

United States Patent

[11] 3,545,491

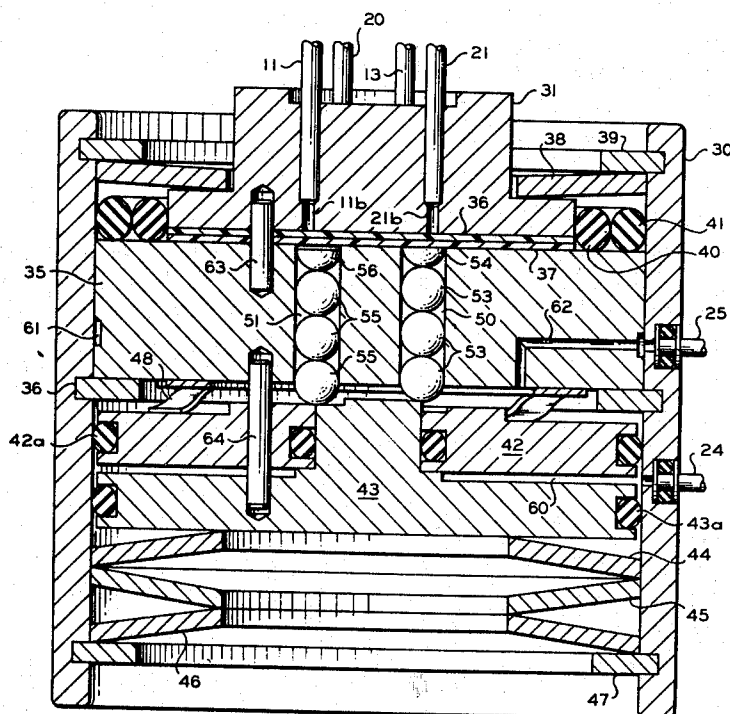
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 [21] Appl. No. **796,718**
 [22] Filed **Feb. 5, 1969**
 [45] Patented **Dec. 8, 1970**
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[56] **References Cited**
UNITED STATES PATENTS
 3,376,894 4/1968 Broerman..... 137/625.48
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[54] **CHROMATOGRAPHIC ANALYZER SAMPLE VALVE**
4 Claims, 6 Drawing Figs.

[52] U.S. Cl..... **137/625.48,**
251/367
 [51] Int. Cl..... **F16k 11/00**
 [50] Field of Search..... **251/367;**
137/625.48

ABSTRACT: A sample valve for a chromatographic analyzer is actuated by a pair of pistons which move plungers to block communication between selected valve ports. The valve components are contained within a cylindrical housing by split rings and spring washers.



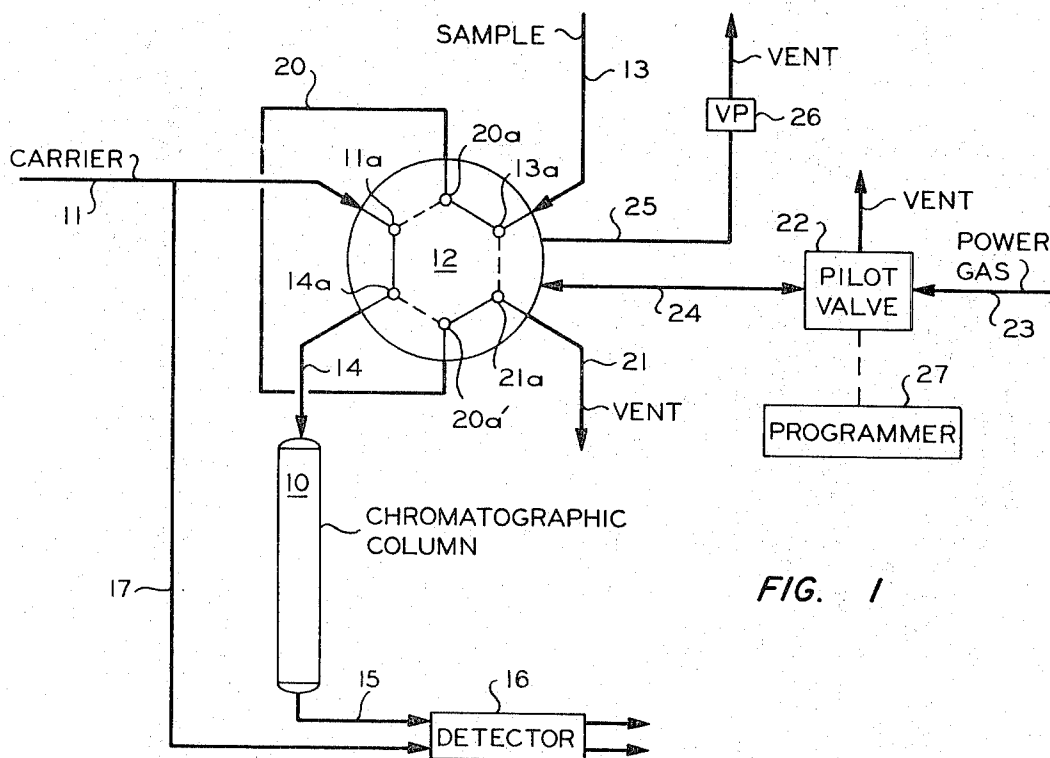


FIG. 1

FIG. 6

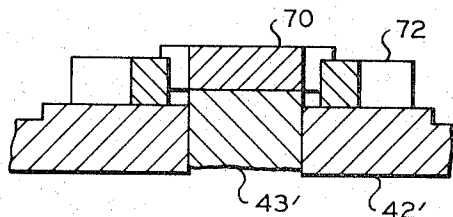
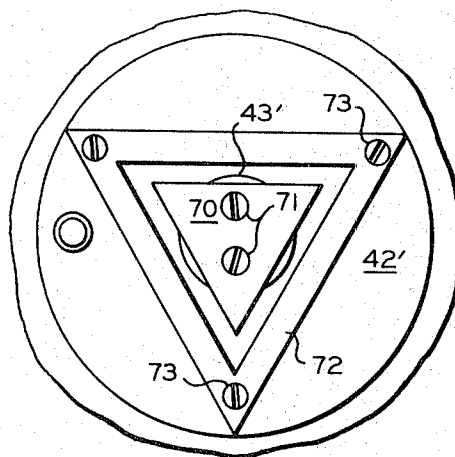


FIG. 5

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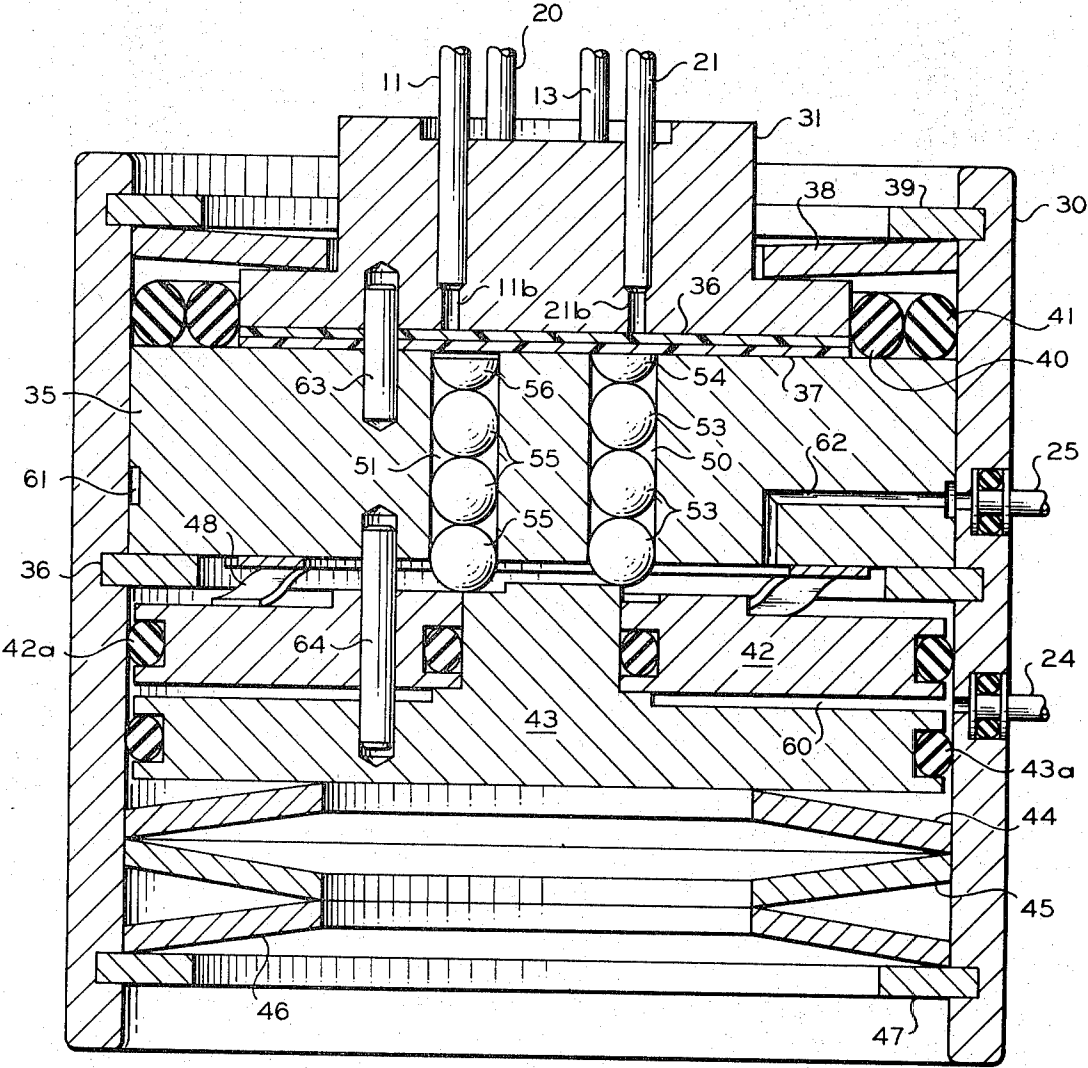


FIG. 2

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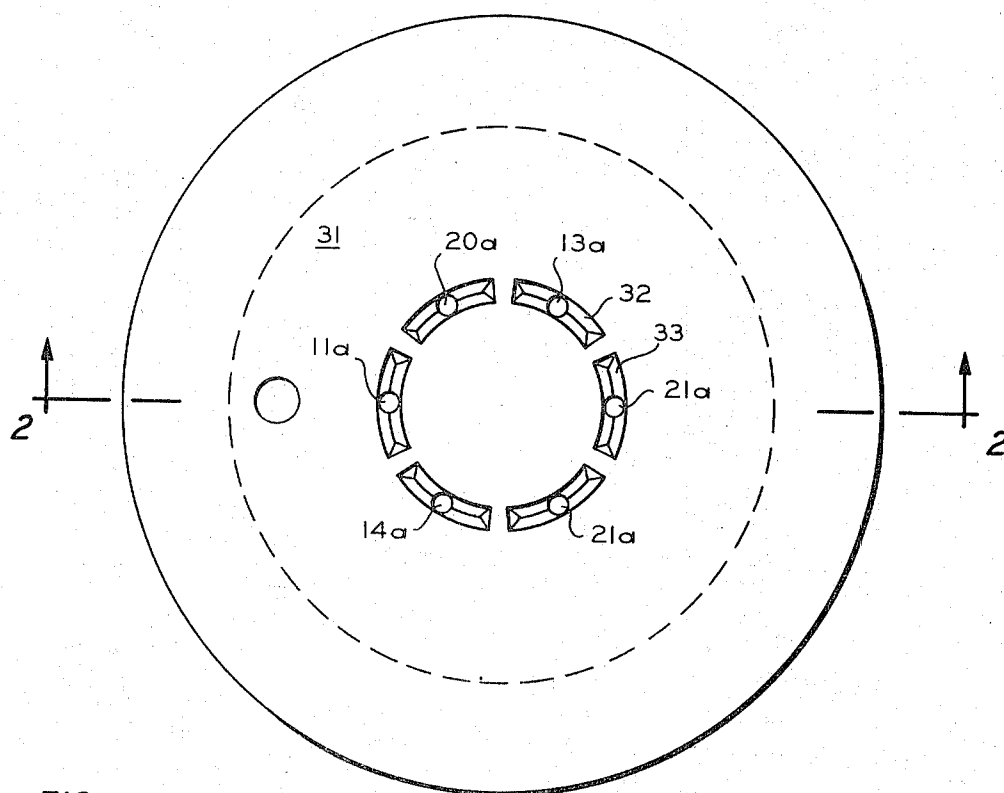


FIG. 3

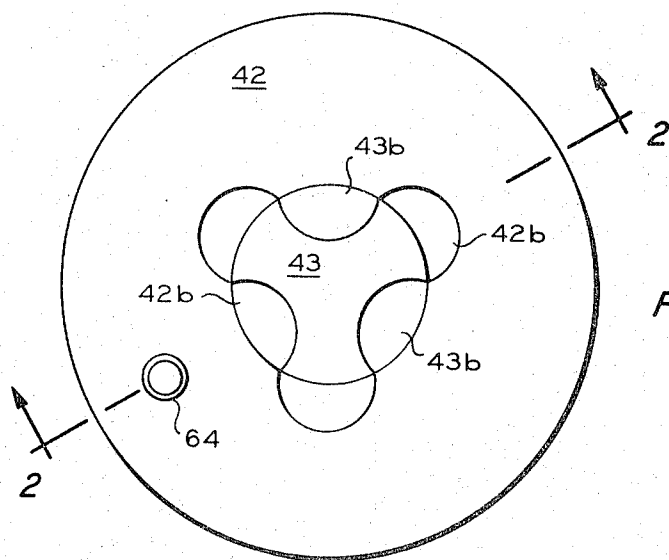


FIG. 4

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CHROMATOGRAPHIC ANALYZER SAMPLE VALVE

It is common practice to analyze fluid mixtures by means of chromatography. In a conventional chromatographic analyzer, a sample of the material to be analyzed is introduced into a chromatographic column, and carrier gas is thereafter passed through the column to elute the constituents of the sample in sequence. In order to obtain reproducible results, it is important that the sample volumes introduced into the column remain constant. This can be accomplished by means of sample valves which trap a predetermined volume of the sample in a loop and deliver this trapped volume to the column when the valve is actuated. A number of pneumatically operated diaphragm valves have been designed which are particularly effective for this purpose. One such valve is described in U.S. Pat. No. 3,376,894.

While the valve described in the foregoing patent operates in a satisfactory manner to control the introduction of carrier fluid and sample through chromatographic analyzers, the valve is somewhat expensive to construct because it is constructed of a number of parts which must be machined rather carefully. In accordance with the present invention, an improved valve of this general type is provided which requires fewer parts and which is less expensive to machine and assemble. This simplification is accomplished in part by the use of a cylindrical housing which supports all of the valve components. The components are retained in place within the housing by means of split retaining rings which are inserted in slots within the housing. Spring washers are employed to maintain a number of the components in assembled relationship under pressure. In one embodiment of this invention, the plungers which control the opening and closing of selected passages in the valve are actuated by triangular plates carried by pistons within the valve.

In the drawing, FIG. 1 is a schematic representation of a chromatographic analyzer having the sample valve of this invention incorporated therein. FIG. 2 is a view, shown partially in section, of the sample valve employed in the analyzer of FIG. 1. FIG. 3 illustrates the lower face of the cap of the valve of FIG. 2. FIG. 4 is a top view of the movable pistons in the valve of FIG. 2. FIG. 5 is a sectional view illustrating a modified form of the actuating mechanism of the valve of FIG. 2. FIG. 6 is a top view of the apparatus illustrated in FIG. 5.

Referring now to the drawing in detail and to FIG. 1 in particular, there is shown a chromatographic column 10 which contains any suitable packing or partitioning material. Carrier fluid is introduced through a conduit 11 which communicates with the first inlet port 11a of a sample valve 12. A sample of fluid to be analyzed is introduced through a conduit 13 which communicates with an inlet port 13a of sample valve 12. A conduit 14 extends from sample valve port 14a to the inlet of column 10. A conduit 15 extends from the outlet of column 10 to the first inlet of a differential detector 16. A portion of the carrier fluid is passed through a conduit 17 to the second inlet of detector 16.

Sample valve 12 is employed to introduce a predetermined volume of sample selectively to the inlet of column 10. This is accomplished by means of a sample loop 20 which extends between ports 20a and 20a' in such a manner that the sample initially flows through the loop to fill the interior thereof. After flowing through loop 20, the sample is vented through a conduit 21 which communicates with port 21a. Initially, carrier fluid flows from conduit 11 through valve 12 to conduit 14 and from there through column 10. When the valve is actuated, the flow of carrier fluid is diverted within the valve so as to pass through loop 20 before entering outlet conduit 14. At the same time, the flow of sample is vented. This results in the carrier fluid forcing the volume of sample which originally occupied the loop out through conduit 14 to column 10. Sample valve 12 is pneumatically operated and receives operating pressure from a pilot valve 22. Power gas is introduced into valve 22 through an inlet conduit 23. A conduit 24 extends between the pilot valve and the sample valve. A conduit 25 extends between a vacuum pump 26 and sample valve 12 to facilitate operation of the sample valve under certain condi-

tions, as described hereinafter in greater detail. The operation of the sample valve can be controlled by a programmer 27 which actuates pilot valve 22 at predetermined intervals.

Sample valve 12 is illustrated in detail in FIGS. 2, 3 and 4. The valve components are contained within a cylindrical housing 30. These components include a cap 31 which receives conduits 11, 20, 13, 21 and 14 of FIG. 1. The conduits communicate with respective passages, two of which are shown as passages 21b and 11b, which connect the respective inlet conduits to spaced points on the lower surfaces of cap 31. The lower ends of these passages are representative of the ports illustrated in FIG. 2. The lower surface of cap 31 is provided with a plurality of recesses which extend a part of the distance between adjacent ports. For example, a recess 32 extends from port 13a toward port 21a. Similarly, a recess 33 extends from port 21a toward port 13a. As illustrated, these recesses extend toward one another but do not meet. In similar fashion, corresponding recesses are provided adjacent each of the ports in the lower surface of cap 31.

A cylindrical body member 35 is positioned within housing 30 and supported on a split retaining ring 36 which is snapped into a central recess in the housing. Two diaphragms 36 and 37 are disposed between body member 35 and cap 31. A spring washer, such as a Belleville washer 38, surrounds the lower flanged portion of cap 31 and is engaged by a second split retaining ring 39. Annular sealing rings 40 and 41 are disposed in the chamber between body member 35, cap 31 and washer 38. These elements of the valve are of such size that washer 38 exerts a substantial force downwardly on the flanged portion of cap 31 to retain diaphragms 36 and 37 under compression.

Two movable pistons 42 and 43 are positioned within housing 30 beneath body member 35. The pistons are supported in this position by a stack of spring washers 44, 45 and 46 which rests on a third retaining ring 47. The washers exert an upward force on piston 43 which tends to move the two pistons toward body member 35. A spring 48 is positioned between body member 35 and upper piston 42 to exert a downward force on the two pistons. Body member 35 is provided with six vertical passages, two of which are shown as passages 50 and 51. The upper ends of these passages lie in the circle defined by the six ports in the lower face of cap 31, with the upper end of each passage being between a pair of adjacent ports in the cap. For example, the upper end of passage 50 is between ports 21a and 13a. Movable plunger means are disposed in each of the passages in body member 35. As illustrated, the plunger means in passage 50 comprises three stacked spheres 53 having a half-sphere 54 at the top of the stack. Similar elements are disposed in the other five passages which extend through body member 35. Spheres 55 and half-sphere 56 are disposed in passage 51. If desired, these spherical elements can be replaced by cylindrical rods.

Pistons 42 and 43 are designed to control movement of the plunger means through the passages of body member 35. As illustrated in FIGS. 2 and 3, an elevated upper surface on piston 43 engages the stack of spheres in passage 50 so as to move upper half-sphere 54 into engagement with the lower surface of diaphragm 37. This serves to compress the diaphragms between ports 13a and 21a of FIG. 3 so as to block communication between these ports. The lowermost sphere 55 in passage 51 rests on a cut out portion of piston 43 and on piston 42 so that half-sphere 56 in passage 51 is depressed from diaphragm 37 when the valve is in the position illustrated. Plunger means in the remaining passages in valve body 35 are connected in a corresponding manner with alternate plunger means engaging diaphragm 37 when the valve is in the position illustrated.

Conduit 24 of FIG. 1 communicates with a chamber 60 between pistons 42 and 43. Conduit 25 communicates with an annular recess 61 in the periphery of body member 35. A passage 62 extends from recess 61 into the region between body member 35 and piston 42. Pins 63 and 64 are provided to maintain alignment of the components when the valve is assembled.

In the absence of power gas flowing into chamber 60 of FIG. 2, the valve is in the position illustrated. Washers 44, 45 and 46 exert an upward force on piston 43, the upper surface of which engages lowermost sphere 53 so that the section of the diaphragms engaged by half-sphere 54 is moved upward to block communication between ports 13a and 21a. In similar fashion, two other sets of plungers, not shown, block communication between ports 11a, 20a and 14a, 21a. Under this condition, the conduits of FIG. 1 are connected in the manner illustrated by the solid lines within valve 12. Carrier gas flows through conduit 11 to conduit 14 and into column 10. The sample fluid flows through loop 20 and is vented through conduit 21. The pressures of these flowing fluids depress diaphragms 36 and 37 into the recesses illustrated in FIG. 3 which are not blocked by elevated plunger means.

When pilot valve 22 is switched to the second position, power gas is introduced into chamber 60 from conduit 24. As the pressure builds up in chamber 60, a force is exerted to move piston 42 upwardly against the force of retaining spring 48. This results in the upper surface of piston 42 engaging lower sphere 55 to raise the spheres through passage 51. As the pressure continues to increase in chamber 60, sufficient force is built up to overcome washers 44, 45 and 47 so that piston 43 moves downwardly. This lowers spheres 53. The combined movement of the two groups of spheres thus switches the valve to the position illustrated by the dotted lines in FIG. 1. Carrier gas is then directed through sample loop 20 to displace the volume originally trapped therein into column 10. The incoming sample is vented through conduit 21. Thus the two positions of the valve depend on the introduction of power gas into chamber 60.

In a second embodiment of the valve operation, vacuum pump 26 of FIG. 1 is connected by conduit 25 to the region between body member 35 and upper piston 42. This permits operation with carrier gas and/or sample fluid at substantially lower pressures because the application of vacuum to the underside of diaphragm 37 assists in the downward displacement of the diaphragm in the absence of the passages between adjacent ports being blocked by the plunger means. In this embodiment, it is often desirable to connect the pilot valve vent conduit to the inlet of vacuum pump 26. This removes residual atmospheric pressure from chamber 60 when an air signal is not being applied. Otherwise, there is a danger of this residual pressure overcoming spring 48.

The valve of this invention can be assembled rapidly because of the use of split rings to hold the components in assembled position. These split rings normally are provided with openings at the end so that they can be compressed for insertion and removal.

A second embodiment of the plunger actuating mechanism is illustrated in FIGS. 5 and 6. In this embodiment, a first triangular plate 70 is secured to the top of piston 43' by screws 71. Plate 70 is of such configuration that the three corners thereof engage the lower ends of respective first columns of spheres in the valve of FIG. 2. A second triangular plate 72 is secured to piston 42' by screws 73. Plate 72 is of such configuration that the central portions of the legs engage respective second columns of spheres in the valve of FIG. 2. The columns of spheres are thus raised and lowered in the same manner as previously described when plates 80 and 72 are raised and lowered by respective pistons 43' and 42'.

While this invention has been described in conjunction with presently preferred embodiments, it obviously is not limited

thereto.

I claim:

1. A sample valve comprising:

a cylindrical housing having three circumferential grooves in the inner wall thereof spaced from one another;

a split retaining ring in each of said grooves;

a body member positioned in said housing and supported by the center of said retaining rings, said body member having six longitudinal passages therethrough, the ends of which lie in a generally circular path;

a diaphragm positioned across the face of said body member which is remote from said center ring;

a cap member positioned in said housing on the side of said diaphragm which is remote from said body member, said cap member having six longitudinal passages therethrough, the ends of which lie in said circle between the passages through said body member;

spring means positioned between the second of said rings and said cap member to urge said cap member into engagement with said diaphragm;

a first annular piston member positioned in said housing on the side of said body member remote from said diaphragm;

spring means positioned between said body member and said first piston member to urge said first piston away from said body member;

a second piston member positioned in said housing on the side of said first piston member remote from said body member, said second piston member being provided with a central section which extends through the opening in said first piston member, said first and second piston members being of such configuration that a chamber is formed therebetween, said housing having an opening therein which communicates with said chamber;

spring means positioned between the third of said rings and said second piston member to urge said piston members toward said body member; and

plunger means positioned in each of the passages in said body member, the lengths of said plungers and the configurations of said piston members being such that first alternate plunger means engage said diaphragm in the absence of external fluid pressure being applied to said chamber and second alternate plunger means engage said diaphragm when external fluid pressure is applied to said chamber.

2. The sample valve of claim 1 wherein the face of said cap member adjacent said diaphragm is provided with six curved recesses, each of which extends from the end of a respective passage in said cap member toward the two adjacent passages, said recesses being of such lengths that adjacent recesses are not joined to one another.

3. The sample valve of claim 1, further comprising a first triangular plate secured to said central section of said second piston member, the corners of said first plate engaging respective first alternate plunger means, and a second hollow triangular plate secured to said first piston member to surround said first plate, said second plate engaging second alternate plunger means.

4. The sample valve of claim 1, further comprising means forming a fluid passage from a region exterior of said housing to the region between said body member and said first piston member.