# United States Patent

**Coelho et al.**

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## [54] GAS AND LIQUID INLET SYSTEM FOR CHROMATOGRAPHY


[72] Inventors: **Norman C. Coelho**, Diamond Bar; **Brian Thompson**, Placentia, both of Calif.

[73] Assignee: **Beckman Instruments, Inc.**



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

[51] **Int. Cl.** ..... **G01n 1/22**

[58] **Field of Search** ..... 73/422 GC, 23.1



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*Primary Examiner*—S. Clement Swisher

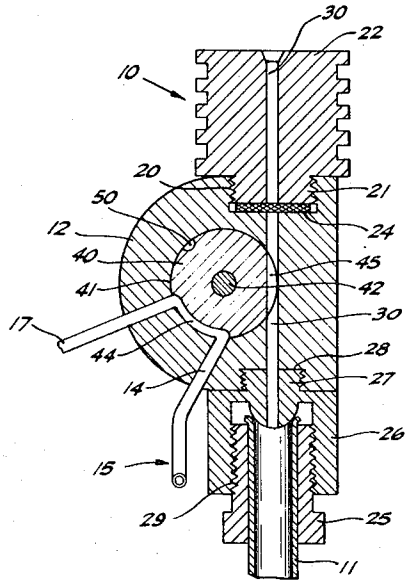
*Attorney*—Ford L. Mehlhoff and Robert J. Steinmeyer



[57] **ABSTRACT**

A valve mechanism which is adapted to be connected directly to the inlet of a gas chromatographic column for selectively directing either a gas or a liquid sample into the column. The valve mechanism comprises a valve body having an elongated channel extending entirely therethrough, the channel being coaxial with the column when the valve is attached thereto whereby a syringe may be extended through the channel into the column for direct column injection of a liquid sample. The valve body receives the opposite ends of a sample loop, sample gas inlet and outlet tubes and a carrier gas inlet tube. The valve mechanism also includes a rotatable valve plug which is operative, in a first position, to connect the carrier gas inlet tube directly to the elongated channel for injection of the liquid sample; the valve plug being further operative, in the first position, to connect the sample gas inlet and outlet tubes to the ends of the sample loop so that the loop is filled with the sample gas. Upon rotation to a second position, the valve plug is operative to connect the carrier gas inlet tube to the elongated channel via the sample loop whereby the carrier gas flushes the sample gas trapped in the sample loop into the column.


**8 Claims, 9 Drawing Figures**



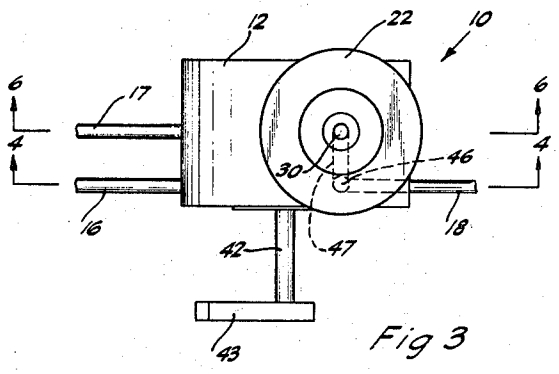



Fig 3

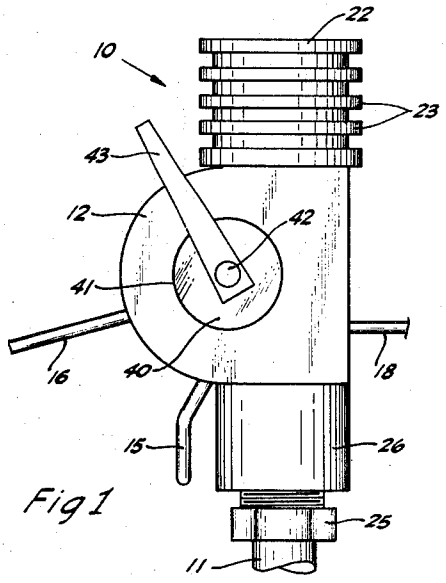



Fig 1

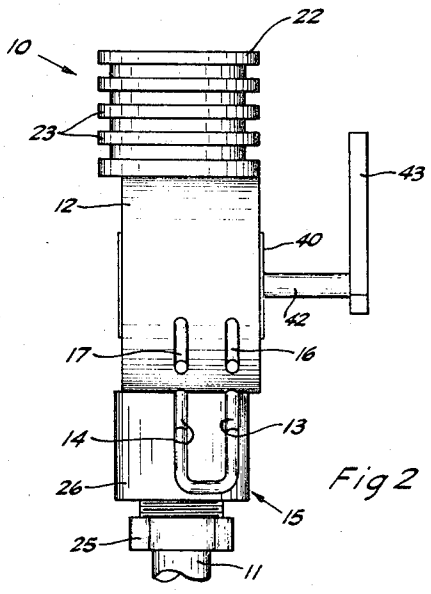



Fig 2

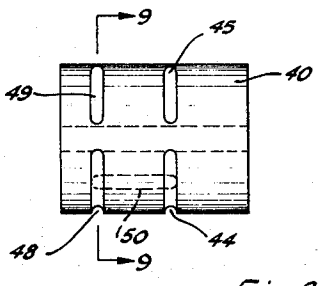



Fig 8

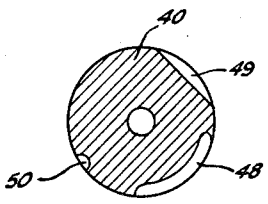



Fig 9

INVENTOR.
NORMAN C. COELHO
BRIAN THOMPSON
BY
Gerald L. Mehlhoff
ATTORNEY

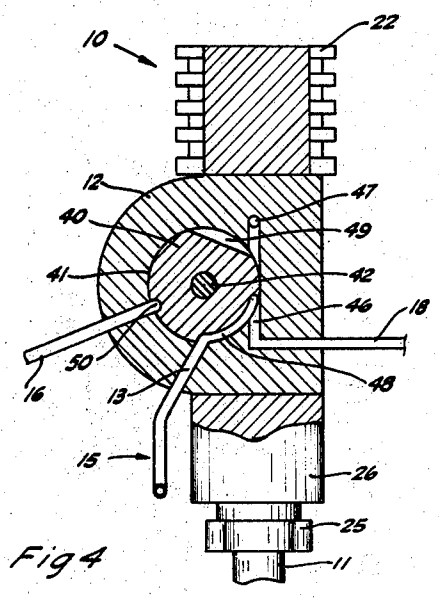



Fig 4

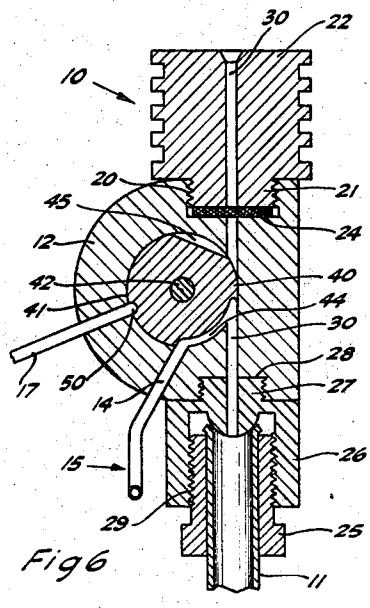



Fig 6

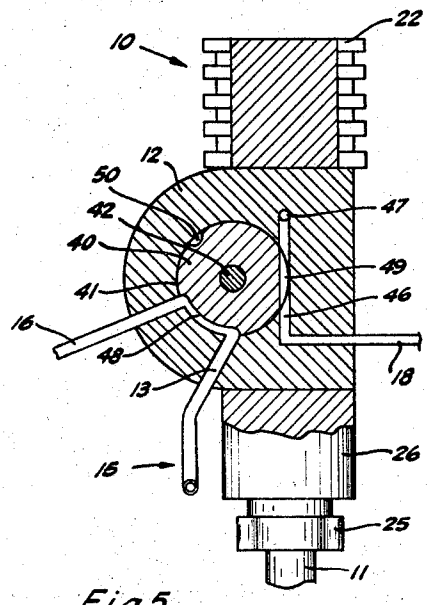



Fig 5

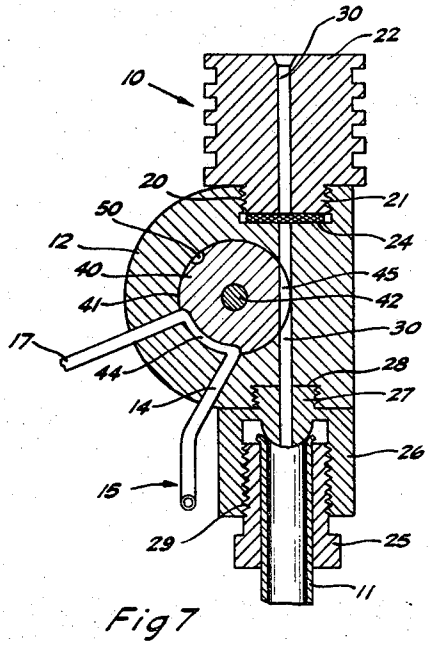



Fig 7

INVENTOR.
NORMAN C. COELHO
BRIAN THOMPSON
BY
Herbert L. Mellhoff
ATTORNEY

# GAS AND LIQUID INLET SYSTEM FOR CHROMATOGRAPHY

## BACKGROUND OF THE INVENTION

1. Field of the Invention.

The present invention relates to a valve mechanism for controlling the inlet of a gas or liquid sample into a column and, more particularly, to a valve mechanism adapted to be connected directly to a chromatographic column for providing a common inlet to the column for a gas or liquid sample.

2. Description of the Prior Art.

Gas chromatography is the phenomenon by which the components in a fluid sample are separated and analyzed by passing the sample through a chromatographic column in a fluid stream which performs the function of a carrier. The analysis is achieved by detecting the sequence in which the components of the sample mixture emerge from the column, and the amount of each component. In such instruments, the carrier fluid is usually a gas, whereas the sample, as inserted into the gas chromatograph analyzer, may either be a gas or a liquid.

Many sampling techniques are presently available for introducing a measured volume of a liquid or gas sample into a chromatographic column. One common technique in the analysis of liquid samples involves the use of a syringe by means of which a measured volume of liquid sample may be drawn from a selected fluid body and introduced into the gas carrier of the chromatographic column by direct injection. With respect to the analysis of gas samples, many different types of valves exist by means of which a measured volume of gas sample may be drawn from a selected gas stream and introduced into the carrier stream for introduction into the chromatographic column.

Although the above-described techniques exist for introducing either a liquid sample or a gas sample into a chromatographic column, such mechanisms have operated independently of each other and there is no presently available, unitary mechanism which provides a common inlet to the column for either a gas or a liquid sample. As a result, if a column includes a valve mechanism for providing an inlet for a gas sample, it has been generally necessary to completely disassemble such valve mechanism in order to introduce a liquid sample into the column. The same situation is true where the original mechanism simply permits the introduction of a liquid sample. Alternatively, where complete disassembly of a gas sample inlet valve has been impractical, it has been necessary to introduce a liquid sample into the gas stream prior to the valve. However, this technique,whereby the liquid sample is not directly injected into the column, has disadvantageous dead volume resulting in a spreading of the gas sample and a resultant spreading of the peaks of the various constituents as they are detected by the sensing means of the analyzer.

## SUMMARY OF THE INVENTION

In accordance with the present invention, there is provided a unitary valve mechanism for providing a common inlet to a chromatographic column for either a gas or a liquid sample. With the present valve mechanism, the liquid sample may be directly injected into the column to maximize the response time of the apparatus. In addition, the present valve mechanism can introduce a measured quantity of a gas sample directly into the chromatographic column by the simple expedient of rotating, from one position to another, a portion of the valve mechanism. The present valve mechanism permits such operation without the disadvantage of a complex valve construction and without the disadvantage of internal dead areas where gas or liquid samples may accumulate. The present valve mechanism is compact, easily mounted to the chromatographic column and can be easily heated.

Briefly, the present invention comprises a valve mechanism which is adapted to be connected directly to the inlet of a gas chromatographic column for selectively directing either a gas or a liquid sample into the column. The valve mechanism



comprises a valve body having an elongated channel extending entirely therethrough, the channel being coaxial with the column when the valve is attached thereto whereby a syringe may be extended through the channel into the column for direct column injection of a liquid sample. The valve body receives the opposite ends of a sample loop, sample gas inlet and outlet tubes and a carrier gas inlet tube. The valve mechanism also includes a rotatable valve plug which is operative, in a first position, to connect the carrier gas inlet tube directly to the elongated channel for injection of the liquid sample; the valve plug being further operative, in the first position, to connect the sample gas inlet and outlet tubes to the ends of the sample loop so that the loop is filled with the sample gas. Upon rotation to a second position, the valve plug is operative to connect the carrier gas inlet tube to the elongated channel via the sample loop whereby the carrier gas flushes the sample gas trapped in the sample loop into the column.

It is therefore an object of the present invention to provide a unitary valve mechanism for controlling the inlet of a gas or a liquid sample into a column.

It is a further object of the present invention to provide a common inlet to a chromatographic column for a gas or a liquid sample.

It is a still further object of the present invention to provide a valve mechanism for controlling the inlet of a gas or liquid sample into a column which permits direct column injection for the liquid sample.

It is another object of the present invention to provide a common inlet to a column for a gas or liquid sample which includes means for drawing a measured amount of gas sample from a fluid stream.

It is still another object of the present invention to provide a compact, easily mounted valve mechanism which will provide a common inlet to a column for a gas or a liquid sample.

Another object of the present invention is the provision of a valve mechanism for a chromatographic column having no dead areas.

Still another object of the present invention is the provision of a valve mechanism in which liquid and gas samples are controlled by a unitary structure at the column inlet.

Still other objects, features and attendant advantages of the present invention will become apparent to those skilled in the art from a reading of the following detailed description of the preferred embodiment constructed in accordance therewith, taken in conjunction with the accompanying drawings wherein like numerals designate like parts in the several figures and wherein:

## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is a front elevation view of the present valve mechanism;

FIG. **2** is a left side elevation view of the valve mechanism of FIG. **1**;

FIG. **3** is a top plan view of the valve mechanism of FIG. **1**;

FIGS. **4** and **5** are front sectional views of the valve mechanism of FIG. **1** taken along the line **4—4** in FIG. **3** and showing the valve plug in its two alternate positions;

FIGS. **6** and **7** are front sectional views of the valve mechanism of FIG. **1** taken along the line **6—6** in FIG. **3** and showing the valve plug in its two alternate positions;

FIG. **8** is an enlarged, side elevation view of the valve plug of the mechanism of FIGS. **1–7**; and

FIG. **9** is a sectional view taken along the line **9—9** in FIG. **8**.

## DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring now to the drawings, there is shown a valve mechanism, generally designated **10**, which is adapted to be connected directly to the inlet of a gas chromatographic column **11**, mechanism **10** providing a common inlet to column **11** for either a gas or a liquid sample. More specifically, valve mechanism **10** comprises a main valve body **12**

having any convenient shape. Valve body 12 receives the opposite ends 13 and 14 of a sample loop 15 having a U-shaped or other convenient configuration. Valve body 12 also receives sample gas inlet and outlet tubes or passages 16 and 17, respectively, and a carrier gas inlet tube or passage 18.

Valve body 12 has a generally flat upper surface having an internally threaded bore 20 extending a short distance thereinto. Bore 20 receives the externally threaded projection 21 of a generally cylindrical injection plug 22 which may include a plurality of fins 23 to aid in heat dissipation. When projection 21 of injection plug 22 is fully extended into bore 20, a space remains between the bottom of projection 21 and the bottom of bore 20. A septum 24 is positioned in this space, for purposes which will appear more fully hereinafter.

As shown most clearly in FIGS. 6 and 7, valve body 12 is adapted to be connected directly to the inlet of gas chromatographic column 11. More specifically, column 11 may have an externally threaded column nut 25 secured to the upper end thereof. Column nut 25 permits attachment of valve body 12 to column 11 through the intermediary of a column adapter attachment 26. Attachment 26 is a generally cylindrical member having an externally threaded projection 27 at the top thereof which extends into an internally threaded bore 28 in the flat, bottom surface of valve body 12. Attachment 26 also includes an internally threaded bore 29 which receives the external threads of column nut 25.

Bores 20 and 28 at the top and bottom, respectively, of valve body 12, are co-axial, not only with each other, but with the axes of injection plug 22, column attachment 26 and column 11. Extending along this axis, through injection plug 22, valve body 12, and column attachment 26, is an elongated channel 30, formed by coaxial channel sections which intersect with the cylindrical or tapered opening 41 and a valve plug 40 positioned therein, which provides an unobstructed passageway from a point external of mechanism 10 into column 11. Channel 30 is sealed by septum 24 positioned between valve body 12 and injection plug 22. However, as will be explained more fully hereinafter, the needle of a syringe (not shown) may be extended through channel 30 and septum 24 directly into column 11 in order to introduce a measured volume of liquid sample into column 11.

Valve mechanism 10 also includes an elongated, cylindrical or slightly tapered, valve plug 40 which is rotatably mounted in valve body 12 for directing either a gas or a liquid sample into column 11. Valve plug 40 is positioned in a generally cylindrical or tapered opening 41 in valve body 12 which extends from the front to the back thereof. A shaft 42 extends through the center of valve plug 40 and terminates in a handle or arm 43 (See FIG. 2) which may be utilized to manipulate valve plug 40. Arm 43 may be manually or automatically moved to its respective positions for operating the valve.

Referring now to FIGS. 6—9, the end 14 of sample loop 15 and sample gas outlet tube 17 extend into valve body 12 so as to be co-planar with elongated channel 30; the axis of valve plug 40 and cylindrical opening 41 preferably being perpendicular to this plane, namely plane 6—6 in FIG. 3. Furthermore, valve plug 40 is positioned in valve body 12 so that it extends across elongated channel 30, the right-hand edge of which, as shown in FIG. 6, being tangent to plug 40. Also as shown in FIGS. 6 and 7, end 14 of sample loop 15 and sample gas outlet tube 17 are spaced around the circumference of valve plug 40, relative to channel 30, by equal angular increments.

Valve plug 40 includes a first, arcuate groove 44 in the surface thereof, co-planar with tube 17, end 14 of loop 15 and channel 30, whose length is equal to the angular spacing between the intersections of sample gas outlet tube 17 and end 14 of sample loop 15 with valve plug 40. This is the same angular spacing as that between the intersections of end 14 of sample loop 15 and the lower half of elongated channel 30 with valve plug 40. Therefore, and as shown in FIGS. 6 and 7, which represent the two alternate angular positions of valve plug 40, groove 44 in valve plug 40 may be used to directly connect either end 14 of sample loop 15 and the lower half of elongated channel 30, as shown in FIG. 6, or sample gas outlet tube 17 and end 14 of sample loop 15, as shown in FIG. 7.

Valve plug 40 also includes a first, substantially straight groove 45, whose depth is equal to the diameter of elongated channel 30. Groove 45 is positioned in the surface of valve plug 40 so as to be co-planar with groove 44, tube 17, end 14 of loop 15 and channel 30. In addition, the angular orientation of groove 45 relative to groove 44 is such that when plug 40 is in the position shown in FIG. 7, where groove 44 connects sample gas outlet tube 17 and end 14 of sample loop 15, the surface of groove 45 is aligned with the surface of channel 30 thereby effectively forming a wall of, and completing channel 30. On the other hand, when valve plug 40 is rotated to the position shown in FIG. 6, channel 30 is blocked by the portion of valve plug 40 between grooves 44 and 45.

Referring now to FIGS. 4, 5, 8 and 9, end 13 of sample loop 15, sample gas inlet tube 16 and carrier gas inlet tube 18 all extend into valve body 12 so as to be co-planar; the axis of valve plug 40 and cylindrical opening 41 being substantially perpendicular to this plane, namely plane 4—4 in FIG. 3. Positioned in plane 4—4 is a channel 46 which extends through valve body 12 from a point just below valve plug 40 to a point just below septum 24 and just above valve plug 40, the right-hand edge of channel 46, as shown in FIGS. 4 and 5, being tangent to plug 40. The bottom of channel 46 is connected to the inner end of carrier gas inlet tube 18, the top of channel 46 being connected by a short channel 47 (see FIG. 3) to elongated channel 30. Also as shown in FIGS. 4 and 5, end 13 of sample loop 15, sample gas inlet tube 16 and channel 46 are spaced around the circumference of valve plug 40, in plane 4—4, by the same amounts that the corresponding elements 14, 17 and 30 are spaced around valve plug 40 in plane 6—6.

Valve plug 40 further includes a second, arcuate groove 48 in the surface thereof, co-planar with tube 16, end 13 of loop 15 and channel 46, whose length is substantially equal to the length of groove 44 and equal to the angular spacing between the intersections of sample gas inlet tube 16 and end 13 of sample loop 15 with valve plug 40. This is the same angular spacing as that between the intersections of end 13 of sample loop 15 and the lower half of channel 46 with valve plug 40. Therefore, and as shown in FIGS. 4 and 5, which represent the two alternate angular positions of valve plug 40, groove 48 in valve plug 40 may be used to directly connect either end 13 of sample loop 15 and the lower half of channel 46, as shown in FIG. 4, or sample gas inlet tube 16 and end 13 of sample loop 15, as shown in FIG. 5.

Valve plug 40 further includes a second, substantially straight groove 49 whose depth is equal to the diameter of channel 46. Groove 49 is positioned in the surface of valve plug 40 so as to be co-planar with groove 48, end 13 of loop 15, tube 16 and channel 46. In addition, the angular orientation of groove 49 relative to groove 48 is such that when valve plug 40 is in the position shown in FIG. 5, where groove 48 connects sample gas inlet tube 16 and end 13 of sample loop 15, the surface of groove 49 is aligned with the surface of channel 46 thereby effectively forming a wall of, and completing channel 46. On the other hand, when valve plug 40 is rotated to the position shown in FIG. 4, channel 46 is blocked by the portion of valve plug 40 between grooves 48 and 49.

Finally, valve plug 40 includes a groove 50 in the surface thereof, extending from plane 4—4 to plane 6—6. In addition, groove 50 is positioned in the surface of valve plug 40 with an angular orientation relative to grooves 44, 45, 48 and 49, so that when valve plug 40 is in the position shown in FIGS. 4 and 6, in which grooves 44 and 48 are used to connect ends 14 and 13, respectively, of sample loop 15 and channels 30 and 46, respectively, groove 50 is aligned with the intersections of sample gas inlet and outlet tubes 16 and 17, respectively, and opening 41. Therefore, and as shown in FIGS. 4 and 6, groove 50 may be used to directly connect inlet and outlet tubes 16 and 17. With valve plug 40 in the position shown in FIGS. 5 and 7, groove 50 has no function.

In operation, valve body **12** of mechanism **10** is adapted to be connected directly to the inlet of a gas chromatographic column **11** by means of column adaptor attachment **26**, as explained more fully hereinbefore. Once so positioned, valve mechanism **10** may be used to selectively direct a gas or a liquid sample into column **11**. More specifically, with valve plug **40** positioned as shown in FIGS. **5** and **7**, the needle of a syringe (not shown) may be extended through channel **30** in injection plug **22**, valve body **12** and attachment **26** directly into column **11**. In this manner, a measured volume of liquid sample may be directly injected into column **11**. In this position, the carrier gas flowing through inlet tube **18** is permitted by groove **49** in plug **40** to pass through channels **46** and **47** into main channel **30** where the carrier gas flows downwardly through channel **30** into column **11**.

At this time, the sample gas flowing in inlet tube **16** is directed by groove **48** into end **13** of sample loop **15**. The sample gas exits from end **14** of sample loop **15** and is directed by groove **44** into sample gas outlet tube **17**. In this manner, during the time that valve plug **40** is in the position shown in FIGS. **5** and **7**, sample loop **15** is filled with sample gas.

In order to trap a measured quantity of sample gas in loop **15** and to flush the sample gas so trapped into column **11**, valve plug **40** is rotated to the position shown in FIGS. **4** and **6**. In this position, the carrier gas entering inlet tube **18** is diverted by groove **48** into end **13** of sample loop **15**. The sample gas trapped in sample loop **15**, as well as the carrier gas, exits from end **14** of sample loop **15** and is diverted by groove **44** in valve plug **40** into channel **30** and eventually into column **11**.

It can therefore be seen that in accordance with the present invention, there is provided a unitary valve mechanism **10** which operates as a common inlet to chromatographic column **11** for either a gas or a liquid sample. With the present value mechanism **10**, a liquid sample may be directly injected into column **11** to maximize the response time of the apparatus. This is achieved by positioning valve plug **40** in the position shown in FIGS. **5** and **7**. In addition, valve mechanism **10** can introduce a measured quantity of a gas sample directly into column **11** by the simple expedient of rotating valve plug **40** from the position shown in FIGS. **4** and **6**. In the first position, a quantity of sample gas is trapped in sample loop **15**, but in such a manner that there is constant circulation of the sample gas into and out of the loop. Once valve plug **40** is rotated to the position shown in FIGS. **4** and **6**, the sample gas which is at that time trapped in sample loop **15** is flushed into column **11**. It should also be noted that when valve plug **40** is rotated to the position shown in FIGS. **4** and **6**, groove **50** connects sample gas inlet and outlet tubes **16** and **17** so that there is continuity in the flow of sample gas into and out of mechanism **10**.

The present valve mechanism permits such operation without the disadvantage of a complex valve construction and without the disadvantage of internal dead areas where gas or liquid samples may accumulate. Finally, the present valve mechanism is compact, easily mounted to the chromatographic column and can be easily heated.

While the invention has been described with respect to a preferred physical embodiment constructed in accordance therewith, it will be apparent to those skilled in the art that various modifications and improvements may be made without departing from the scope and spirit of the invention.

We claim:

**1.** A unitary valve mechanism for controlling the inlet of a gas or liquid sample into a gas chromatograph column comprising:

a valve body adapted to be connected directly to said column, said valve body having a substantially cylindrical or conical shaped opening therein;

a valve plug mounted in said opening in said valve body and being rotatable between first and second positions;

a sample loop, the opposite ends of which extend into said valve body and communicate with said valve plug;

a sample gas inlet passage and a sample gas outlet passage in said valve body each communicating with said valve plug;

a carrier gas inlet passage having a portion thereof in communication with said valve plug;

said valve body having elongated channel sections extending therethrough, said channel sections communicating with said valve plug;

said valve plug having a first straight groove therein cooperating with said elongated channel sections in said valve body when said valve plug is in its first position to form an elongated channel extending entirely through said valve body and said plug, said channel being coaxial with said column whereby a syringe needle may be inserted to inject a liquid sample into said column, said valve plug also being operative when in its first position to connect said carrier gas inlet passage directly to said elongated channel, said valve plug being further operative, in said first position, to connect said sample gas inlet and outlet passages to said ends of said sample loop, respectively, whereby said sample gas circulates through said loop, said valve plug being operative, upon rotation to its second position, to connect said carrier gas inlet passage to said elongated channel via said sample loop whereby carrier gas from said carrier gas inlet passage flushes the sample gas trapped in said sample loop into said channel and thence into said column.

**2.** A unitary valve mechanism according to claim **1** wherein said valve body has a second channel extending on opposite sides of said cylindrical opening therein and communicating with said carrier gas inlet passage and also connecting with said elongated channel, and wherein said valve plug has a second parallel, spaced, substantially straight groove therein, said first and second grooves being aligned with said elongated channel and said second channel, respectively, when said valve plug is in said first position thereby permitting carrier gas to flow through said elongated channel into said column.

**3.** A unitary valve mechanism according to claim **2** further comprising:

a septum, said septum extending across said elongated channel, said valve plug intersecting said elongated channel between said septum and said column whereby a syringe may be extended through said septum, said channel and said first groove directly into said column when said valve plug is in said first position.

**4.** A unitary valve mechanism according to claim **3** wherein said second channel intersects said elongated channel between said septum and said valve plug.

**5.** A unitary valve mechanism according to claim **1** wherein said ends of said sample loop and said gas inlet and outlet passages communicate with said valve plug in said cylindrical opening and wherein said valve plug has first and second parallel, spaced, arcuate grooves therein, said first and second arcuate grooves being operative to connect said sample gas inlet and outlet passages to said ends of said sample loop when said valve plug is in said first position whereby said loop is filled with sample gas.

**6.** A unitary valve mechanism according to claim **5** wherein said first arcuate groove is operative, when said valve plug is rotated to said second position, to divert the gas in said carrier gas inlet passage into one end of said sample loop and wherein said second arcuate groove is simultaneously operative to divert the gas exiting from the other end of said sample loop into said elongated channel whereby said carrier gas flushes said sample gas into said column.

**7.** A unitary valve mechanism according to claim **6** wherein said valve plug has a third, substantially straight groove therein, parallel to the axis thereof, said groove being operative to connect said sample gas inlet and outlet passages when said valve plug is in said second position.

**8.** A unitary mechanism according to claim **1** further comprising:

a septum in said valve body, said septum extending across said elongated channel whereby a syringe may be extended through said channel and said septum directly into said column.

* * * * *