

[54] **LIQUID SAMPLE VALVE**

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[58] Field of Search **73/422 R, 422 GC; 417/509, 417/498**

[56] **References Cited**

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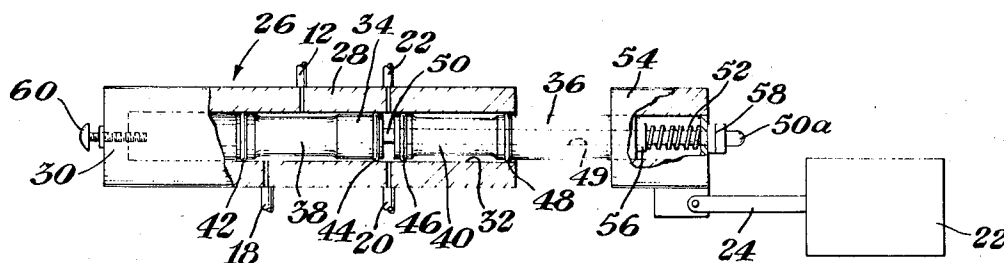
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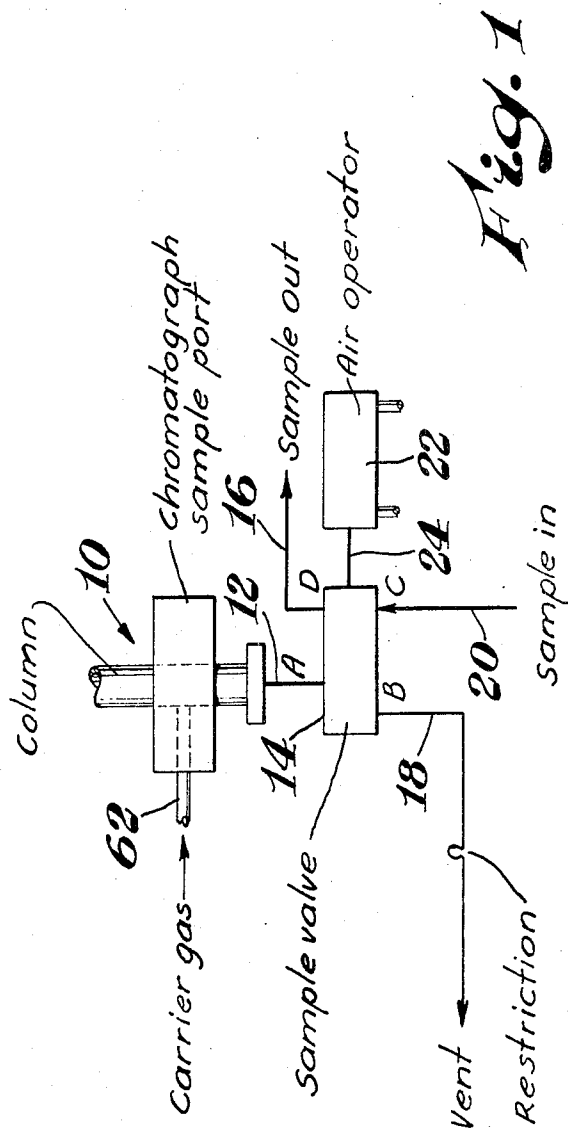
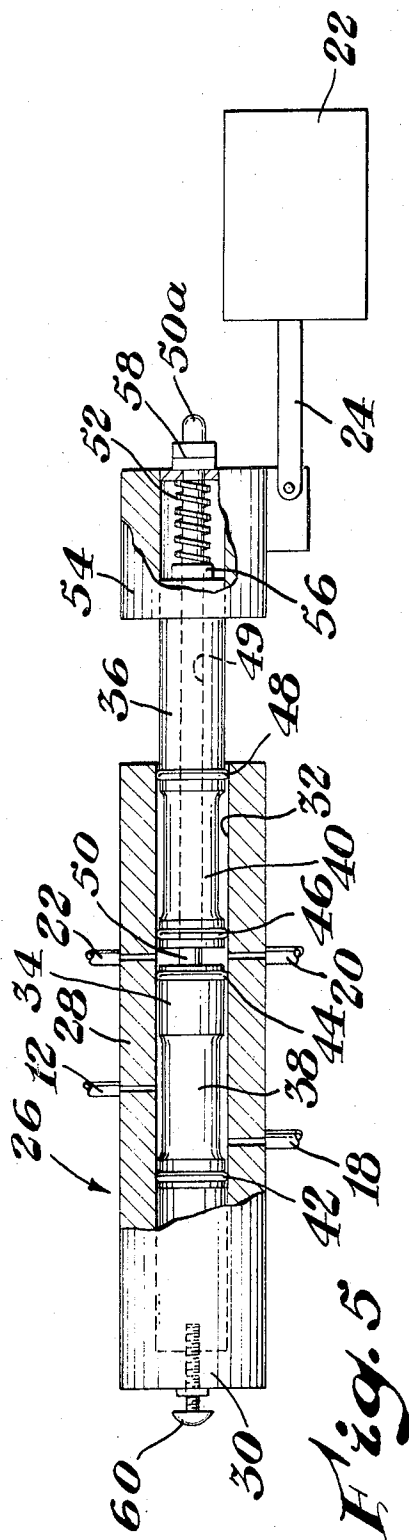
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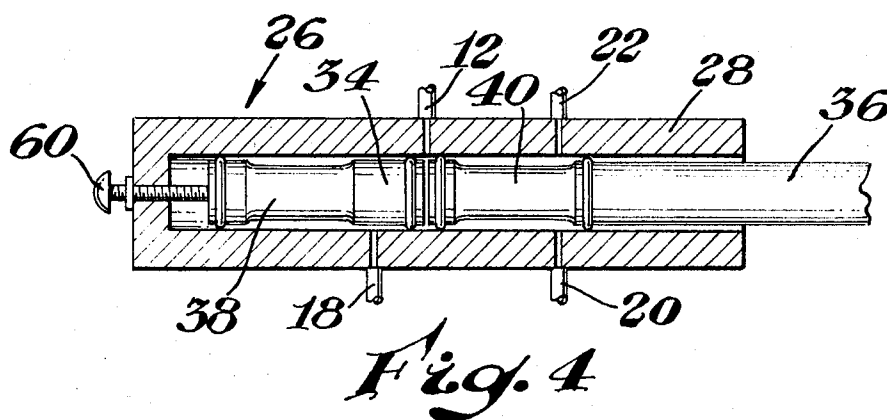
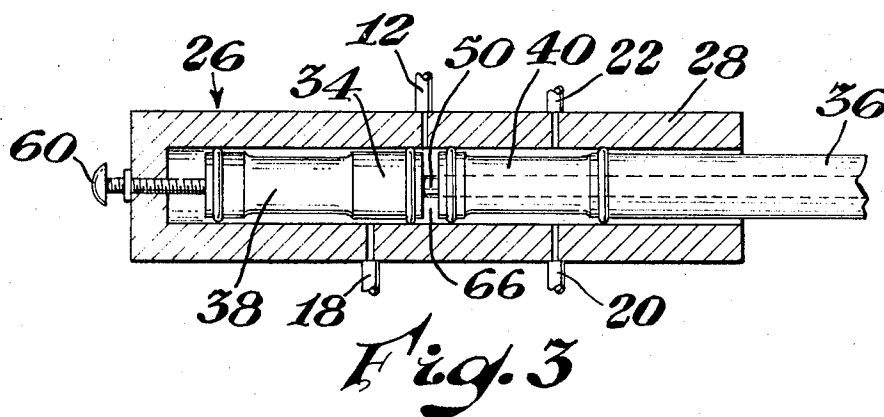
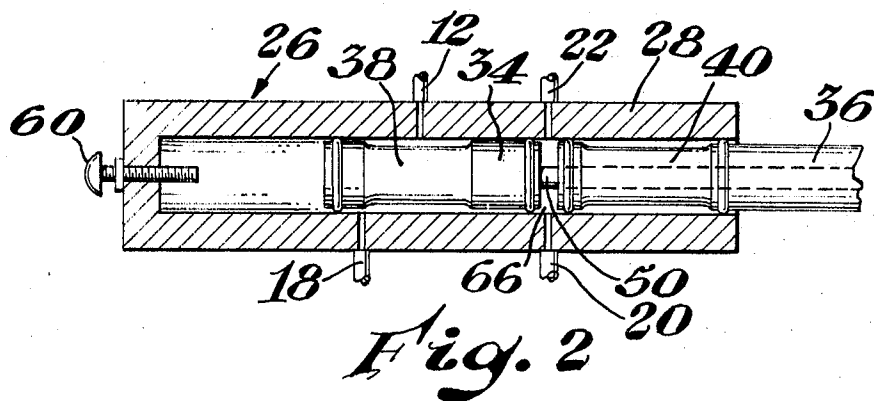
[57] **ABSTRACT**

A small hydraulic pressure injection valve having spaced apart plungers which are spring loaded with respect to each other for use in injecting a liquid sample into a chromatographic column without awaiting the action of a carrier flow gas. The sample, placed in the space between the plungers, is injected immediately into the chromatographic column as the plungers advance and are forced together. There is no problem of evaporation of the sample, as the flow path to the column is flushed as a new sample is loaded into the valve.

6 Claims, 5 Drawing Figures







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LIQUID SAMPLE VALVE

BACKGROUND OF THE INVENTION

This invention relates to liquid sample valves and particularly to positive injection liquid sample valves for use in gas chromatography.

In conventional liquid sample valves the sample is swept into the chromatographic column by carrier gas flow; with small liquid samples, this results in the sample being coated onto the walls of the connecting tubing, then evaporating slowly in order to reach the chromatographic column. Since it is best to inject the sample quickly, this coating action results in the loss of separation in component peaks.

Accordingly, a principal object of this invention is to provide an improved liquid sample valve for use in chromatography.

Another object of this invention is to provide an improved, positive displacement liquid sample valve for use in gas chromatography.

In accordance with this invention there is provided a positive injection liquid sample valve comprising a hollow cylinder, enclosed at one end, containing two plungers placed in end-to-end relationship with respect to each other. The plungers are spring loaded so that on maximum advancement into the cylinder they contact each other and substantially eliminate any space between them. When the plungers are in their retracted position, liquid sample circulates through the space between the plungers by means of opposed ports in the cylinder walls. At the same time carrier gas from the chromatographic column flows around a reduced cross section part of the forward plunger, purging the flow path into the column of any previously injected sample.

As the plungers advance into the cylinder, the sample caught between the plungers is carried forward until the space is aligned with the port leading to the column, and then the following plunger is forced forward against the forward plunger, forcing the sample into the chromatographic column.

While the sample is being advanced forward through the cylinder and into the column, sample continues to flow, by means of the opposed ports, through the valve around a reduced cross section portion of tee following plunger.

The invention, as well as additional objects and advantages thereof, will best be understood when the following detailed description is read in connection with the accompanying drawing, in which:

FIG. 1 is a diagrammatical view of a valve in accordance with this invention shown coupled to a chromatographic column;

FIG. 2 is a fragmentary sectional view showing the valve in its retracted position;

FIG. 3 is a fragmentary sectional view showing the valve in an intermediate position just prior to the discharge of sample;

FIG. 4 is a fragmentary sectional view showing the valve in sample discharging position, and

FIG. 5 is a side elevational view partly broken away and in section, of a valve assembly, including its spring loaded drive means, in accordance with this invention.

Referring to the drawing, and particularly to FIG. 1, there is shown a chromatographic column, indicated generally by the numeral 10, having a carrier gas inlet 62 at its sample port and a small diameter sample inlet

line 12 running between the sample port of the column and the sample valve 14 (in FIGS. 2-5 the valve is numbered 26). The valve contains opposed "sample in" and "sample out" ports through which sample is substantially continuously flowed through the valve cylinder.

A vent port 18 is offset from the sample inlet port communicating with the line 12 whereby the sample inlet line may be purged by leaking carrier gas therethrough while the valve is in its retracted position in which sample is loaded through it. An air operator 22 advances and retracts the valve mechanism through rod 24.

Referring now to FIGS. 2-5, there is shown a valve assembly, indicated generally by the numeral 26, comprising an elongated hollow cylinder 28 having one closed end 30 and containing a pair of plungers 34, 36 disposed in axially aligned, spaced apart end-to-end relationship.

The plungers 34 and 36 each contain parts, 38, 40 respectively, of reduced cross section which are bordered adjacent to each end thereof by O-ring type seal elements 42, 44 and 46, 48 respectively.

The plunger 36 contains an axial bore 49 through which a rod 50, coupled to the plunger 34, fitting closely but slidably, extends.

The rod 50 extends from the outer end of plunger 36. A hollow cylindrical coupling element 54 fits slidably over the outer end of plunger 36 with the end 50a of rod 50 extends. The end 50a has retainer nuts 58 thereon.

A spring 52, partially compressed, is disposed around the rod 50 between the end 56 of plunger 36 and the end of the coupling element 54 through which the rod 50 extends.

The air (or other) operator 22, coupled by means of arm 24 to the element 54, provides reciprocating movement to actuate the valve assembly.

Opposed ports, coupled to lines 20, 22, extend through the wall of cylinder 28 and are aligned with the space between the plungers 34, 36 when the valve is in its retracted position.

The line 12 and its associated port extend through the wall of cylinder 28 in alignment with the rear end of plunger 34 and the space between plungers 34, 36 as the plungers are in their most forward position. The line 18 is displaced towards the closed end 30 of cylinder 28 by a distance less than the length of the reduced cross section part 38 of the plunger 34.

The length of the reduced cross section part 40 of plunger 36 is such that sample continues to flow almost continually through lines 20, 22 as the valve is being actuated.

In operation, with the arm 24 withdrawn and the plungers pulled back as shown in FIG. 2, liquid sample flows through ports 20, 22 filling sample volume between plungers 34 and 36. A small amount of carrier gas flows from sample line 12 around relief (reduced cross section part) cut in plunger 34 and out line 18. This action keeps tube 12 flushed of any remaining sample.

To advance the liquid sample to the sample inlet line 12, force is applied to plunger 36 towards cylinder end 30. Plungers 34, 36 move together until plunger 34 contacts positive stop 60. Sample volume is carried

over, line 12 is open to this volume, line 18 is sealed off. Sample continues to flow around relief (reduced cross section part) cut around plunger 36.

Plunger 34 cannot move any farther. Since it is spring-loaded by spring 52, it will allow plunger 36 to continue moving until plunger 36 contacts plunger 34. This action reduces sample volume in the space 66 to zero, forcing sample out through line 12 into the chromatograph sample port. After a few seconds of operation, plungers will be returned to original position (by air operator 22) so that sample may again fill sample volume 66. The small amount of carrier gas glowing through line 12 will flush out this line, preventing sample for bleeding into chromatograph sample port.

Thus, it may be seen that the valve assembly in accordance with this invention is positive acting, provides means for flushing the inlet line 12 through restricted line 18 between sample injections, and introduces the relatively large volume (as compared with the small line 12) of sample into the column without need of carrier gas to drive it.

What is claimed is:

1. Sample valve apparatus comprising an elongated hollow cylinder having a side wall, a closed end, an open end and an array of four ports extending through said side wall, two of said ports lying essentially in a common plane perpendicular to the longitudinal axis of said cylinder, first and second normally spaced apart plungers having forward and rear ends, each of said plungers fitting closely but slidably in said cylinder in end to end relationship, each of said plungers having a portion of reduced cross section intermediate its ends, said first plunger being closest to the closed end of said cylinder and having a rod coupled thereto and extending from the rear end thereof, said second plunger having an axially extending bore therethrough through

which said rod fits closely but slidably, means for spring loading said rod with respect to said second plunger, means for driving said plungers in reciprocating motion, said two ports lying essentially in a plane perpendicular to the longitudinal axis of said cylinder being aligned with the space between said plungers and the other two ports being aligned with the reduced cross section portion when said plungers are in their withdrawn reciprocating motion position, said space between said plungers being aligned with one of said other two ports when said plungers are in their forward position, the other of said other two ports being disposed closer to said closed end of said cylinder and being isolated from said space between said plungers at all times during operation of said apparatus, said means for spring loading applying pressure to closed the space between said plungers on maximum forward movement of said reciprocating motion.

2. Apparatus in accordance with claim 1, wherein said closed end of said cylinder has adjustable stop means extending therethrough.

3. Apparatus in accordance with claim 1, wherein means coupled to said rod are provided for adjusting the space between said plungers.

4. Apparatus in accordance with claim 1, wherein said ports lying in a common plane are in opposed position to each other.

5. Apparatus in accordance with claim 1, wherein said ports lying in a common plane communicate with said reduced cross section portion of said second plunger when said reciprocatory motion is at other than the withdrawn part thereof.

6. Apparatus in accordance with claim 1, wherein seal means are provided on each plunger adjacent to each end of said reduced cross section portions.

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