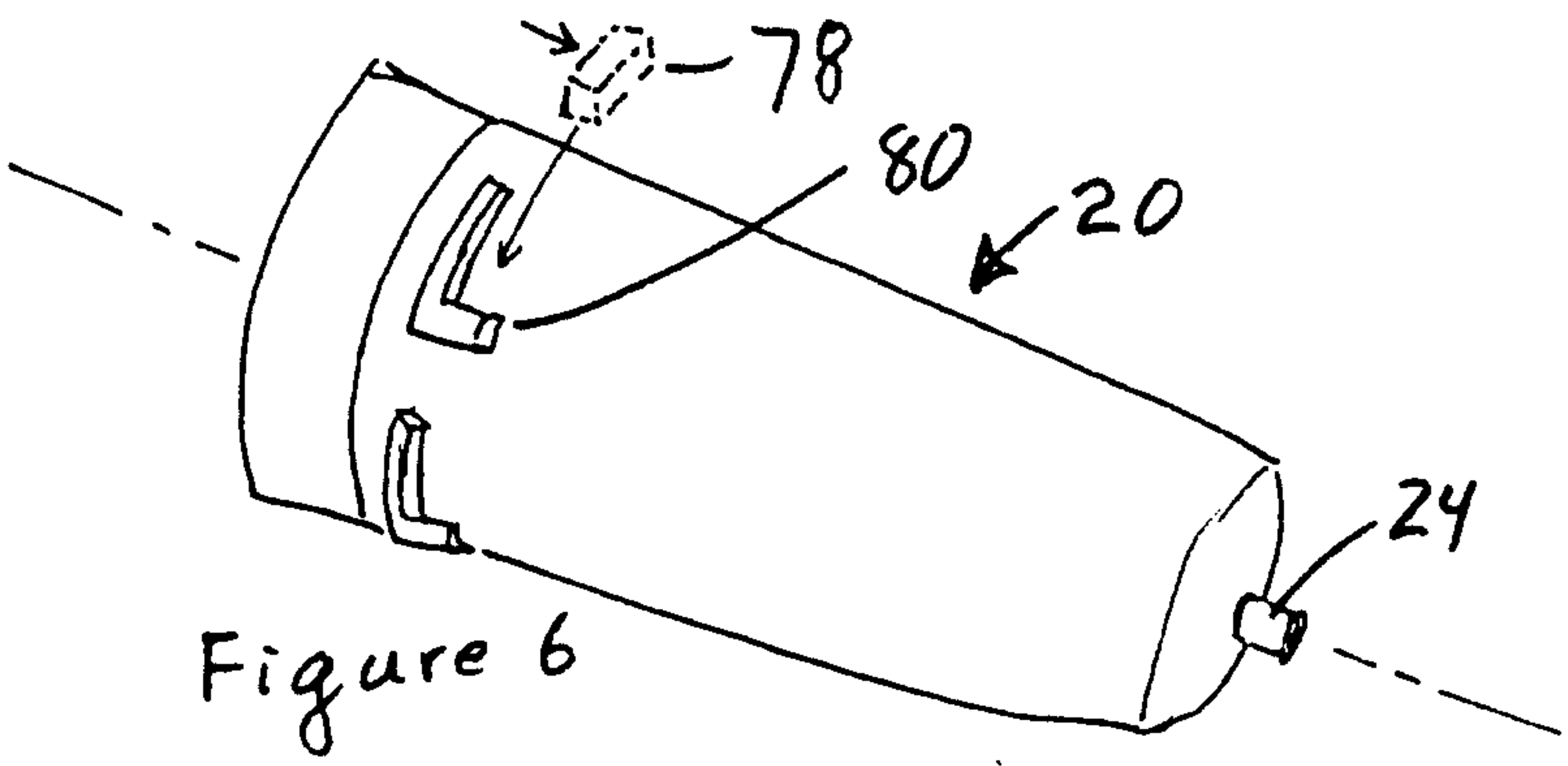


Figure 6

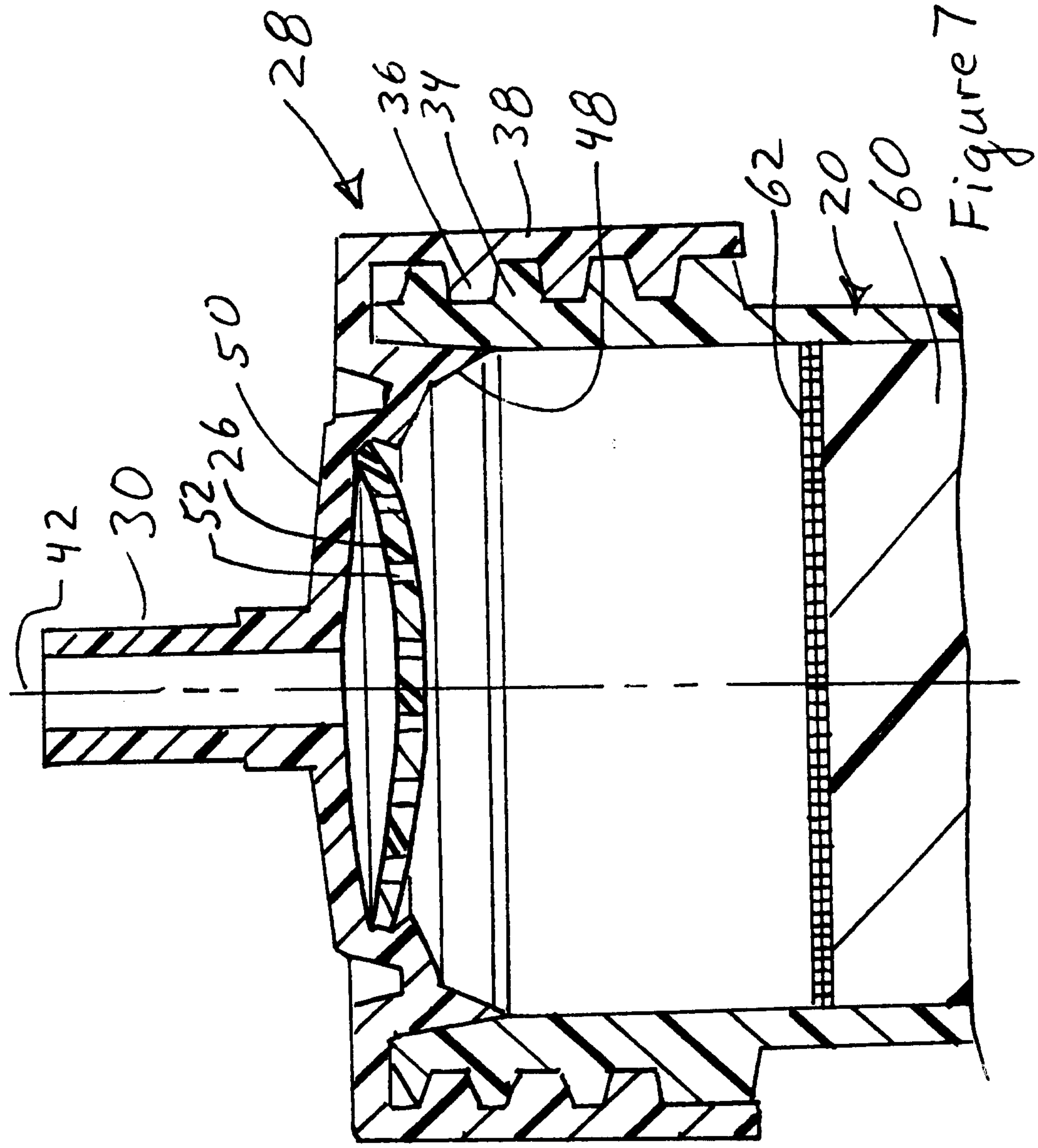


Figure 7

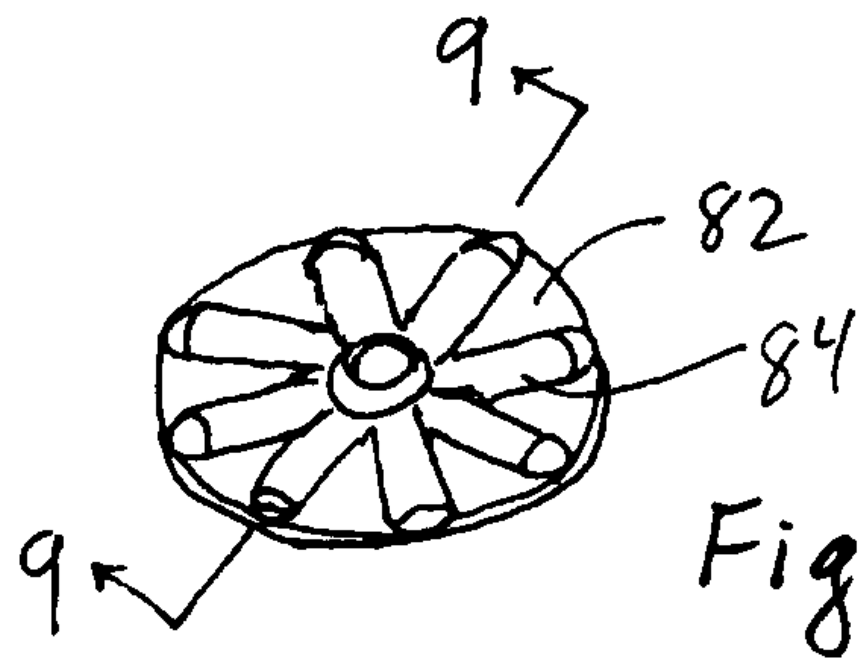


Figure 8

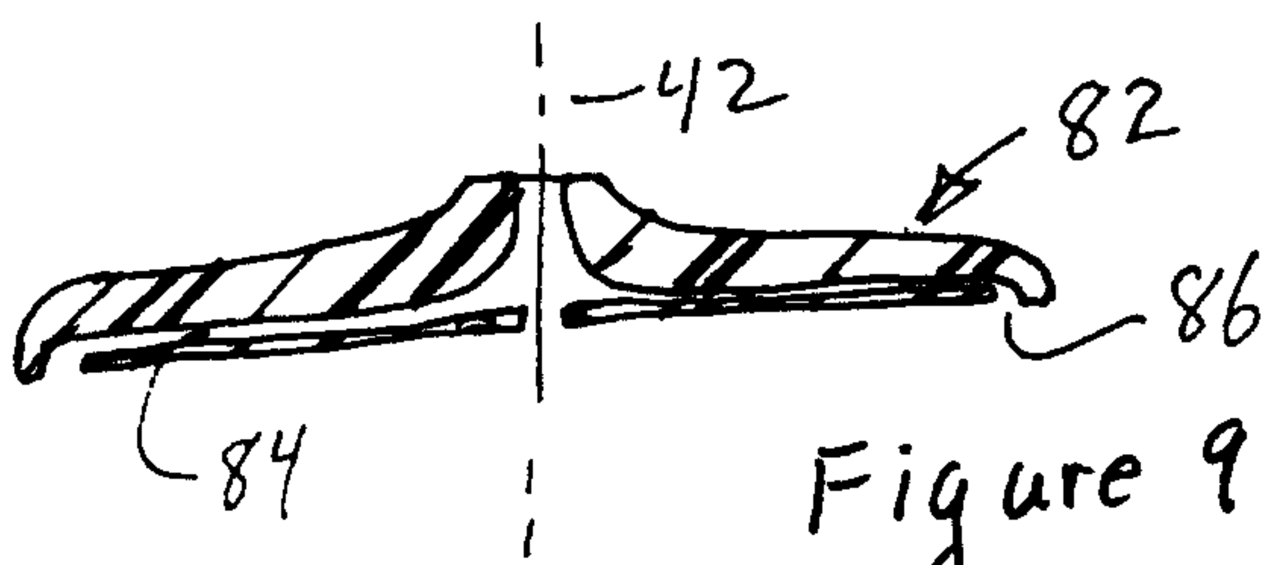


Figure 9

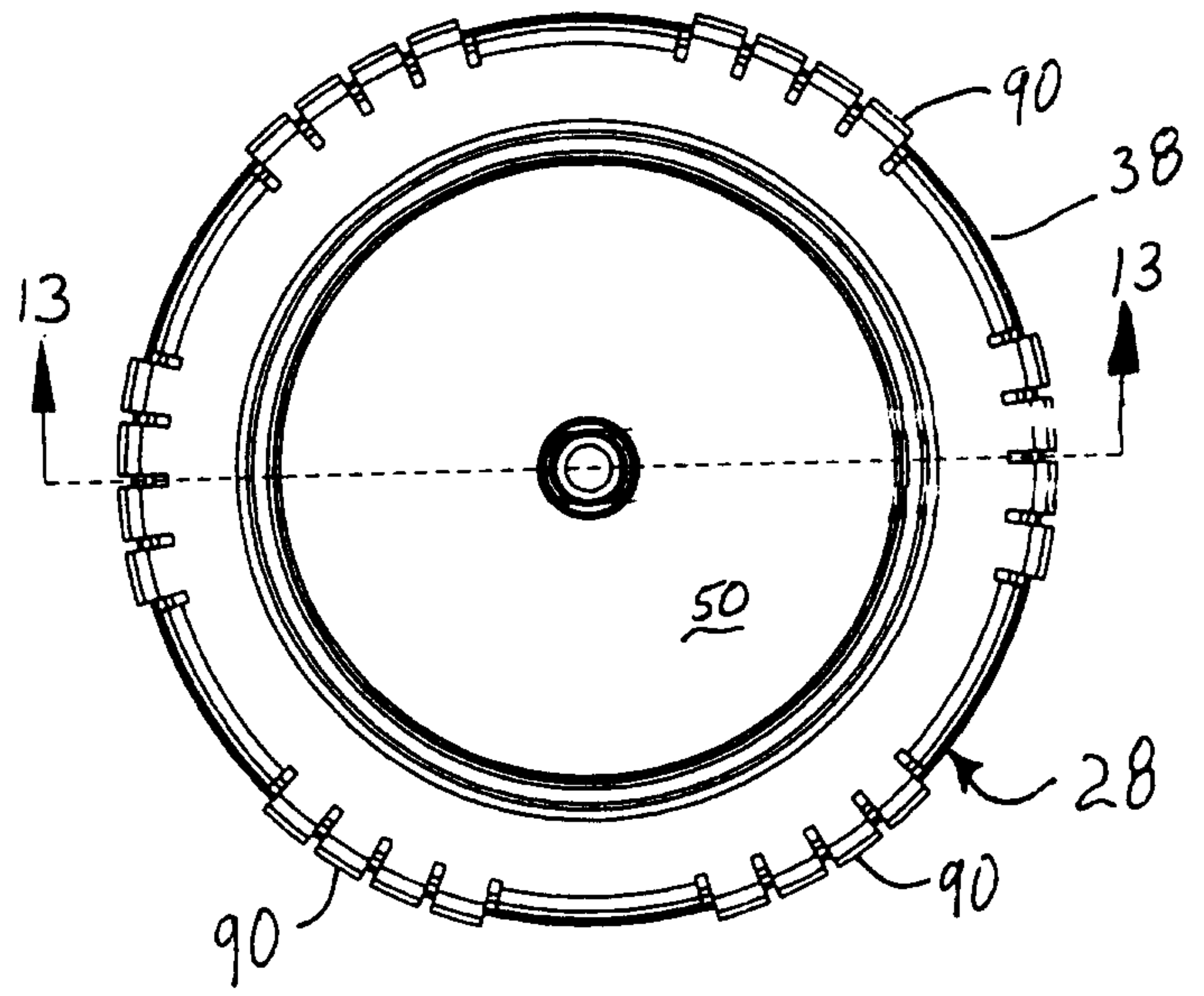


Fig. 12

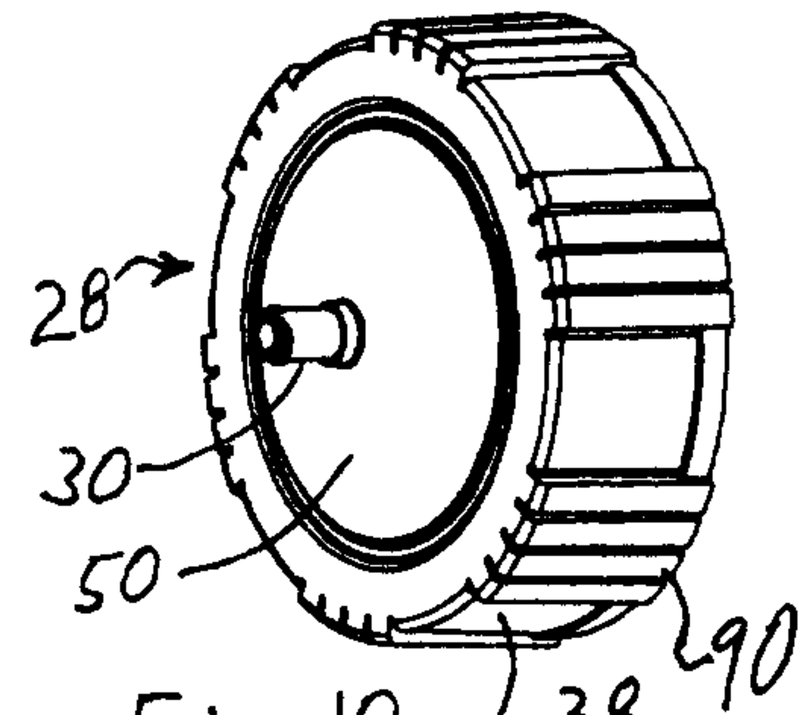


Fig. 10

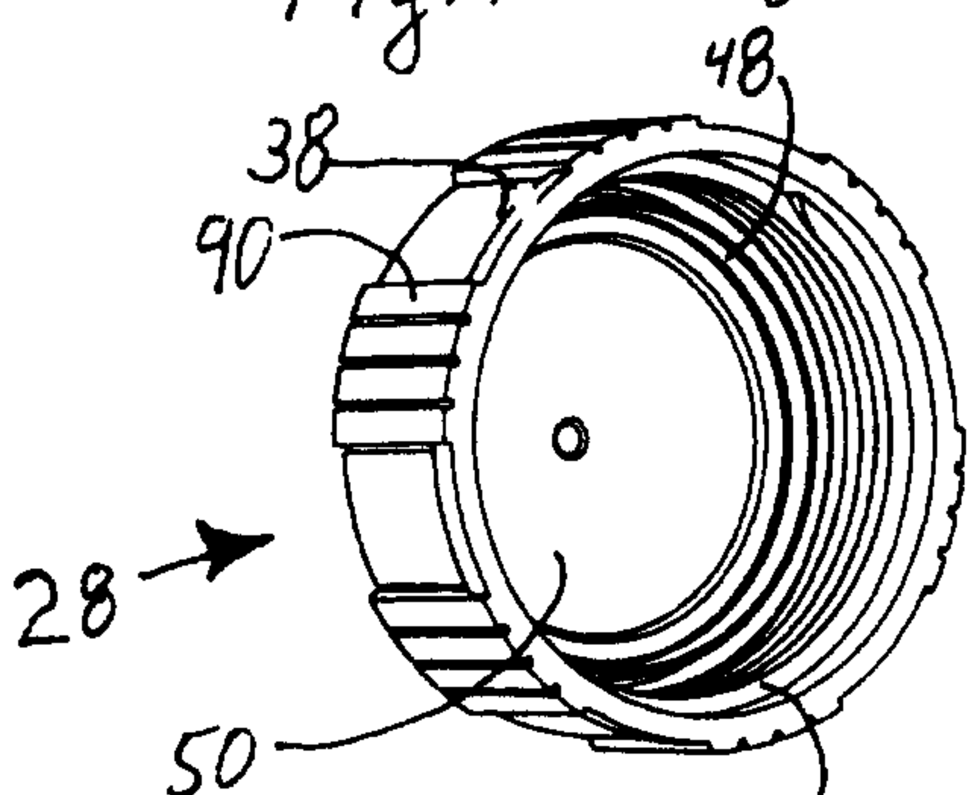


Fig. 11

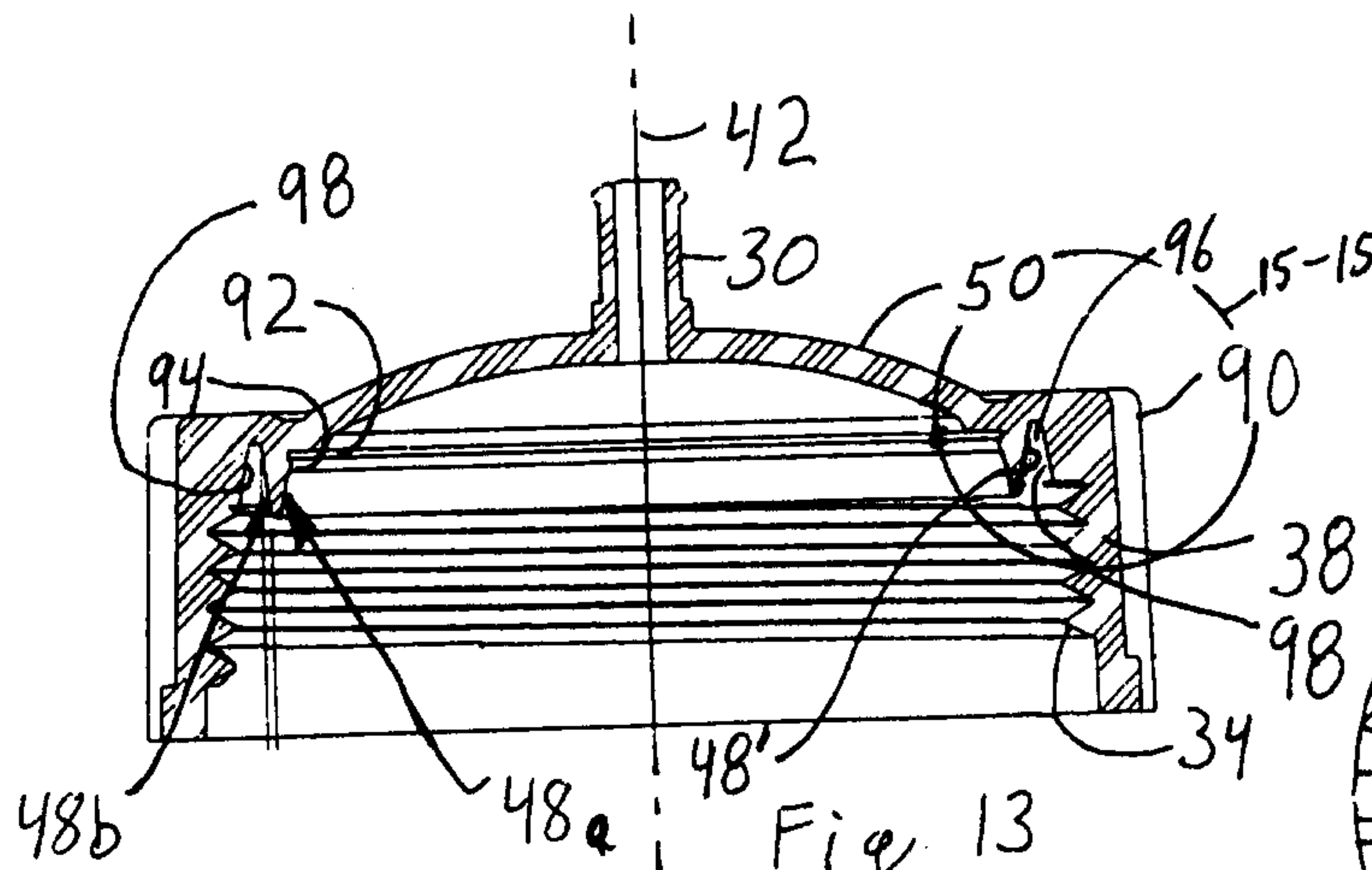


Fig 13

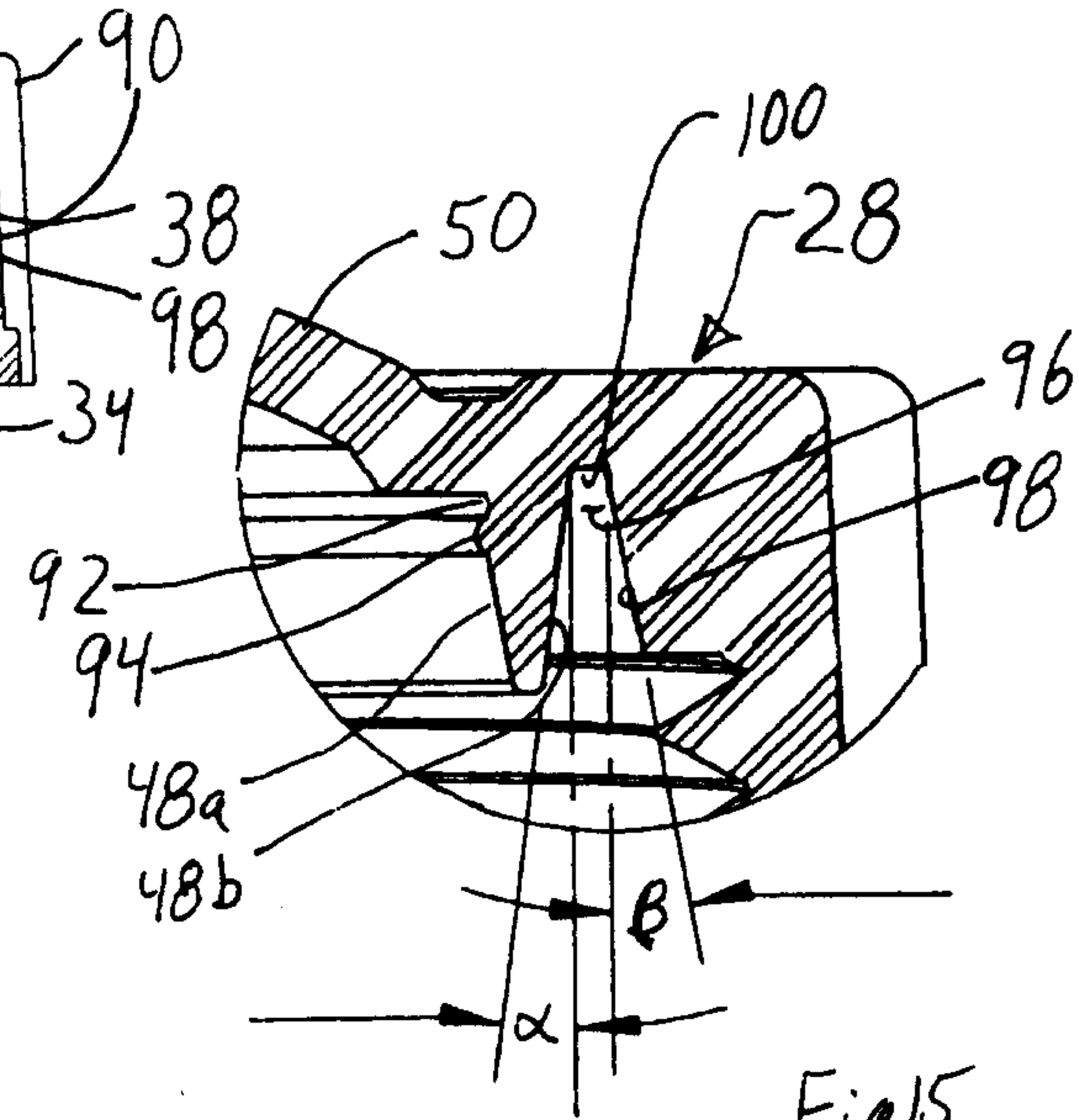


Fig 15

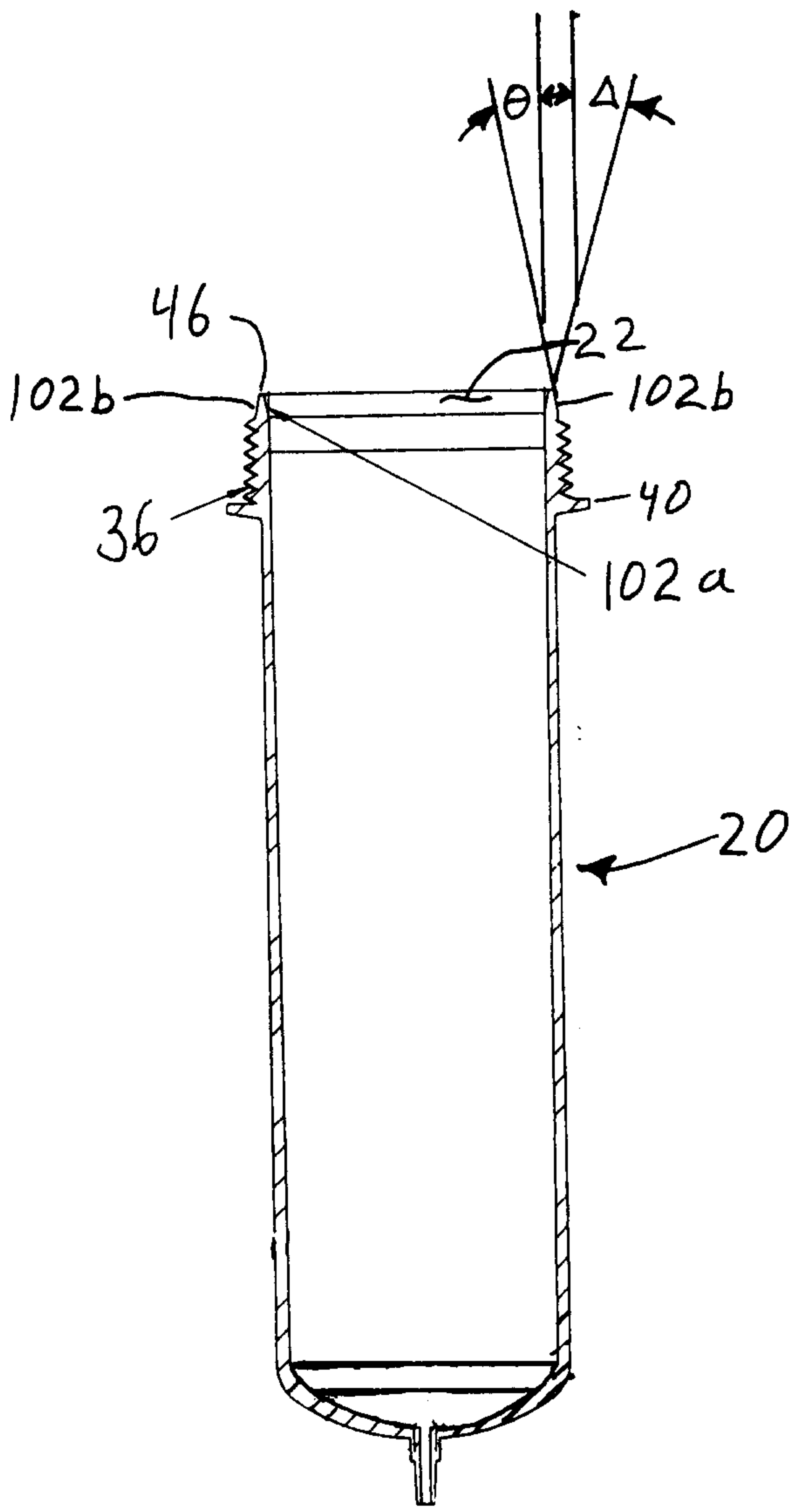


Fig. 14

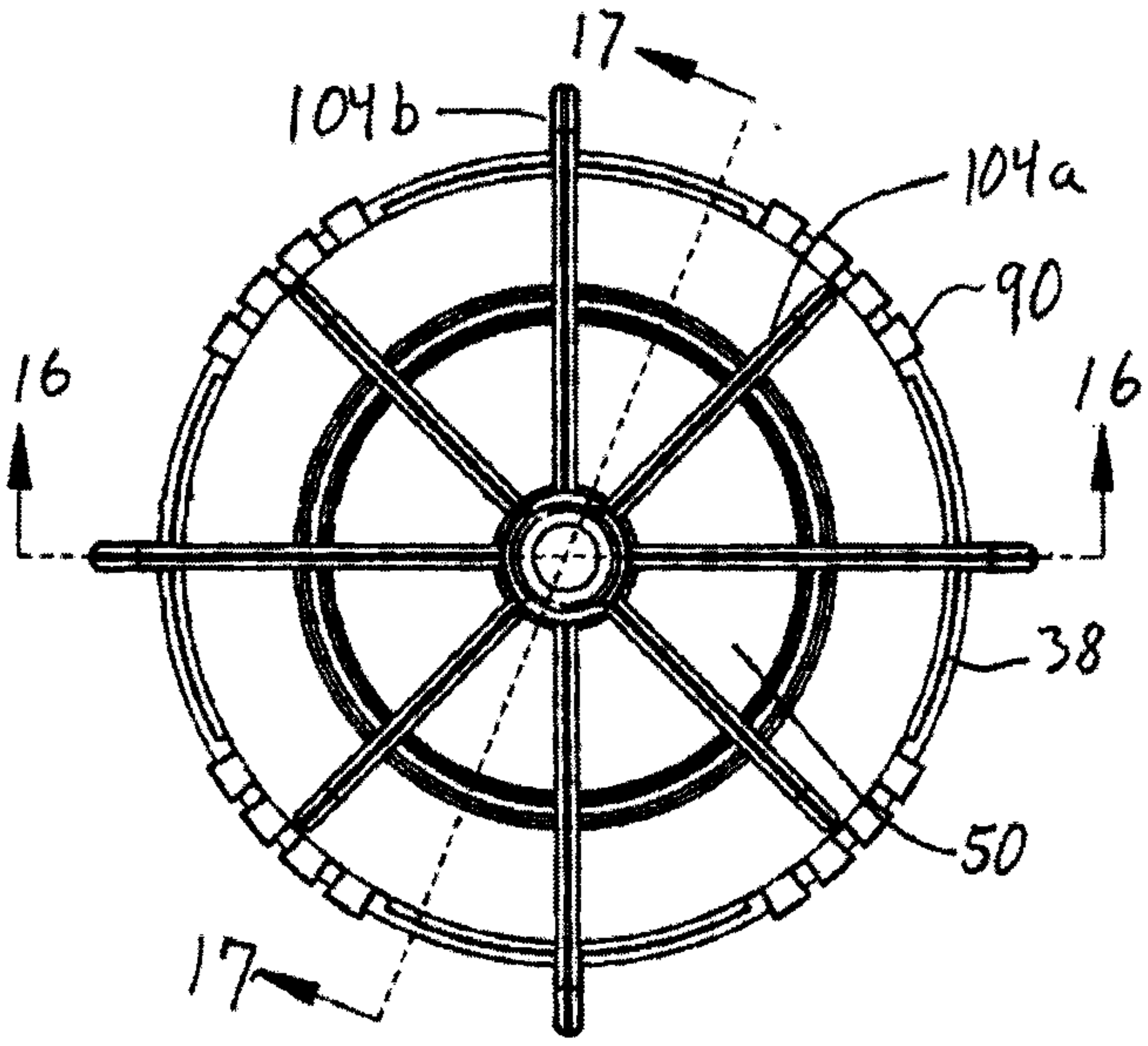


Fig. 19

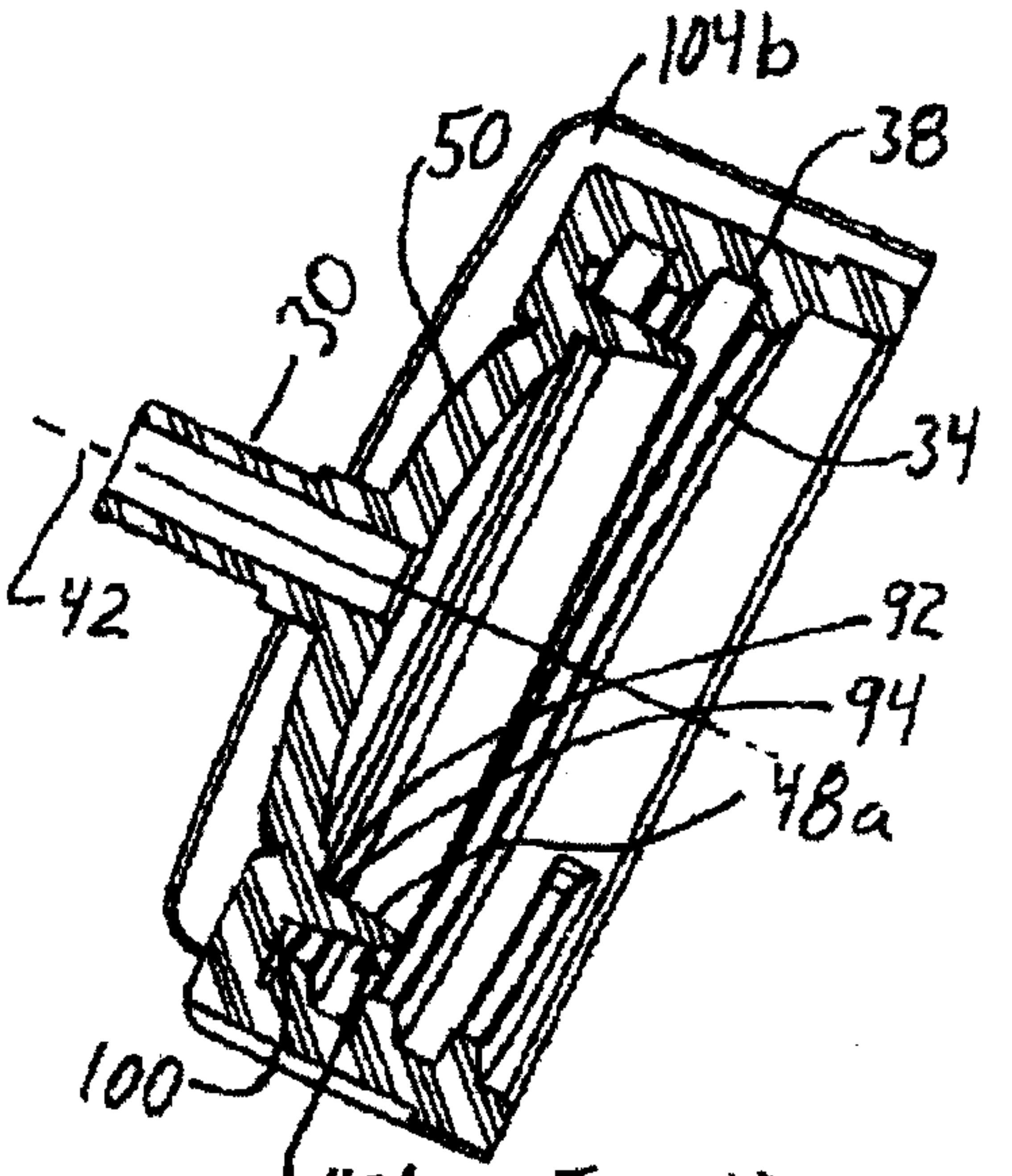


Fig. 17

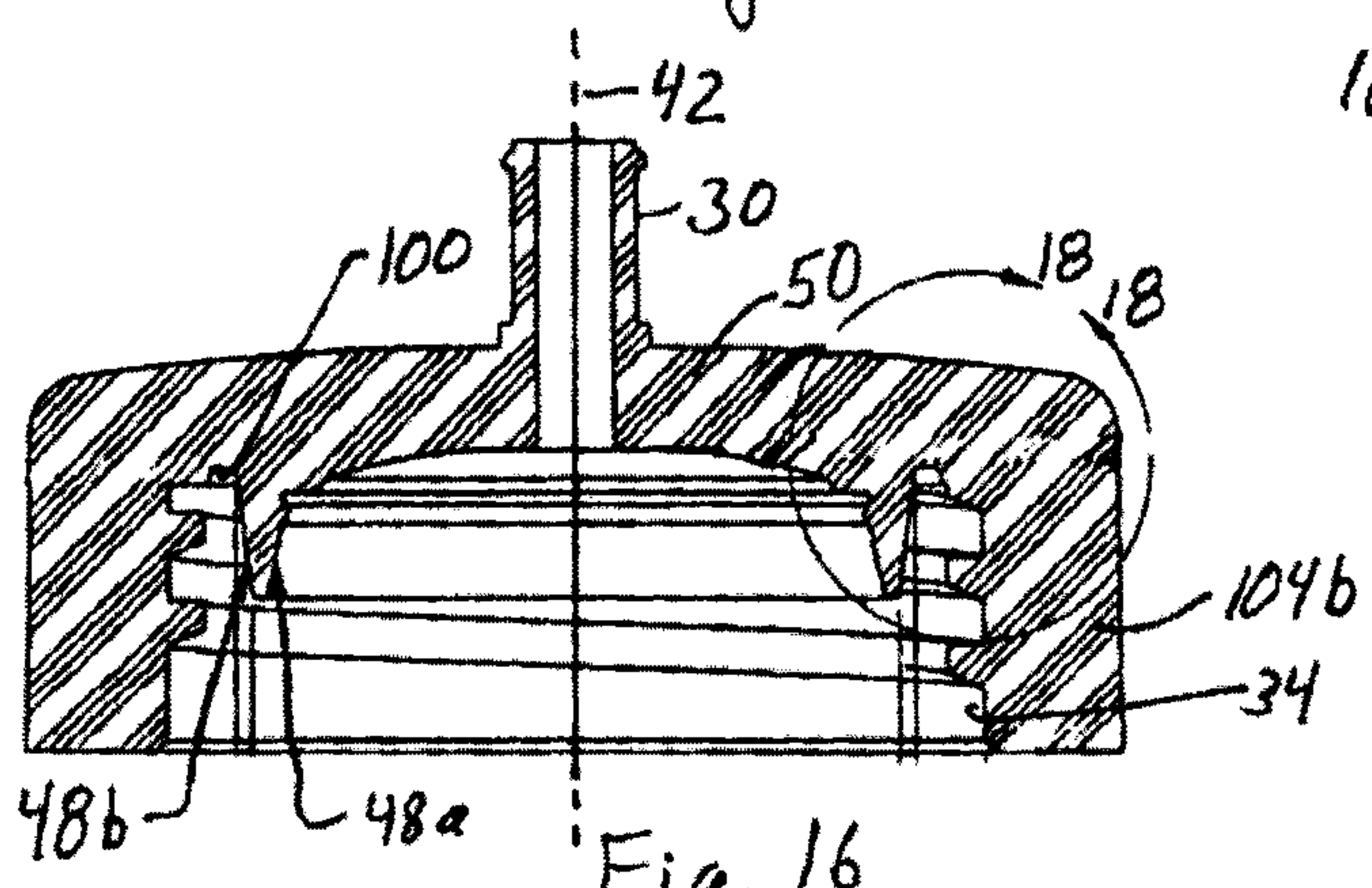


Fig. 16

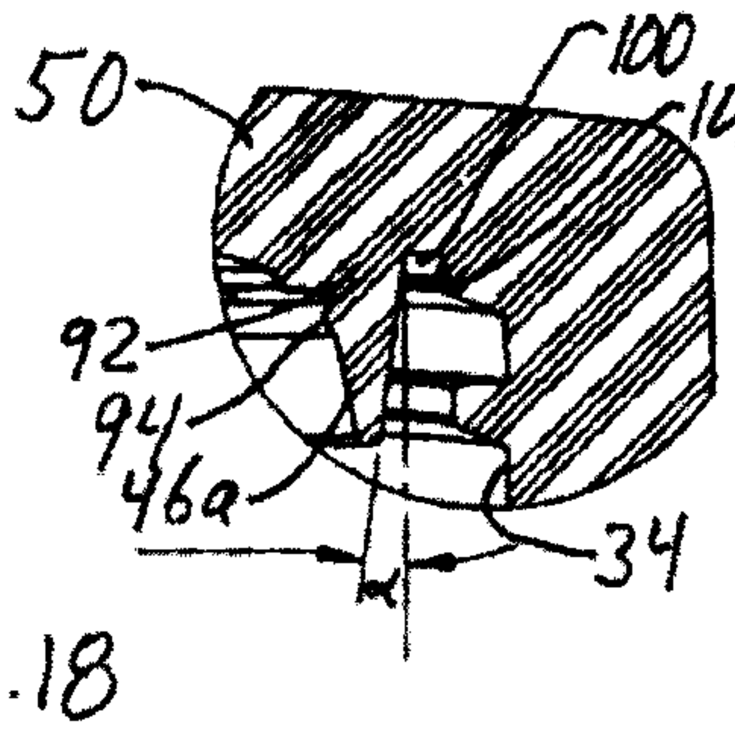
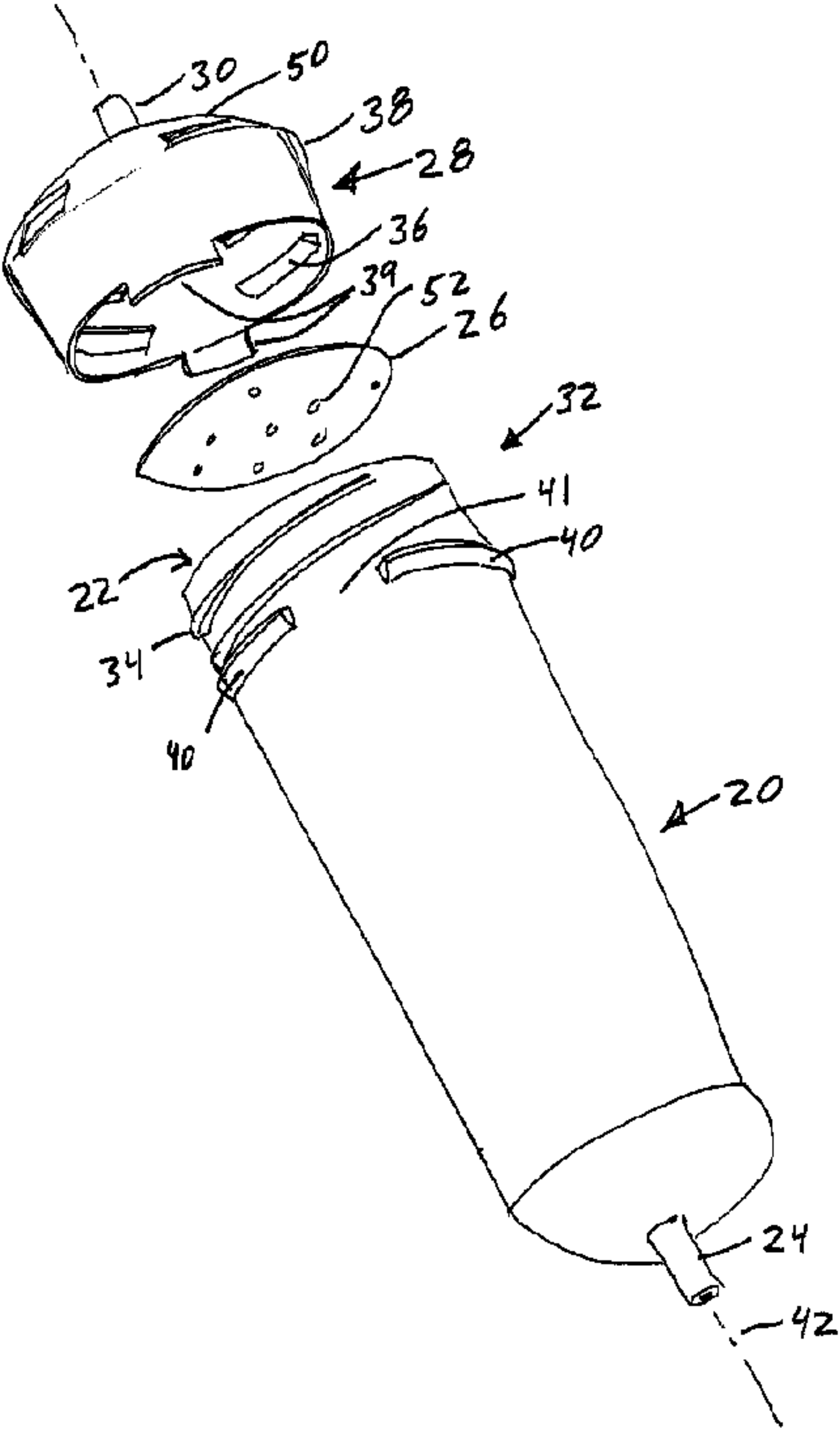


Fig. 18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