United States Patent [19]

Harris, Sr. et al.

[11] **3,824,859**

[45] **July 23, 1974**

[54] **AUTOMATIC FLUID INJECTOR**

[76] Inventors: **Rano J. Harris, Sr.,** 1945 Carolyn Sue Dr.; **Rano J. Harris, Jr.,** 9880 S. Riveroaks Dr., both of Baton Rouge, La.

[22] Filed: **Feb. 16, 1973**

[21] Appl. No.: **333,120**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 223,663, Feb. 4, 1972, Pat. No. 3,754,443.

[52] **U.S. Cl.** ..... **73/422 GC,** 73/423 A

[51] **Int. Cl.** **G01n 1/14**

[58] **Field of Search**..... 73/423 A, 422 GC, 425.6; 128/218 A; 23/259

[56] **References Cited**

UNITED STATES PATENTS

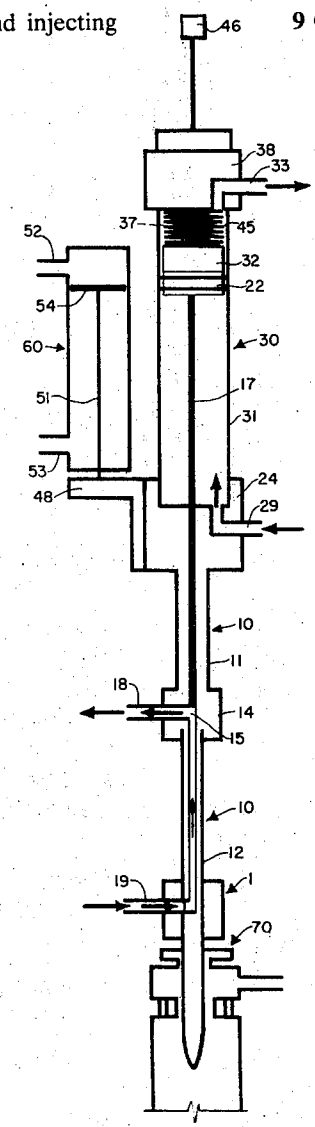
3,754,443 8/1973 Harris..... 73/422 GC

Primary Examiner—S. Clement Swisher

[57] **ABSTRACT**

An apparatus for accurately measuring and injecting quantities of fluid specimens, or samples, as received from a flowing sampled stream, into various media, e.g., a receptacle or inlet of a modern analytical instrument. The automatic fluid injector, or continuous flow analyzer, is comprised generally of (a) a support means, (b) a tubular mounting member, generally mounted on said support means, said member providing an axial opening which can be aligned upon said inlet, and including a transverse opening entering therein through which fluid can be transferred from a flowing stream, (c) a needle syringe, including a barrel and a plunger reciprocable within the barrel and bore of a needle, mounted on the forward end of the syringe, for insertion into the axial opening and for alignment of a side opening provided in the needle for alignment with the transverse opening of the tubular mounting member for receipt of a fluid specimen into the needle bore on withdrawal of the plunger, and (d) an associated cylinder piston unit, the needle syringe of which is mounted on the forward end of the piston. Both the plunger of the syringe and piston of the cylinder piston unit are reciprocable, on actuation in response to automatic control means to accurately measure, trap and then inject fluid specimens from the flowing stream, as desired.

9 Claims, 15 Drawing Figures



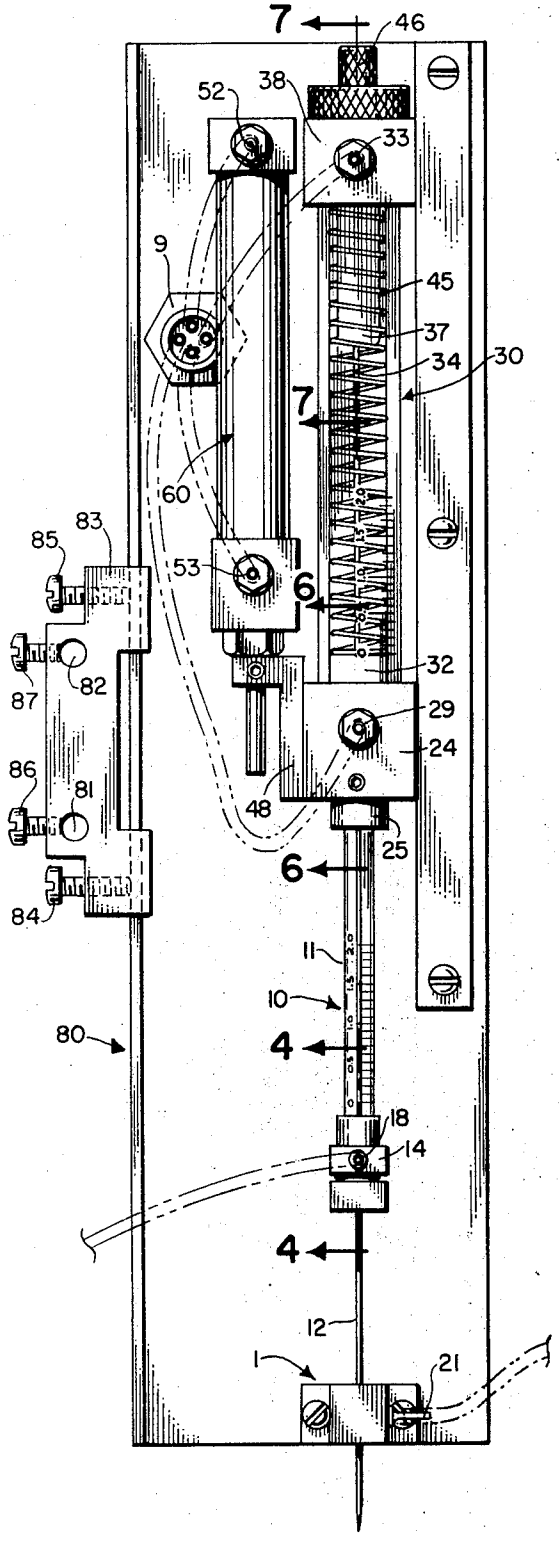

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FIG. 1.

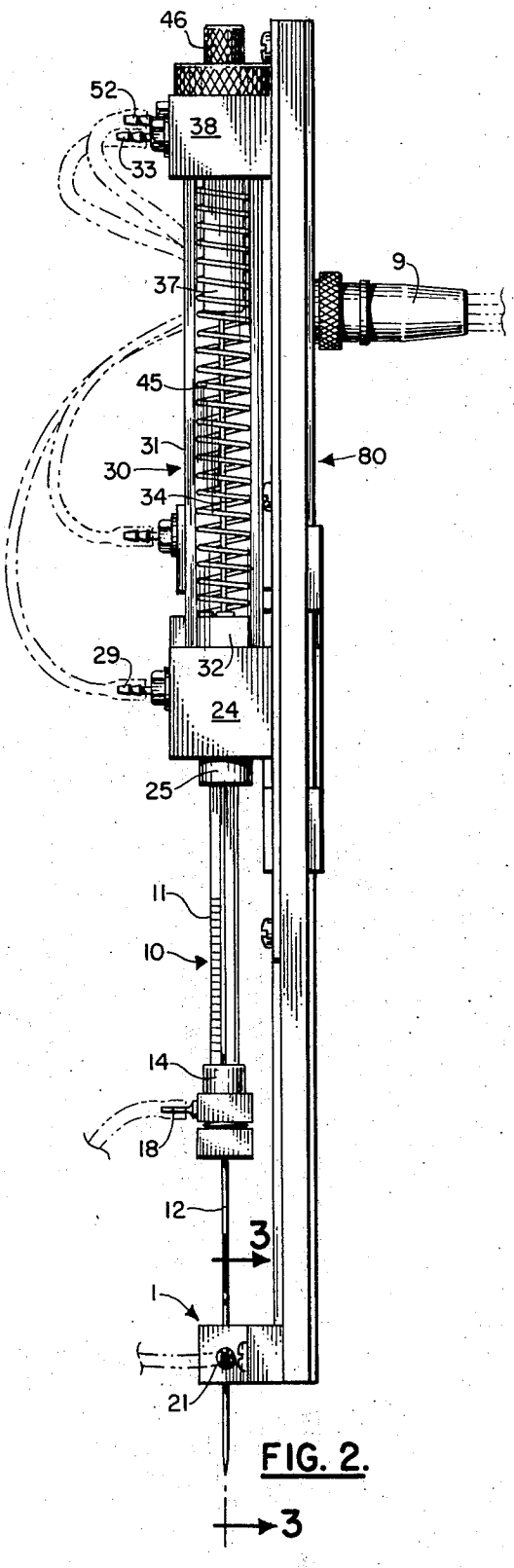

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FIG. 2.

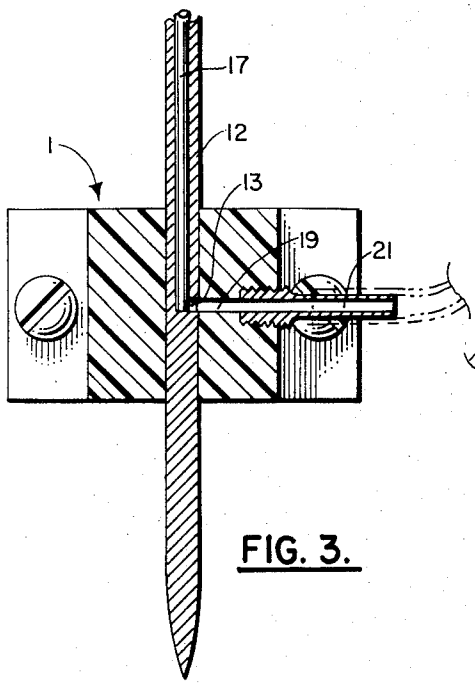


FIG. 3.

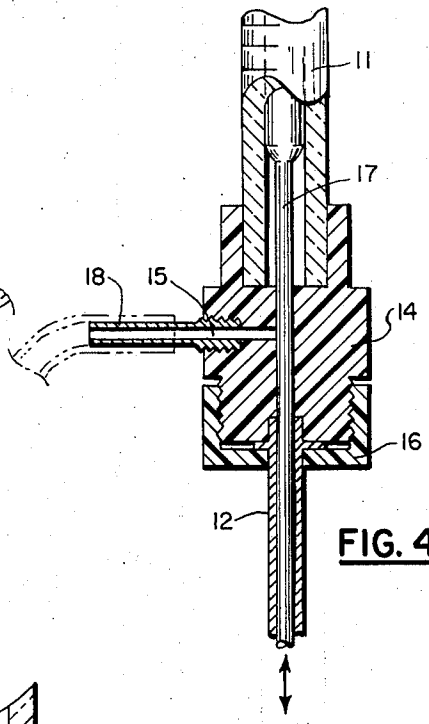


FIG. 4.

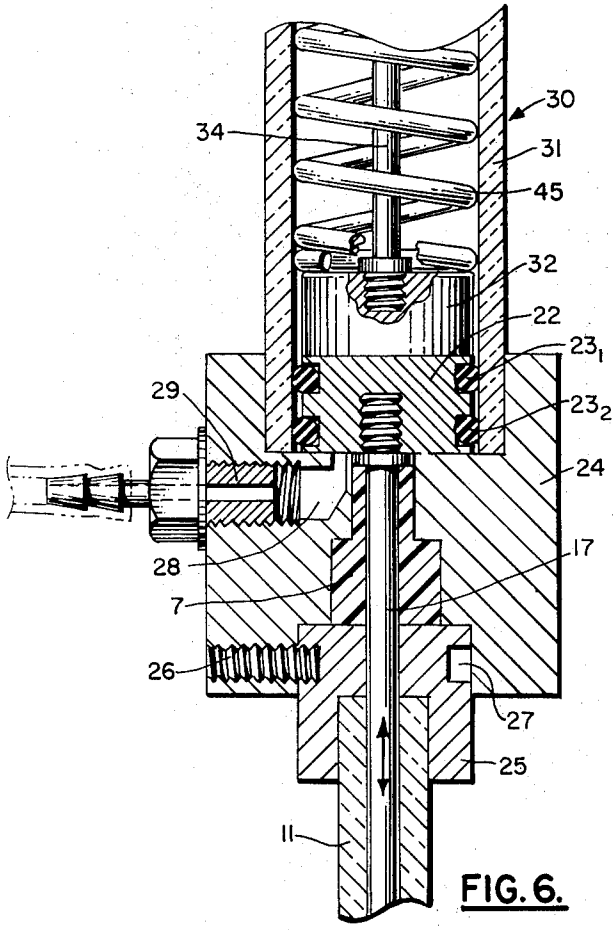


FIG. 6.

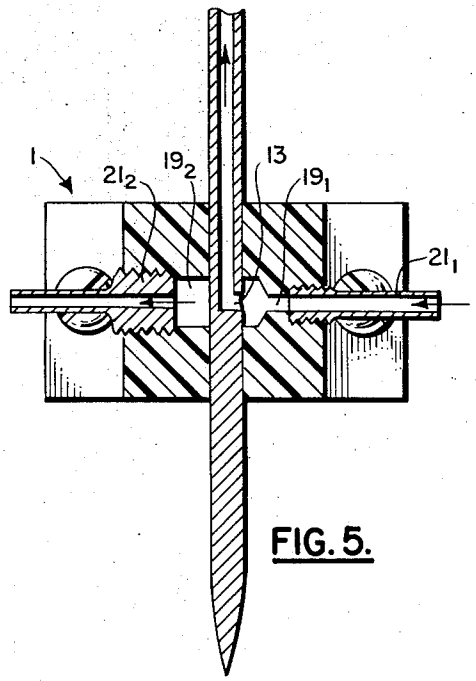


FIG. 5.

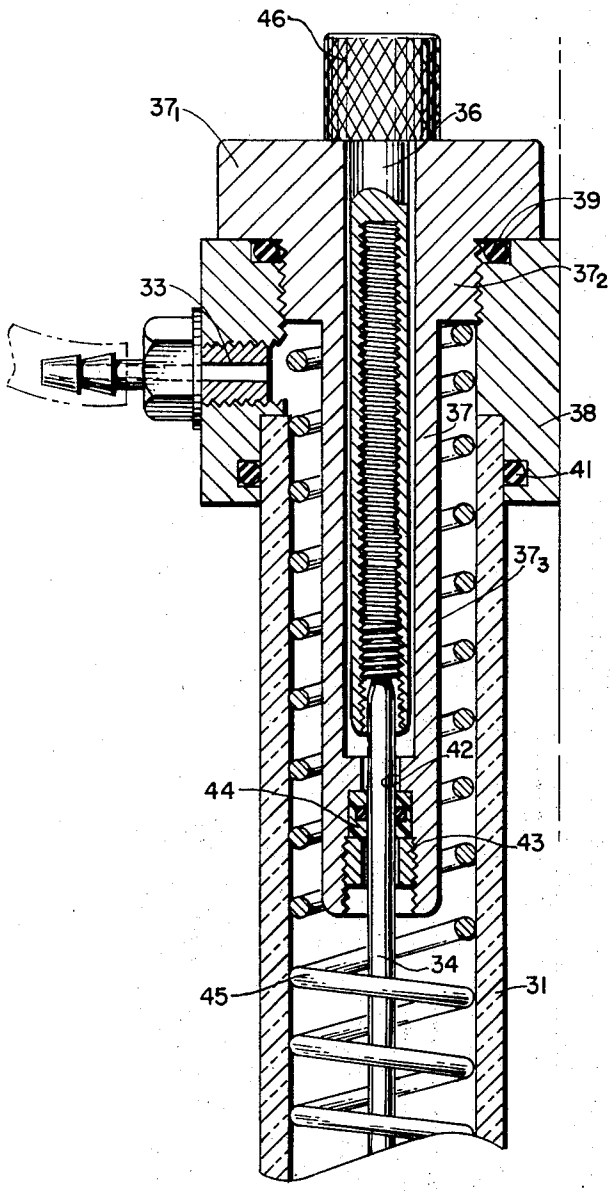


FIG. 7.

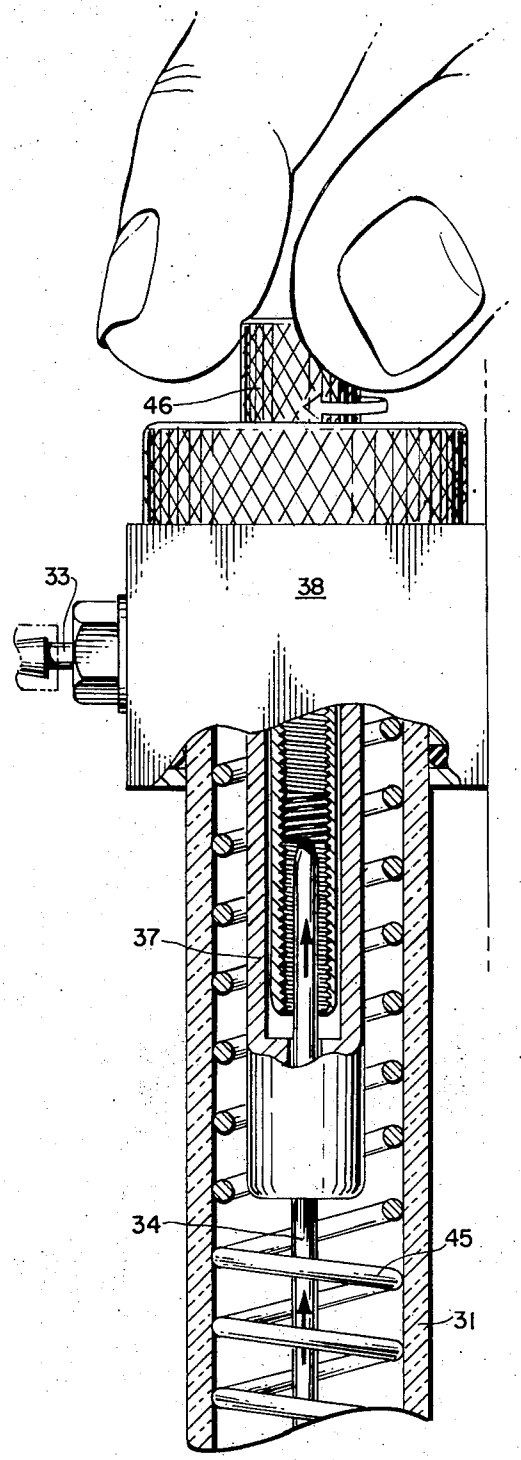


FIG. 8.

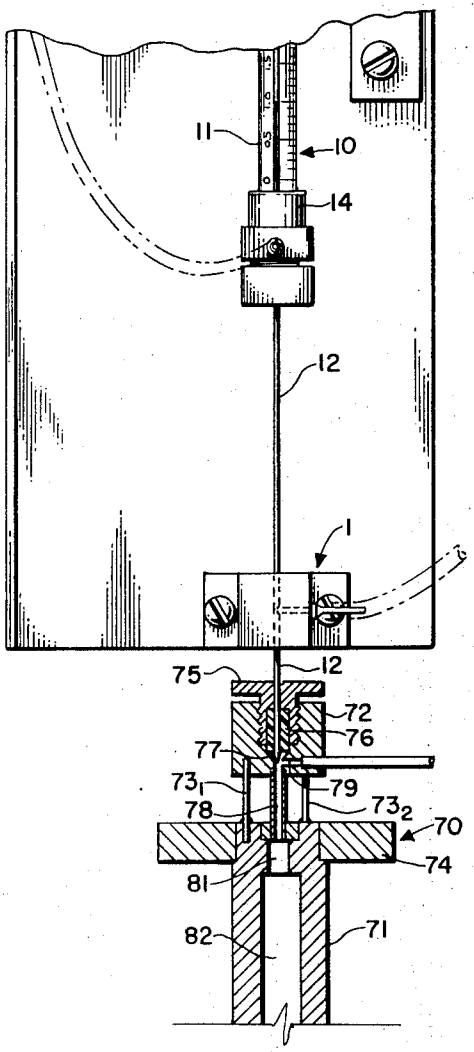

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FIG. 9.

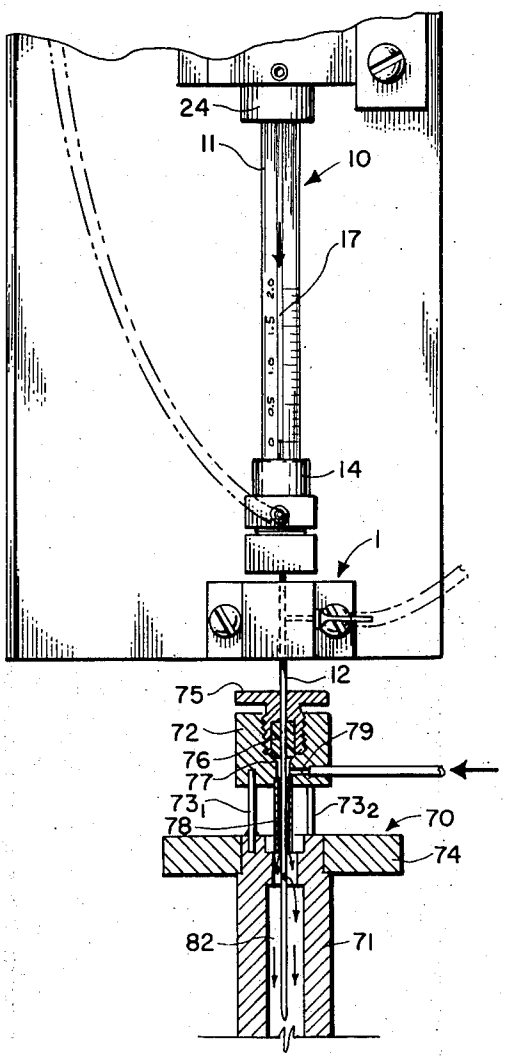

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FIG. 10.

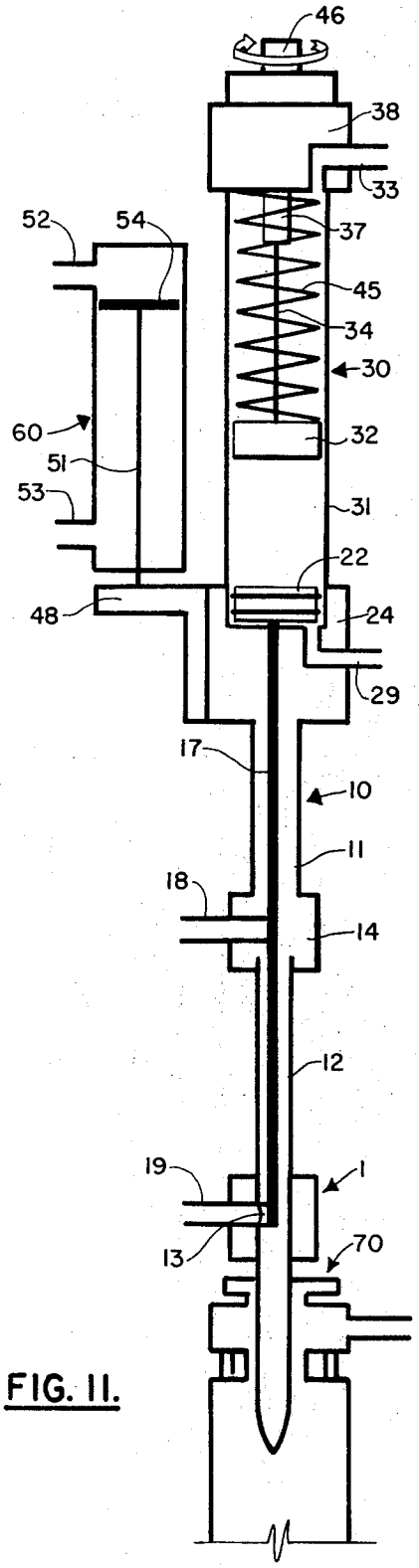

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FIG. 11.

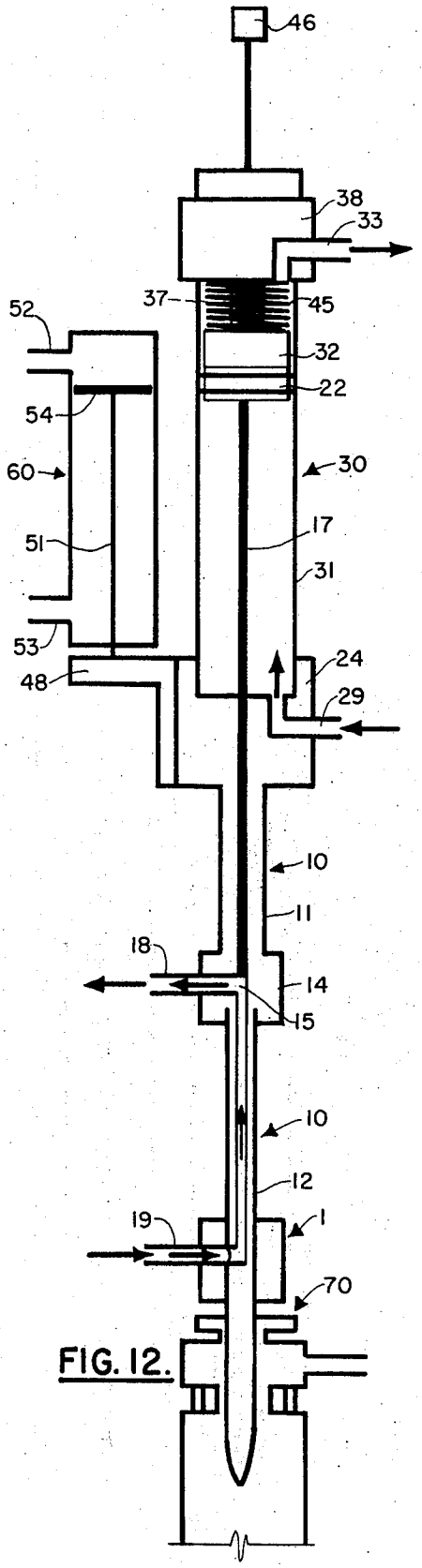

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FIG. 12.

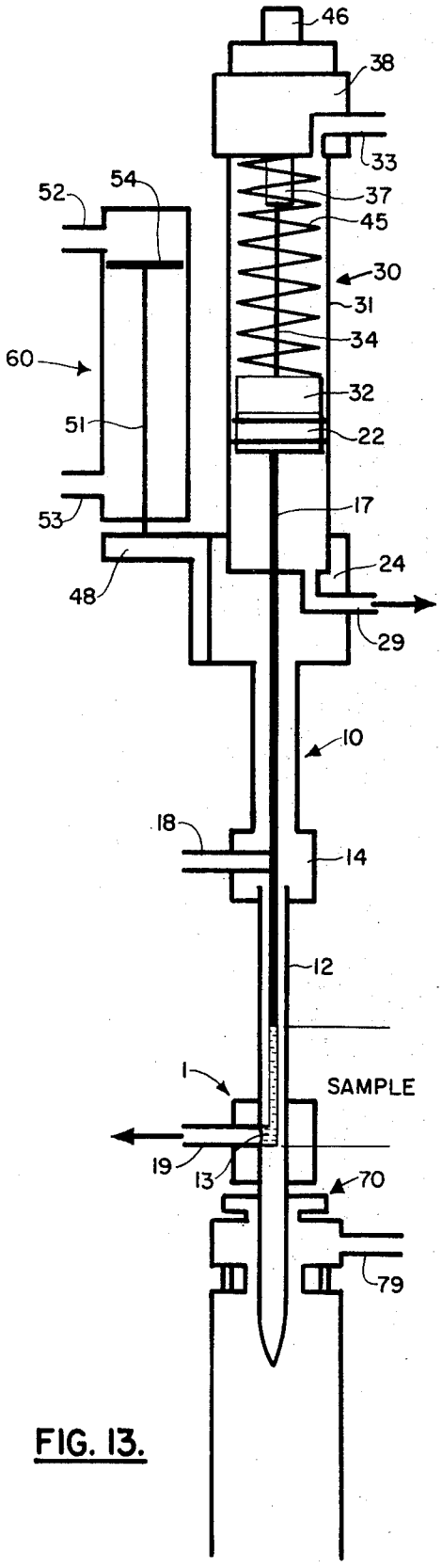

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FIG. 13.

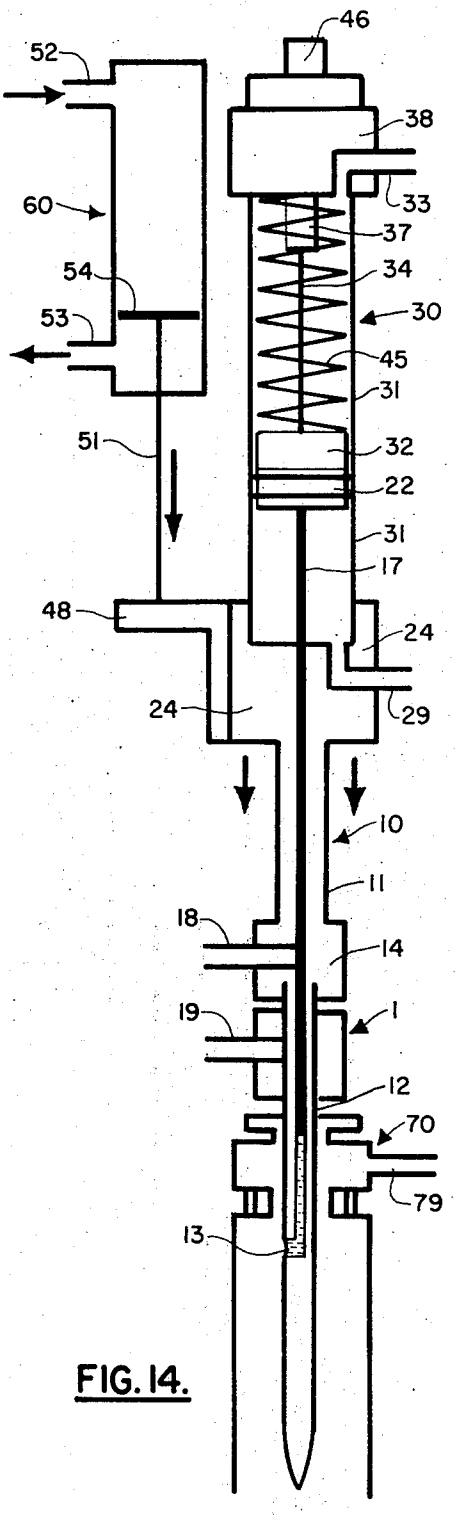

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FIG. 14.

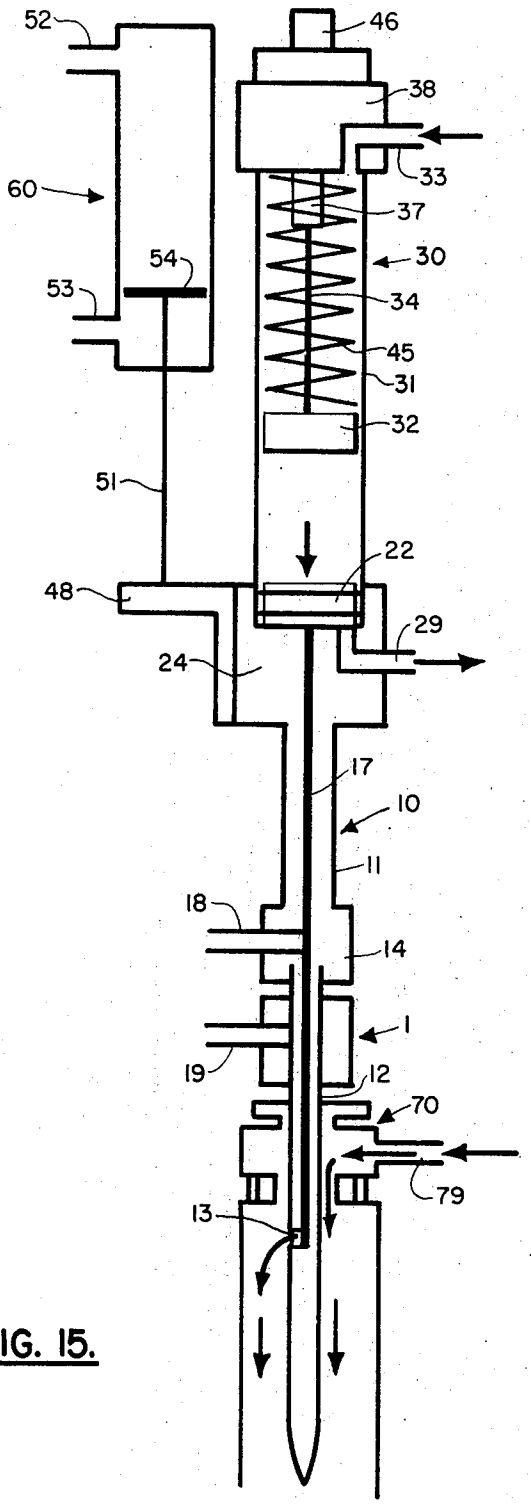

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FIG. 15.

AUTOMATIC FLUID INJECTOR

REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of related U.S. Pat. application Ser. No. 223,663, filed Feb. 4, 1972, now U.S. Pat. No. 3,754,443, the disclosure of which is herewith fully incorporated by way of reference.

The present invention relates generally to a continuous flow analyzer, or apparatus, for automatically measuring and injecting measured quantities of fluids. More particularly, it relates to apparatus, especially fluid injection devices or syringes, for continuous automatic measurement, and injection of very small, accurately measured quantities of liquid specimens into various media, e.g., modern analytical instruments.

Fluid injection devices, particularly needle syringes, have gained wide acceptance by the industry, and by the scientific community, generally, for use in dispensing infinitesimally small, accurately measured fluid specimens, e.g., to modern analytical instruments such as mass spectrometers and gas chromatographs. Such syringes embody apparatus comprising a tubular body or barrel, on the forward end of which is fitted a hollow or tubular needle and, at the opposite end, a slidable plunger which travels within the barrel. In the most accurate of existing syringes, the measured fluid is dispensed by the positive action of a very small diameter wire plunger, or portion of a plunger, which travels the length of the needle itself. Syringes of this character are capable of dispensing very small fluid specimens, accurately measured, on the order of very small fractions of a microliter.

Typically, micro syringes of such character are used by operators, chemists or other persons to inject liquid specimens on the order of only about 0.01 to about 5 microliters, or fractional parts thereof, into modern analytical instruments, e.g., mass spectrographs, gas chromatographs (GC) or the like. A GC, e.g., is provided with an entry port covered with a rubber membrane which is pierced by the dispensing end of the needle, whereupon a measured quantity of the contents of the bore of the needle or syringe is injected into the instrument.

In recent years, due to the obvious advantages offered by automatic fluid injection instruments, and modern data gathering techniques, which greatly reduce operating manpower without decrease in accuracy, there is considerable demand for improved devices of these types.

The continuous flow analyzer embodied by the present invention provides further improvements over prior art devices.

Among the objects of this invention are:

To provide a new and improved automated fluid injection device, or syringe, capable of more rapidly measuring out infinitesimally small, accurately measured fluid specimens, particularly fractional increments of liquid received from a source via a flowing stream.

To provide a continuous flow analyzer, or syringe, of simple structure which can be repetitively filled to a preselected setting, with precision and accuracy, and the fluid specimen then automatically injected with similar precision and accuracy.

These objects and others are achieved in accordance with the present invention which embodies improvements in automatic fluid injector systems. A preferred type of automatic fluid injector is comprised generally of (a) a support means, base, plate or housing located adjacent an inlet leading into an analytical instrument, (b) a tubular mounting member wherein is included a transverse opening leading into the axial opening through the tubular mounting member, generally or preferably located or affixed upon said support means such that the axial opening thereof can be aligned upon said inlet leading into the analytical instrument, (c) a needle syringe, provided with a reciprocable plunger slidable within the needle in response to actuating means, and the needle portion of which is projected and slidably mounted within the axial opening of the said axial opening through the said mounting member, and into the inlet, (d) which needle syringe is also reciprocably mounted upon the forward end of the piston of a cylinder piston unit such that actuation of the cylinder piston unit will reciprocate the piston and needle syringe associated therewith, and actuation of the plunger can be caused to accurately measure out, trap and inject fluid specimens into the inlet.

The needle syringe is comprised of a barrel formed by an enclosing wall defining an axial opening, within the forward end of which a needle is mounted, and within the opposite end of which is mounted a reciprocable plunger. The tip of the needle is sealed and provided with a forward side opening, and preferably (a) a rear opening forming a communication between the transverse opening of the tubular mounting member and the forward opening of the needle through the bore of the needle or (b) an enlarged annulus within the axial opening adjoining the transverse opening through the tubular mounting member, with a conduit therefrom providing a communication between the said transverse opening, forward needle opening and bore of the needle, and a by-pass. The forward end of the plunger is reciprocable and traversable from the forward opening of the needle rearward, and where a rearward opening is provided it is traversable in the rearward direction beyond the opening so that a communication is formed between the transverse opening of the mounting member, the forward side needle opening, the needle bore and the rearward opening. A fluid specimen from an external source to be sampled can thus flow continuously through the transverse opening of the mounting member, through the forward side needle opening, through the needle bore, and then through the rear opening (or in the reverse direction).

The plunger includes a small diameter forward portion and an enlarged diameter head portion, reciprocably mounted so that the small diameter portion is transversable within the needle bore, and the enlarged head portion is sealed off from the small diameter portion and contained within a barrel (or extension thereof) within which the plunger can be acted upon and reciprocated, preferably by actuating fluid as in a cylinder-piston unit. The needle syringe, inclusive of needle, barrel (and barrel extension) is, in turn, reciprocable upon the end of the piston of a cylinder piston unit and both the plunger and cylinder piston unit can be actuated in timed controlled response to automatic control means to form the functions of measuring, trapping, and injecting fluid specimens from a monitored stream from which analyses are desired. These several functions, the structural embodiments, and the best modes by which this can be accomplished are described in the

following detailed description, and in the appended drawings to which reference is made in the description.

The features which are considered as characteristic of the preferred automatic fluid injector, or continuous flow analyzer, and its principle of operation, will be more fully understood by reference to the following detailed description of a preferred embodiment, and to the enclosed drawings to which reference is made in the description. Similar numbers are used to represent similar parts or components in different figures, and subscripts are used, with a given whole number, to designate a plurality of similar parts or components. Where, thereafter, in the description a whole number is used without reference to its subscripts, its use is intended in generic sense, the number carrying a subscript representing a species.

In the drawings:

FIG. **1** is a perspective view of a preferred continuous flow analyzer or automatic fluid injector (or automated syringe) mounted on a base plate useful for positioning the said injector, or syringe, in relation to a septum inlet or location wherein an accurately measured fluid specimen is to be injected;

FIG. **2** depicts a side elevation view of the continuous flow analyzer, automatic fluid injector, or syringe, and base plate upon which the latter is mounted, as illustrated in the preceding figure;

FIG. **3** is a cross-section view taken along line **3—3** of FIG. **2**;

FIG. **4** is a cross-sectional view taken along line **4—4** of FIG. **1**, this figure with that of FIG. **3** showing the portion of the syringe within which a desired fluid specimen is measured, trapped and finally injected;

FIG. **5** is a preferred, and optional, construction of the forward portion of the injector, or syringe, as contrasted with the structure depicted in FIG. **3**;

FIG. **6** is a cross-section taken along line **6—6** of FIG. **1** showing the details of plunger head, and associated structure by means of which the plunger is actuated;

FIG. **7** is a cross-section taken along line **7—7** of FIG. **1** showing further details of the plunger actuating and plunger adjustment mechanism;

FIG. **8** depicts the structure and means for adjustment of the plunger stroke, or length of movement of the plunger, and consequently the precise amount of the fluid specimen that is selected for injection;

FIG. **9** is a fragmentary view of the forward portion of the automatic fluid injector, or syringe, including a portion of the base plate, showing the relationship between the said injector, or syringe, and a septum inlet, particularly a septum inlet of preferred type as depicted;

FIG. **10** is a view similar to that described by reference to FIG. **9**, but taken in connection with FIG. **9** shows the manner in which a trapped fluid specimen can be injected; and

FIGS. **11–15** depict a progressive series of schematic views of the automatic fluid injector, or syringe, showing the functions and operating sequence of sampling, trapping, measuring, and injecting an accurately measured volume of fluid into a preferred type of septum inlet.

Referring to FIGS. **1** and **2**, in general, there is shown a continuous flow analyzer which includes a fluid injector, or syringe **10**, attached or affixed upon the forward end of a plunger reciprocation and adjustment mechanism **30**, a primary purpose of which is to reciprocate and adjust, by preselected settings, the length of stroke of the reciprocable plunger associated with the syringe. Both the fluid injector, or syringe **10**, and plunger reciprocation and adjustment mechanism **30** are affixed to and carried upon the forward end of the piston of a double-acting cylinder piston unit **60**. The syringe **10** and plunger reciprocation and adjustment mechanism **30** move linearly, as a unit, with movement or reciprocation of the piston on actuation of the double-acting cylinder piston unit **60**, and the orientation of these members **10, 30** is maintained, relative to a base or support means comprising, e.g., the base plate **80**, by virtue of a tubular block, or mounting member **1**, within and through an opening of which the needle of the syringe **10** is slidably mounted (but movement thereof otherwise restricted) and by virtue of the fact that the double-acting cylinder piston unit **60** is itself affixed upon the base plate **80**. The plunger reciprocation and adjustment mechanism **30** and double-acting cylinder piston unit **60** carry out their normal operation and function in response to an automatic control and timing mechanism (not shown) which pulses a fluid (e.g., air or a hydraulic fluid, preferably the former) via flexible hoses (shown in phantom) which connect to double acting cylinder piston unit **60** via inlet and outlet connections **52, 53** and to the plunger reciprocation and adjustment mechanism **30** via connections **29, 33**, respectively to effect their actuation. The flexible hoses are passed through plate **80** via the tubular member **9**, and connected in place. Suitable automatic control and timing mechanisms for such use are known to the art and are sold commercially. A preferred type of automatic control and timing mechanism is illustrated and described in detail in U.S. Pat. application Ser. No. 223,663, supra. The continuous flow analyzer can be mounted in virtually any desired orientation with respect to a septum inlet, i.e., horizontally, vertically or diagonally, above, below or on either side of a septum inlet, and the device can be transported from one station to another (i.e., one septum inlet to another) and aligned therewith by movement of the base plate **80** itself as along parallel bars (not shown) conveniently passed through the openings **81, 82** of a bracket **83** on which the base plate **80** can be conveniently mounted. Thus, as shown by specific reference to FIG. **1**, the base plate **80** is provided with a bracket **83** fixed thereto as via bolts **84, 85**, and the entire assembly can be affixed in relation to a septum inlet by sliding the entire assembly along parallel bars or rods (not shown) which pass through openings **81, 82** of the bracket **83**, and its position can be fixed relative to a septum inlet, as desired, by tightening down the bolts **86, 87** upon the rods (not shown).

With reference to either of FIGS. **1** or **2**, the syringe is indicated generally by the numeral **10**. The syringe **10** includes generally a hollow barrel or tubular body **11**, provided by an enclosing wall which defines a central axial opening or bore. In its preferred form the barrel **11** is formed of glass or transparent plastic, most preferably the former, and outwardly scribed with indicia marks representative of the volume of the bore. A cannula or tubular needle **12** is tightly mounted, and sealed, preferably in a manner hereinafter described, within the forward end of the barrel **11**, the bore of the needle being aligned upon the axial opening or bore through the center of the barrel **11**. The forward end

of the needle **12** is sealed, or closed, and in effect is provided with two side openings, a forward inlet opening and a rearward outlet opening. These openings form a communication through the bore of the syringe through which a fluid specimen can be continuously passed and monitored by periodically trapping, measuring and injecting, at pre-determined intervals, a desired amount of the fluid into the septum of an analytical instrument for analysis. Referring to FIG. **3**, it will be observed that the needle **12** is thus provided with a forward side opening **13** which serves as a needle inlet. The rearward terminal end of the needle **12**, it will be observed, is tightly fitted and sealed within a resilient tubular member **14** to which the forward end of barrel **11** is also adjoined and sealed, and the said tubular member **14** is provided with a top or side outlet opening **15**. The resilient tubular member **14**, in effect, thus serves as an extension of the needle which contains an outlet opening **15** through which fluid can be passed from the rearward end of the needle **12**. A capping member **16**, preferably of metal, is provided to protect the forward end of the resilient tubular member **14**.

The forward end of needle **12**, as observed by reference to FIG. **3**, is permanently affixed, but slidably movable, in a linear direction, within the block or mounting member **1**, and the tip thereof is projected beyond and outside the mounting member **1** for penetration of a septum, or for mounting within a preferred type of septum inlet such as described hereafter by reference to FIGS. **9** and **10**. A plunger **17**, reciprocably movable through the bore of the needle **12**, is provided for movement from a location behind the inlet opening **15**, communicated with the rearward end of the needle **12**, to the forward end of the bore of the needle **12** where needle inlet opening **13** is located. Thus, it will be observed that the inlet **13**, with the bore of the needle **12**, can form a communication through which fluid can be flowed. Fluid entering, via a hose connection through a nozzle **21** and flowing into inlet opening **19**, will pass rearwardly through the bore of needle **12** (as can occur when the plunger **17** is retracted beyond the outlet opening **15**). The fluid will exit from the bore via the outlet opening **15** (when the needle is positioned as illustrated in FIGS. **3** and **4** except that the plunger **17** is withdrawn) and nozzle **18** from which it will be conveyed via the hose connection. (It is also apparent that the direction of fluid flow can be reversed through this communication without effect on the principle of operation.)

In the operation of that portion of the continuous flow analyzer depicted by reference to FIGS. **3** and **4**, it will be readily observed that several functions can be performed by the regulation and controlled movement of plunger **17**. For example, when the plunger **17** is retracted so that its tip is rearward of the outlet opening **15** a fluid specimen from a monitored stream can be continuously flowed through the communication formed by nozzle **21**, inlet opening **19** of mounting member **1**, the needle opening **13**, the bore of needle **12**, the axial opening through the resilient tubular member **14**, and outlet opening **15**; and the fluid specimen will exit via nozzle **18** when the needle **12** is positioned as in said Example **3**. It will also be apparent that forward movement of plunger **17** to a position which blocks outlet opening **15** will cut off the flow of fluid. Adjustment of the distance between the tip of the plunger **17** and the forward inlet needle opening **13** can be made to measure out a desired volume of fluid specimen for subsequent injection. This is accomplished by causing the tip of plunger **17** to move forward past outlet opening **15** for a preselected distance to eject all but an accurately measured portion of fluid specimen located between the tip of plunger **17** and needle inlet opening **13**. Forward movement of needle **12** itself, as opposed to further forward movement of plunger **17**, will then move needle inlet opening **13** out of alignment with inlet opening **19** to trap or contain the accurately measured fluid specimen for subsequent injection.

A more preferred type of forward inlet is depicted by reference to FIG. **5**. The structure is precisely the same as shown by reference to FIG. **3** except that an inlet opening 19_1 is in effect cut or projected all the way through the mounting member **1**, and the central portion thereof is enlarged to form a chamber 19_2, provided with an outlet 21_2, so that fluid from a monitored stream continuously flows therethrough regardless of the particular operation being performed by the continuous flow analyzer. Thus, fluid continuously flows via nozzle 21_1 into inlet opening 19_1 and into the chamber 19_2. Where the needle inlet opening **13** is positioned within enlarged chamber 19_2, and plunger **17** is retracted, as shown by reference to FIG. **5**, a portion of the fluid stream flows into opening **13** and the bore of needle **12**, while another portion of fluid flows outside the system via the axial opening through nozzle 21_2. When the needle inlet opening **13** is blocked by the tip of plunger **17**, or the said inlet opening **13** is moved outside the chamber 19_2 , then the entire quantity of fluid exits from chamber 19_2 via the axial opening through nozzle 21_2. When, on the other hand, it is desired to withdraw an accurately measured amount of fluid specimen it is only necessary to withdraw plunger **17** a calibrated amount whereupon fluid will flow via needle inlet **13** into the base of needle **12** and fill up to the point where it is blocked by the tip of plunger **17**. One advantage of this feature is that the outlet **15** can be eliminated, if desired.

The details of the plunger reciprocation and adjustment mechanism **30**, and its function, are best described by specific reference to FIGS. **4** and **6** through **8**. Referring first to FIGS. **4** and **6**, plunger **17** is thus comprised of an upper portion of enlarged diameter, which for added strength, conforms generally with the inside diameter of the axial opening through barrel **11**, while the lower portion thereof is of greatly reduced diameter, conforming generally with the inside diameter of the bore through needle **12**. The smaller diameter, or wire portion, of plunger **17** is generally secured to the larger diameter portion of plunger of plunger **17** via threadable engagement, the smaller diameter member fitting within the larger diameter member. The wire portion of plunger **17** traverses the full length of the bore through needle **12**, the tip thereof at its maximum forward position flushing with and closing needle inlet **13** (FIG. **3**). The smaller diameter wire portion of plunger **17** at the position of its maximum rearward movement is sufficient that the tip thereof is clear of and cannot block outlet opening **15**. The rearward, or larger diameter, terminal end of plunger **17** (FIG. **6**) is threadably engaged, via a center tap, with an enlarged diameter cylindrical shaped head **22**, provided with one or more circumferential grooves (not numbered) within which O-rings 23_1, 23_2 are fitted to provide an air-tight seal between the inside wall of tubular member

31 of plunger reciprocation and adjustment mechanism 30, which is, in effect, with tubular member 24 an extension of the barrel 11. Thus, tubular member or barrel extension 31 is adjoined to barrel 11 through a block or tubular assembly 24, barrel extension 31 being snugly fitted into one side of tubular assembly 24 while barrel 11 is snugly fitted into a cylindrical-shaped gland 25 held snugly in place within the opposite side of tubular assembly 24 by a bolt 26 which is extended from the outside surface of assembly 24 into a circumferential slot 27 of gland 25 to hold the latter securely in place.

In one of its essential aspects, the portion of the plunger reciprocation and adjustment mechanism 30, it is stressed, acts as a double-acting cylinder-piston unit for reciprocation of piston 17. An opening 28 (FIG. 6) into which is fitted a nozzle 29 through which a driving fluid such as air or liquid can be introduced via a suitable hose connection, leads into the confines of barrel extension 31, this constituting one side of the cylinder-piston drive unit. A tubular shaped seal 7 comprised of a resilient material such as natural or synthetic rubber, plastic or plastic-like material, e.g., Teflon, through which the plunger 17 is passed forms a barrier and effectively seals and prevents escape of the driving fluid into barrel 11. In FIG. 6, it will be noted that the head 22 of plunger 17 is located entirely within barrel extension 31 and, but for the physical presence of the stop 32 and associated structure, that pressure exerted on head 22 of plunger 17 would drive plunger 17 to the rear. By specific reference to FIG. 7 it will also be apparent that injection of a driving fluid into the opposite end, and within the confines of barrel extension 31 via nozzle inlet 33 will permit a driving fluid to impinge on the opposite side of head 22 of plunger 17 and hence, but for the presence of stop 32 and associated structure, the piston 17 can readily be reciprocated in the same manner as any other conventional double-acting cylinder piston unit. The stop 32 and associated structure, however, serves a profoundly important function which is to limit the length or distance of movement of plunger 17 to the extent desired and thus permit pre-selection of the amount of the fluid specimen to be injected as well as to permit and facilitate ready change of the amount thereof for injection, as desired.

The plunger adjustment assembly of the plunger reciprocation and adjustment mechanism 30, again referring specifically to FIGS. 6 and 7, are comprised of a plunger stop 32, of cylinder shape and of diameter somewhat less than the inside diameter of barrel extension 31, the member being threadably engaged via a central tap upon the forward terminal end of a shaft 34, and the shaft 34, in turn, being threadably engaged at its opposite terminal end to an internally threaded tubular member 36 which is rotatably fitted within an outer larger diameter tubular member 37. It will be observed that the larger tubular member 37 makes up with and is extended into the rearward end of barrel extension 31 by virtue of a sleeve 38 which fits over and is adjoined to the rearward end of barrel extension 31. The tubular member 37 includes a flange portion 37_1, a smaller diameter externally threaded shank portion 37_2 against the face of which an end of coil spring 45 is seated, and an elongate tubular portion 37_3, the central axis of which is coaxially aligned with the central axis of the barrel extension 31. The said tubular member 37 is affixed in place via mating engagement between externally threaded shank portion 37_2 thereof, and the internally threaded sleeve 38. O-ring seals 39, 41 located between the tubular member 37 and sleeve 38, and between sleeve 38 and the rearward end of barrel extension 31, respectively, provide an air-tight fitting. It will be observed that the inside of tubular member 37 is also provided with a cross-section of reduced diameter 42 which, with externally threaded collar 43 engaged in the internally threaded forward end of tubular portion 37_3 of tubular member 37, maintains better alignment and stability of shaft 34, provides a seating surface within which is contained a tubular seal 44, and further provides a surface for limiting the forward displacement of shaft 34. The coil spring 45, of external diameter approximating the inside diameter of barrel extension 31, is fitted concentrically about the shaft 34 and the forward portion of tubular member 37, which spring 45 is seated between the rearward face of the plunger head 22 of piston 17 and the face of shank 37_2 at the rearward end of barrel extension 31. It will be noted that a knurled cylindrical shaped knob 46 is integrally located on the rearward end of tubular member 36, the diameter thereof being larger than the central opening through tubular member 37 so that it cannot enter therein. Rotation of the knob 46 in clockwise direction, it will be noted, will cause the shaft 34 to move rearwardly, this producing withdrawal of plunger stop 32 and compression of coil spring 45. Conversely, rotation of knob 46 in a counter-clockwise direction will cause the shaft 34 to move forwardly, or move in the opposite direction, until such time that the head 22 of plunger 17 is moved to its furthermost forward position as is indicated by specific reference to FIG. 6.

In the drawings depicted by FIGS. 1 through 4, and 6 and 7, the plunger stop 32 is shown in its maximum forward position (FIG. 6), in which position also the head 22 of plunger 17 is in its extreme forward position and the forward tip of plunger 17 flushes with and closes needle inlet port 13 (FIG. 3). The instrument is calibrated by clockwise rotation of knurled knob 46 (FIG. 8), and tubular member 36 which is threadably engaged with shaft 34, such that the latter is retracted or moved toward the rear, this in turn moving plunger stop 32 rearwardly from the position shown by reference to FIG. 6. The distance over which the plunger stop 32 is moved will permit a corresponding and equidistant rearward movement of plunger 17 (or the tip of plunger 17 within the bore away from the needle inlet port 13). Hence, the volume of fluid specimen desired for injection can be readily preselected by the distance of such setting from the zero mark. For example, by reference to any of FIGS. 1 through 4 and 6 and 7, the plunger stop 32 is "zeroed," or set at zero, at which setting no fluid specimen would be ejected by operation of the instrument. To set the instrument for injection of a predetermined volume of a fluid specimen, therefore, e.g., one (1.0) microliter, knob 46 is rotated in clockwise direction until the plunger stop 32 has been retracted, or moved, to the rear sufficiently that the reading shows that the plunger 17 has moved a distance which will correspond to a 1.0 microliter setting. This is readily determined by reference to indicia marks which can be appropriately scribed on the forward end of barrel 11 (FIG. 9) or on the forward end of barrel extension 31 (FIG. 1), or both. Thus, e.g., starting with the location of transition wherein the plunger 17

changes from relatively small diameter to large diameter (FIG. 4), which corresponds to a zero setting knob 46 is rotated clockwise until such time that this transition location of the plunger 17 is moved to a setting of 1.0 microliter. Calibration of the instrument in this manner also corresponds with the distance the forward face of plunger stop 32 can be moved away from the rearward face of head 22 of piston 17, which in effect sets the distance the tip of plunger 17 can be moved from a position which flushes with needle inlet port 13 to the location wherein the rear face of the head 22 of plunger 17 impinges against the front face of plunger stop 32.

An especially preferred type of septum inlet 70 for use in a modern analytical instrument, e.g., a mass spectrograph, gas chromatograph, or the like, and with the continuous flow analyzer described herein, is illustrated by specific reference to FIGS. 9 and 10. The septum inlet 70 is comprised of an inner tubular member 71 and an outer tubular section 72, the inner and outer tubular members 71, 72 being separated one from the other by a plurality of spacers or support rods 73_1, 73_2, 73_3 (not shown). The spacers 73, by offering a limited cross-sectional area between tubular members 71, 72, aid in suppressing the transfer of heat from tubular member 71 and tubular member 72. The inner tubular member 71 is optionally provided with a flange 74. It will be observed that the side of the outer tubular member 72, facing the continuous flow analyzer, is provided with an internally threaded center top, and therein is fitted, via threadable engagement therewith, a cap 75 provided with a centrally located axial opening therethrough. The forward face of the externally threaded shank portion of cap 75 is deeply recessed and a tubular shape seal 76, of resilient material such as natural or synthetic rubber, plastic or plastic-like material (e.g., Teflon), is fitted therein. The forward end of needle 12, it will be observed, is projected through the axial opening through cap 75 and seal 76, and hence by tightening down the cap 75 the material constituting the seal will be thrust inwardly by extrusion into the frusto conic entry portion 77 and tightening against the outer wall of needle 12 to form an effective leak-proof seal. The needle 12, an operation of the continuous flow analyzer, is movable reciprocably through the axial opening formed by the openings through the cap 75, the tubular seal 76 and the central opening through tubular member 78 located between outer and inner members 71, 72. On deep introduction of the needle 12, as shown by reference to FIG. 10, by forward movement of syringe 12, and on forward movement of plunger 17, a fluid specimen is injected and swept by carrier gas introduced into the septum inlet 70 via line 79, the fluid specimen being carried into the relatively narrow opening 81, thence into the larger opening 82, and into the analytical instrument for analysis. By comparison of the readings shown by the indicia markings on the barrel 11 of syringe 10 (FIGS. 9 and 10) it will be observed, e.g., that 1.0 microliter of fluid specimen is delivered into septum inlet 70, and dispersed therein and into an analytical instrument (not shown) by carrier gas.

The operation and function of the continuous flow analyzer of this invention will be further illustrated by specific reference to FIGS. 11 through 15 which depict, in schematic fashion, a progressive series of views describing a single cycle which includes sampling, trapping, measuring and injecting an accurately measured volume of a fluid specimen into a preferred type of septum inlet. FIG. 11 depicts the continuous flow analyzer as the instrument would be positioned for use in monitoring a stream of the flowing fluid specimen.

In FIG. 11 the syringe 10 and reciprocation and adjustment mechanism 30 are maintained in their maximum rearward position, being so positioned by the location of piston 51 of double-acting cylinder piston unit 60. Thus, it will be observed that syringe 10 and reciprocation and adjustment mechanism 30 are mounted via the mounting bracket 48 upon the forward terminal end of piston 51 and reciprocable therewith. The tip of plunger 17 is flush with the forward port or opening 13 of needle 12, plunger 17 being in its maximum forward position. Fluid from the flowing stream to be sampled cannot enter into needle 12 via line 19 because of the blocking presence of the tip of plunger 17. In order to pre-set the instrument to measure out and inject the desired amount of fluid specimen for injection into septum inlet 70, knob 46 is rotated clockwise and plunger stop 32 of the plunger reciprocation and adjustment 30 is retracted. The distance of retraction of plunger stop 32, i.e., the distance between the rear face of the head 22 of plunger 17 and the forward face of plunger stop 32, is directly related to the volume of fluid specimen which will be measured out, trapped and injected.

Referring to FIG. 12, air is injected via line 29 into barrel extension 31 of the plunger reciprocation and adjustment mechanism 30, impinging upon head 22 of piston 17 driving piston 17 to its extreme rearward position, plunger head 22 pressing against plunger stop 32, compressing helical spring 45. The forward end of tubular member 37 acts as a stop which arrests further rearward movement of plunger stop 32. Simultaneously, with the rearward movement of plunger 17, air is expelled or vented via line 33. The tip of plunger 17 moves rearwardly uncovering needle opening 13, and then opening 15 on the rearward side of needle 12, this permitting ingress of a fluid specimen from a source via inlet line 19, through the bore of needle 12 and egress of fluid from the bore via opening 15 and outlet 18. Thus, a loop through which a fluid specimen can be flowed is formed by line 19, opening 13, the bore of needle 12, opening 15, and line 18. This flow is generally continued for a sufficient length of time to purge the bore of needle 12 of any possible contamination from previous sampled specimens, to eliminate any possible presence of bubbles, and to obtain a representative sample. A plurality of reciprocations of plunger 17 can be made, if desired.

Referring to FIG. 13, pressure is now released from the forward end of barrel extension 31 via release of air from line 29. The plunger stop 32, as the pressure is released and the balancing force applied upon helical spring 45 becomes unbalanced, moves forward pushing against head 22 of piston 17 carrying the plunger 17 forward closing outlet 15 and interrupting the egress of fluid from outlet 18. Fluid is expelled from needle opening 13, the fluid exiting via line 19 as the plunger 17 moves forward. On release of the tension on spring 45, the plunger 17 discontinues its forward movement. The egress of fluid from needle opening 13, and line 19, ceases leaving a portion of fluid specimen, in measured amount, contained within the bore of needle 12. The fluid volume of the contained specimen indicated as "sample" in the figure, as heretofore suggested, is pre-

determined by the setting described by reference to FIG. **11.** The distance heretofore referred to, i.e., distance between the forward face of plunger stop **32** and rear face of plunger head **22,** is equal to the distance between the forward face of plunger head **22** and the forward end of barrel extension **31,** which in turn determines the volume of the bore of needle **12** within which fluid specimen is contained.

Referring now to FIG. **14,** double-acting cylinder piston unit **60** is next actuated by injection of pressurized air into nozzle **52,** the air impinging upon piston head **54** driving piston **51** forward, carrying with it plunger reciprocation and adjustment mechanism **30** and syringe **10.** The needle **12** is pressed forward through mounting frame **1,** wiping contact shearing away any excess of fluid at the needle opening **13.** This action traps the accurately measured fluid specimen within the bore of needle **12** forward of the tip of plunger **17.** As will be observed, the needle **12** is at this time thrust deeply into septum inlet **70.**

Referring to FIG. **15,** air is injected via line **33** into the rearward side of barrel extension **31,** the air passing around the annulus of plunger stop **32** and impinging upon the rearward face of plunger head **22** of plunger **17.** During the forward movement of the plunger head **22,** air is simultaneously expelled from the forward end of barrel extension **31** via line **29.** Plunger **17** is thus driven forward through the bore of needle **12,** causing ejection of the fluid specimen contained therein, the fluid being expelled through the needle opening **13** at which time it is picked up by carrier gas which enters the septum inlet **70** via line **74** and carried into the analytical instrument (not shown). The cycle is repeated ad infinitum.

It is apparent that various modifications and changes can be made without departing the spirit and scope of the present invention. The apparatus is constructed of materials substantially inert or nonreactive to the chemical or corrosive action of the fluid specimens to be measured and dispensed. The barrel is normally constructed of glass, but can be constructed of a plastic or plastic-like material. The barrel extension can be similarly constructed, or constructed of metal. The seals are normally constructed of plastic, and the rest of the syringe of various metals.

The seals are preferably formed of a rigid or semi-rigid, resilient form of plastic or plastic-like material. The self-lubricated plastics are especially preferred in this capacity, and can also be applied as a laminate or protective film. The polyfluorinated ethylene polymers, notable among which is polytetrafluoroethylene (Teflon), are particularly outstanding. Conventional resilient or elastic-like materials, such as natural or synthetic rubbers, can also be employed.

The plunger stop assembly and the needle of the syringe are preferably constructed of metals, e.g., ferrous metals such as iron, iron alloys, steel, stainless steels, and the like; or such metals as aluminum, magnesium, brass, copper, bronze, chrome, alloys of these and other metals, and the like.

It is apparent that various changes, such as in the absolute or relative dimensions of the parts, materials used, and the like, as well as the suggested mode of withdrawing or delivering fluids, can be made without departing the spirit and scope of the invention, as will be apparent to those skilled in this art.

Having described the invention, what is claimed is:

1. In apparatus for use in repetitively dispensing small, pre-selected accurately measured quantities of fluid specimens into a media such as an inlet to an analytical instrument, the combination comprising

a support means, which can be located adjacent an inlet leading into the analytical instrument,

a tubular mounting member, an enclosing wall thereof defining an axial opening which can be aligned upon the inlet to the analytical instrument, and which axial opening is intersected by a transverse second opening located within the said enclosing wall, said tubular mounting member being supported upon said support means,

a needle syringe wherein is included,

a barrel formed by an enclosing wall defining an axial opening,

a tubular needle mounted with the front end of the barrel, said tubular needle being provided with a sealed tip, a forward side opening leading into the bore of the needle,

a plunger, including a small diameter forward portion and an enlarged head portion at its rearward end, reciprocably mounted such that the small diameter portion is traversable within the needle bore from the side opening at the front of the needle rearwardly, while the head portion thereof is located within the barrel, and reciprocable and traversable therein

a seal located rearward of the tubular needle and within the barrel, through which the plunger is passed and is reciprocably movable,

a plunger adjustment and stop means located at the rear of the barrel, wherein is included an outer tubular member and an internally threaded tubular member rotatably mounted therein, a shaft provided with a stop member at its forward end, and external threads located on its rearward end, which end is projected into and threadably engaged with the threads of the internally threaded tubular member, a spring seated between the stop member and the rearward end of the barrel, such that rotation of the internally threaded tubular member in either direction will cause the shaft to telescope inwardly or outwardly within the inner tubular member and thereby adjust, by lengthening or shortening, the distance between the stop member of the shaft and the head of the plunger, to permit pre-selection and accurate measurement of the desired amount of a fluid specimen to be subsequently injected,

openings located within the forward and rearward ends of the barrel on opposite sides of the plunger head through which a pressurized fluid can be injected, and subsequently released, for driving and reciprocating the plunger,

a cylinder piston unit supported on said support means, provided with means for actuation of a piston rod on the end of which the needle syringe is mounted and reciprocable therewith,

whereby, when the plunger is retracted and side opening of the needle is in alignment with the second transverse opening of the tubular mounting member a fluid specimen can be flowed through the said second transverse opening of the tubular mounting member, into the forward side opening of the needle and into the bore of the needle in preselected accurately measured quantity, the cylin-

der piston unit then activated such that the piston thereof is moved forward to thrust the needle deeply into the inlet, close off the said second transverse opening of the tubular mounting member, and trap the fluid specimen within the bore of the needle as the needle is thrust through the axial opening through said mounting member, after which time the plunger is thrust forward on actuation of the cylinder piston unit to expel the fluid specimen into the sample inlet.

2. The apparatus of claim **1** wherein the needle is also provided with a rearward opening entering therein, the rearward opening being located between the forward needle opening and the rearward face of the seal.

3. The apparatus of claim **2** wherein the rearward opening is located within the seal.

4. The apparatus of claim **1** wherein the transverse second opening located within the tubular mounting member extends through both sides of the wall into the axial opening thereof into which the needle is projected, and a portion of the axial opening thereof through which the needle is projected is enlarged to permit a portion of fluid to flow from one side of the transverse opening into the forward side opening of the needle when the plunger is retracted and the forward side opening of the needle is aligned therewith, while another portion of fluid flows around the needle and egresses through the said transverse opening on the opposite side of the axial opening.

5. In apparatus for use in repetitively dispensing small, preselected accurately measured quantities of fluid specimens into a media such as an inlet to an analytical instrument, the combination comprising

a support plate, which can be located adjacent an inlet leading into the analytical instrument,

a tubular mounting member, an enclosing wall thereof defining an axial opening which can be aligned upon the inlet to the analytical instrument, and which axial opening is intersected by a transverse second opening located within the said enclosing wall, said tubular mounting member being supported upon said support plate such that the axial opening thereof is in alignment with the septum inlet,

a needle syringe wherein is included

a barrel formed by an enclosing wall defining an axial opening,

a tubular needle mounted within the forward end of the barrel, said tubular needle having a sealed tip, a forward side opening leading into the bore of the needle, and a rearward opening leading into the rearward end of the needle, said pair of openings forming, with the bore of the needle, a continuous loop communication through which a fluid specimen can ingress into and egress from the bore of the needle,

a plunger, including a small diameter forward wire portion and a large diameter head portion at its rearward end, reciprocably mounted such that the wire portion of the plunger is traversable within the needle bore from the forward side opening of the needle to a location rearwardly of the opening leading into the rearward end of the needle,

a tubular section, formed by an enclosing wall defining an axial opening, within the front side of which the barrel is adjoined and mounted, and on the opposite side of which a barrel extension of diameter larger than that of the barrel is adjoined and mounted, said tubular section containing a tubular seal constructed of resilient material within the axial opening therethrough, and through the axial opening of which the plunger is mounted, a small diameter portion of the plunger being snugly fitted through the opening through the seal whereas the enlarged diameter head portion thereof is permanently mounted within the barrel extension, the plunger being reciprocably movable,

a plunger adjustment and stop means located at the rear of the barrel extension, wherein is included an outer tubular member adjoined upon and closing the rearward end of the barrel extension, its forward small diameter end protruding into the confines of the larger diameter barrel extension, an internally threaded smaller diameter tubular member rotatably mounted within the outer tubular member and extending forwardly into the smaller diameter portion of the outer tubular member, a shaft provided with a cylindrical shaped stop member, of smaller diameter than that of the barrel extension, mounted on the forward end thereof, the rearward end of said shaft being externally threaded, projected within and engaged with the internal threads of the said internally threaded tubular member, a helical spring concentric with the outer tubular member and seated between the cylindrical head of the stop member and the closed rearward end of the barrel extension, such that rotation of the internally threaded tubular member in one direction will cause the shaft to telescope inwardly and put tension on the coil spring, or outwardly within the inner tubular member to release the tension on the spring and thereby adjust, by shortening or lengthening, the distance between the forward face of the cylindrical shaped stop member and the rearward face of the head of the plunger, so as to permit preselection and calibration of the amount of a fluid specimen to be subsequently injected,

an opening within the tubular section located between the barrel and barrel extension leading into the barrel extension, through which a pressurized fluid can be injected to impinge upon the head of the plunger to effect its retraction, and through which fluid can be exhausted to release tension on the coil spring and permit forward movement of the plunger,

an opening within the rear of the tubular section through which a pressurized fluid can be injected to impinge upon the head of the plunger to effect its forward movement, and through which fluid can be exhausted on rearward movement of the plunger,

a double-acting cylinder piston unit supported on said support plate, containing a pressurized fluid actuated piston on the end of which the needle syringe is mounted and reciprocable therewith,

whereby, when the plunger is retracted and the side opening of the bore of the needle is open to the second transverse opening of the tubular mounting member, a fluid specimen can be flowed through the communication formed by the said side needle

opening, the bore of the needle, and the opening at the rearward end of the needle, pressurized fluid can then be expelled from the opening at the forward end of the barrel extension such that the plunger is carried forward by release of tension on the coiled spring, previously tensioned by the calibration and by additional rearward movement of the plunger, to expel the excess and accurately measure out a preselected quantity of fluid specimen within the needle bore for subsequent injection, after which time the cylinder piston unit can be actuated and the piston rod thereof driven forward disaligning the forward side opening of the needle and the transverse second opening of the mounting member to trap the accurately measured quantity of fluid and thrust the needle deeply into the inlet, thereafter admitting pressurized fluid into the opening into the rearward end of the barrel extension to drive the plunger all the way forward to expel the accurately measured quantity of the fluid specimen into the inlet.

6. The apparatus of claim **5** wherein the rearward opening leading into the needle is located within the tubular section to which the barrel and needle are adjoined.

7. The apparatus of claim **6** wherein the tubular section constituted of Teflon.

8. The apparatus of claim **5** wherein the outer tubular member located at the rear of the barrel extension is flanged, an externally threaded projecting shank of smaller diameter and an elongated portion of even smaller diameter extends forward thereof, and the shank portion thereof is threadably engaged with the internal threads of a connecting union which holds the outer tubular member in place at the end of the barrel extension.

9. The apparatus of claim **5** wherein the needle is permanently mounted within a septum inlet comprising outer and inner tubular members connected one member with another via a central conduit, the outer and inner members being spaced apart one from another but held in relative spaced position via a plurality of connecting support rods, the outer tubular member being internally recessed, the inside walls thereof threaded, and provided with a forward frusto conic opening leading into the said central conduit, and therein is placed a resilient tubular shaped seal, held in place via an externally threaded forwardly recessed cap threadably engagable with the internal walls of the outer tubular member such that the cap can be screwed tightly into place causing forward extrusion of the resilient material into the frusto conic opening and against the wall of the needle to form a leak-proof seal.

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