Dec. 12, 1967 L. B. ROOF 3,357,233

FLUID CONTROL SYSTEM FOR ANALYZERS

Filed Oct. 5, 1964 2 Sheets-Sheet 1

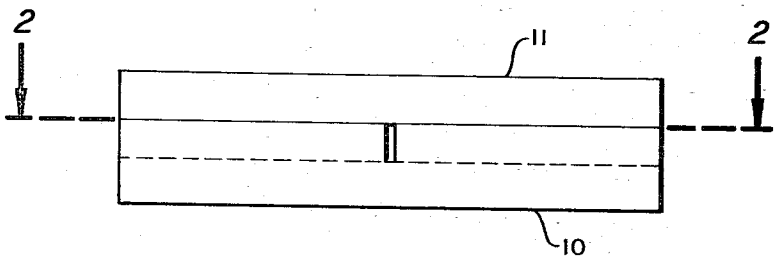


FIG. 1

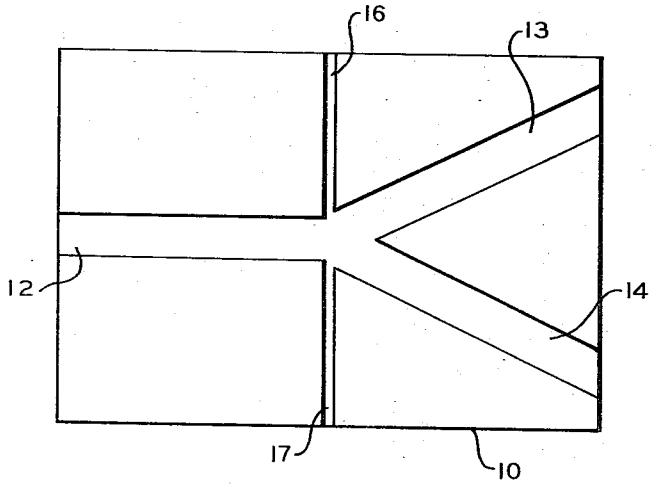


FIG. 2

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FLUID CONTROL SYSTEM FOR ANALYZERS

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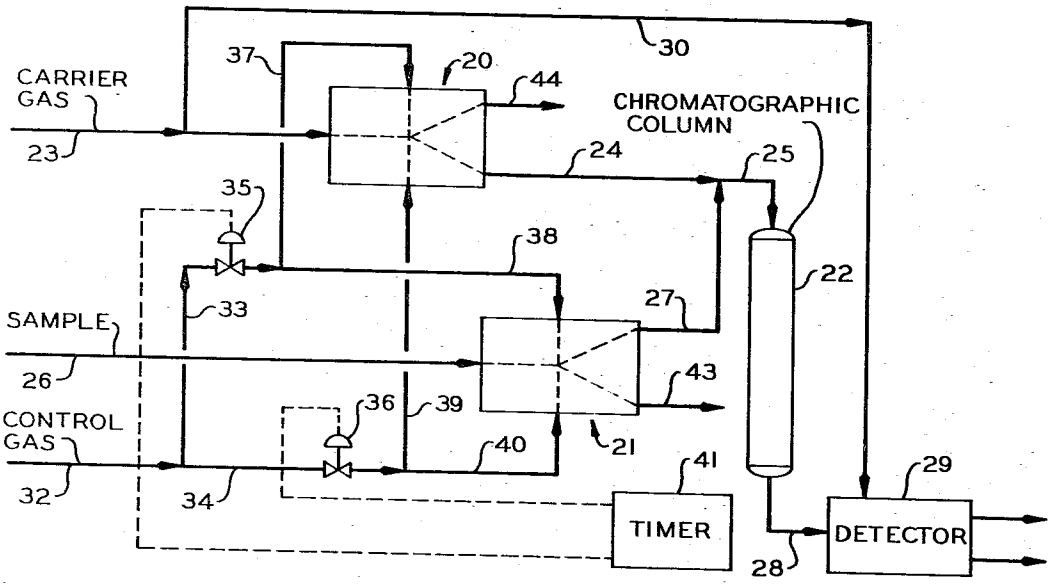


FIG. 3

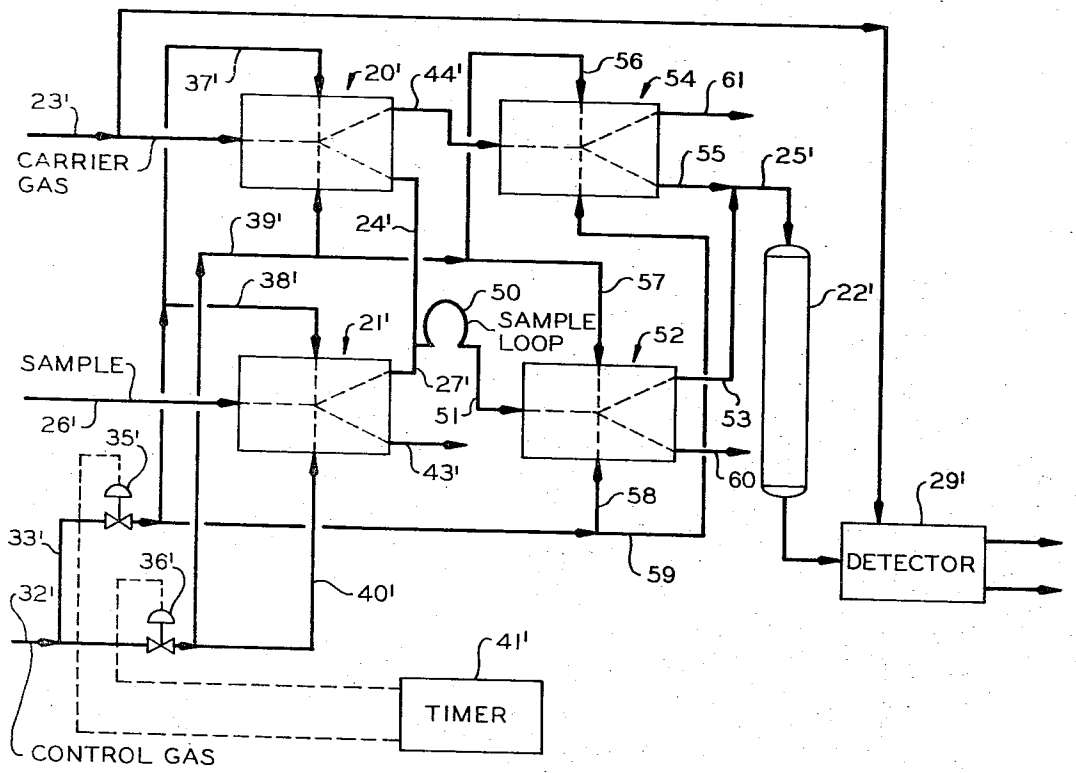


FIG. 4

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FLUID CONTROL SYSTEM FOR ANALYZERS

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ABSTRACT OF THE DISCLOSURE

A fluid control system for supplying to a conduit either a first or a second fluid, in which system the means for switching from the first to the second fluid and vice versa comprises a plurality of fluid actuated fluid stream diverting 3-way fluid valves with one outlet of each valve connected in parallel to said conduit. Said conduit may be used to supply a chromatographic column for analysis of one of said fluids. By using four of said valves, two in series in each of two parallel paths, a more accurately measured sample of said fluid to be analyzed is supplied to said conduit and/or column.

This invention relates to the distribution of fluids. In another aspect it relates to systems for introducing fluid samples and carrier gas into chromatographic columns.

In various analytical and industrial operations, there is a need for systems which are capable of controlling the flows of fluid streams in a preselected manner. One particular need for such a system occurs in chromatographic analyzers. It is common practice to analyze fluid mixtures by introducing a sample of the mixture to be analyzed into the inlet of the column which contains material that selectively retards the passage of the individual constituents of the sample. A carrier gas is then introduced into the column to push the constituents of the sample toward the outlet. A detector is employed to measure changes in composition of the effluent from the column in order to provide information which is representative of the various constituents of the sample. Various types of sample valves have been developed to control the introduction of preselected amounts of the sample into the column. These valves generally employ rotary elements or diaphragms which selectively connect various valve ports to perform the necessary switching operations. While these valves are generally satisfactory, they often tend to become complex and expensive to construct and maintain.

In accordance with the present invention, a fluid distribution system is provided wherein moving elements are eliminated from the actual stream switching mechanism. The flow control systems of this invention utilize control valves which are provided with an inlet passage which joins first and second diverging outlet passages. The passages are constructed so that a fluid introduced into the inlet passage can be made to flow through either of the outlet passages. Two control passages are provided in the valve through which small quantities or pulses of control fluids can be introduced. These control passages are positioned so that the introduction of fluid through one of the control passages diverts the main stream into the opposite outlet passages. Similarly, the introduction of control fluid through the second control passage diverts the main stream into the other outlet passage. Two or more of these valves are arranged in accordance with this invention so as to control the addition of a first fluid into a stream of a second fluid. These control systems are particularly effective in introducing samples into chromatographic columns.

Accordingly, it is an object of this invention to provide improved systems for controlling the distribution of fluids.

Another object is to provide novel valve systems for use with analytical instruments.

A further object is to provide chromatographic analyzers having improved sample systems incorporated therein.

Other objects, advantages and features of the invention should become apparent from the following detailed description, taken in conjunction with the accompanying drawing in which:

FIGURE 1 shows a control valve of the type employed in the fluid distribution systems of this invention.

FIGURE 2 is a view taken along a line 2—2 in FIGURE 1.

FIGURE 3 is a schematic representation of a first embodiment of the fluid control system of this invention employed in a chromatographic analyzer.

FIGURE 4 is a schematic representation of a second embodiment of the fluid control system.

Referring now to the drawing in detail and to FIGURES 1 and 2 in particular, there is shown a control valve of the type employed in this invention. As illustrated in FIGURE 1, the control valve is formed of two flat plates **10** and **11** which are suitably connected to one another. As illustrated in FIGURE 2, several passages are machined or etched in element **10**. The first of these passages is designated as inlet passage **12**. The fluid to be controlled is introduced into the valve through passage **12**. Passage **12** joins diverging outlet passages **13** and **14**. Control passages **16** and **17** are formed in element **10** at substantially right angles to inlet passage **12**. If a control fluid is introduced into the valve through passage **16** for example, the main fluid stream tends to be displaced downwardly so as to emerge through outlet passage **14**. Conversely, the introduction of fluid through passage **17** tends to displace the main stream into outlet passage **13**. This operation is described in greater detail hereinafter.

It should be evident that the control valve illustrated in FIGURES 1 and 2 can be formed by various procedures. As illustrated, the passages can be machined or etched in element **10** and can be of generally rectangular cross section. As an alternative, the valve can be formed of a single block wherein the individual passages are drilled. Suitable connectors, not shown, are attached to the ports of each passage to permit conduits to be attached.

A first embodiment of the fluid control system of this invention is illustrated in FIGURE 3. Two control valves **20** and **21** are employed, each of which can have the configuration illustrated in FIGURES 1 and 2. The control system of FIGURE 3 is employed to introduce carrier gas and a sample into a chromatographic column **22**. A carrier gas is introduced through a conduit **23** which communicates with the inlet port of valve **20**. Conduits **24** and **25** connect one of the outlet passages of valve **20** with the inlet of column **22**. A fluid sample to be analyzed is introduced through a conduit **26** which communicates with the inlet passage of valve **21**. Conduits **27** and **25** connect one of the outlet passages of valve **21** with column **22**. A conduit **28** extends between the outlet of column **22** and a detector **29**. A conduit **30** can be connected between conduit **23** and a second inlet of detector **29**.

A conduit **32** introduces a control fluid into the system. In general, it is desirable that this control fluid be the same as the carrier gas to avoid introduction of an additional material into the column. Conduits **33** and **34**, which have respective control valves **35** and **36** therein, communicate with conduit **32**. Conduits **37** and **38** communicate with conduit **33** downstream of valve **35** and first control passages of valves **20** and **21**, respectively. Conduits **39** and **40** communicate between conduit **34** downstream of valve **36** and the second control passages of valves **20** and **21**, respectively. Control valves **35** and **36** are actuated by a timer **41**. This timer can be any conventional device, such as motor-driven cams which control electrical

switches. Valves 35 and 36 can be solenoid operated, for example, in response to electrical signals from timer 41.

In normal operation, timer 41 first opens valve 35 and closes valve 36. This introduces control gas into the upper control passages of valves 20 and 21. The carrier gas thus passes from conduit 23 to conduit 24 and then to column 22. The sample is directed to a vent conduit 43. These flows continue until column 22 is purged and ready for operation. Timer 41 then opens valve 36 and closes valve 35 to direct the carrier gas to a vent conduit 44 and the sample into column 22. Thereafter, timer 41 again opens valve 35 and closes valve 36. This passes additional carrier gas to the column to elute the sample. The volume of sample introduced is determined by the time sample is permitted to flow to the column.

Valves 20 and 21 are constructed so that only a pulse of control fluid is needed to divert the main stream from one outlet passage to the other. For example, if the flow from conduit 23 is initially through valve 20 to conduit 44, a pulse applied to valve 20 from conduit 37 is sufficient to divert the carrier gas to conduit 24. The carrier gas will then continue to flow in this path, even without the continued application of fluid from conduit 37, until fluid is applied from conduit 39. Thus, the foregoing control procedure, and the procedures described hereinafter, can be modified to include the application of pulses of control fluids in place of a steady application of control fluid.

It is common practice to employ thermistors as the sensing elements in detector 29. A stream of carrier gas can be passed by conduit 30 to the detector so that thermal conductivity of the carrier gas is compared with the thermal conductivity of the column effluent. However, this comparison is not always required. Merely measuring changes in thermal conductivity of the effluent stream is adequate in many analyses. Furthermore, it should be evident that other types of detectors known in the art can be employed in place of thermistors.

A second embodiment of the chromatographic analyzer sample valve system is illustrated in FIGURE 4. The apparatus of FIGURE 4 employs a number of components which are similar to those of FIGURE 3 and which are designated by like prime reference numerals. Conduits 24' and 27' communicate with the inlet of a sample storage loop 50. This loop can be a conduit of any suitable volume to store a desired quantity of sample prior to the introduction thereof into column 22'. The outlet of sample loop 50 is connected by a conduit 51 to the inlet passage of a third control valve 52. A first outlet passage of valve 52 is connected by a conduit 53 to conduit 25'. A conduit 44' communicates with the inlet passage of a fourth control valve 54. One of the outlet passages of valve 54 is connected by a conduit 55 to conduit 25'. Conduits 56 and 57 extend between conduit 39' and the upper control passages of respective valves 54 and 52. Conduits 59 extend between conduit 33' downstream of valve 35' and the lower control passages of respective valves 52 and 54.

In normal operation, valve 35' is first opened and valve 36' is closed. This results in carrier gas passing through valve 20', sample loop 50, and valve 52 to column 22'. This carrier gas flow purges the sample loop and the column. Valve 36' is then opened and valve 35' is closed. This directs the flow of sample through valve 21', sample loop 50, and valve 52 to a vent conduit 60. The flow continues in this manner for a sufficient time to fill sample loop 50 with sample. Valve 35' is once again opened and valve 36' is closed. This causes the carrier gas to again flow through the sample loop to the column, and in so doing carries the sample into the column for analysis. It should thus be apparent that the system of FIGURE 4 automatically introduces a constant volume of sample into the column, as determined by the volume of the sample loop.

It is desirable that the various conduits employed in the systems of FIGURES 3 and 4 be relatively short in order to avoid stagnant accumulations of fluid therein during switching operations. In addition, it may be desirable to employ an individual flow valve immediately adjacent each of the control valves to provide more rapid control of the sample and carrier gas flows to the control valves.

While the invention has been described in conjunction with presently preferred emodiments, it should be evident that it is not limited thereto.

What is claimed is:

1. In an analyzer comprising a column adapted to contain a material that selectively retards passage therethrough of individual constituents of fluid mixtures to be analyzed, a source of carrier gas, a fluid sample source, and means communicating with the outlet of said column to detect changes in composition of the effluent from the column; a system to introduce carrier gas and sample into the column comprising:

first and second members, each having a ffuid inlet passage which joins first and second diverging outlet passages, and first and second control passages which communicate with the junction of the inlet and outlet passages in directions so that the introduction of fluid into said first control passage directs fluid from said inlet passage to said second outlet passage and the introduction of fluid into said second control passage directs fluid from said inlet passage to said first outlet passage;

first conduit means communicating between said carrier gas source and the inlet passage of said first member;

second conduit means communicating between said sample source and the inlet passage of said second member;

third conduit means connecting the second outlet passage of said first member and the first outlet passage of said second member with the inlet of said column;

fourth conduit means communicating with the second control passage of said first member and the second control passage of said second member to introduce fluid simultaneously therein;

fifth conduit means communicating with the first control passage of said first member and the first control passage of said second member to introduce fluid simultaneously therein;

sixth conduit means to introduce a fluid, a first valve connecting said sixth conduit means to said fourth conduit means, a second valve connecting said sixth conduit means to said fifth conduit means, and control means to open and close said first and second valves selectively.

2. A fluid control system comprising:

first, second, third and fourth members, each having a fluid passage which joins first and second diverging outlet passages, and first and second control passages which communicate with the junction of the inlet and outlet passages in directions so that introduction of fluid into said first control passage directs fluid from said inlet passage to said second outlet passage and introduction of fluid into said second control passage directs fluid from said inlet passage to said first outlet passage;

first conduit means communicating with the inlet passage of said first member to introduce a first fluid into the system;

second conduit means communicating with the inlet passage of said second member to introduce a fluid into the system;

third conduit means communicating between the first outlet passage of said first member and the inlet passage of said third member;

fourth conduit means communicating between the second outlet passage of said first member and the first outlet passage of said second member;

fifth conduit means communicating between said fourth conduit means and the inlet passage of said fourth member;

sixth conduit means communicating with the second outlet passage of said third member and the first outlet passage of said fourth member to remove fluid from the system;

seventh conduit means communicating with the first control passages of said first and second members and the second control passages of said third and fourth members to introduce fluid simultaneously therein;

eighth conduit means communicating with the second control passages of said first and second members and the first control passages of said third and fourth members to introduce fluid simultaneously therein;

ninth conduit means to introduce a fluid, a first valve connecting said ninth conduit means to said seventh conduit means, and a second valve connecting said ninth conduit means to said eighth conduit means.

3. In an analyzer comprising a column adapted to contain a material that selectively retards passage therethrough of individual constituents of fluid mixtures to be analyzed, a source of carrier gas, a fluid sample source, and means communicating with the outlet of said column to detect changes in composition of the effluent from the column; a system to introduce carrier gas and sample into the column comprising:

first, second, third and fourth members, each having a fluid passage which joins first and second diverging outlet passages, and first and second control passages which communicate with the junction of the inlet and outlet passages in directions so that introduction of fluid into said first control passage directs fluid from said inlet passage to said second outlet passage and introduction of fluid into said second control passage directs fluid from said inlet passage to said first outlet passage;

first conduit means communicating with the inlet passage of said first member to introduce a first fluid into the system;

second conduit means communicating with the inlet passage of said second member to introduce a fluid into the system;

third conduit means communicating between the first outlet passage of said first member and the inlet passage of said third member;

fourth conduit means communicating between the second outlet passage of said first member and the first outlet passage of said second member;

fifth conduit means communicating between said fourth conduit means and the inlet passage of said fourth member;

sixth conduit means communicating with the second outlet passage of said third member and the first outlet passage of said fourth member to remove fluid from the system;

seventh conduit means communicating with the first control passage of said first and second members and the second control passages of said third and fourth members to introduce fluid simultaneously therein;

eighth conduit means communicating with the second control passages of said first and second members and the first control passages of said third and fourth members to introduce fluid simultaneously therein;

ninth conduit means to introduce a fluid, a first valve connecting said ninth conduit means to said seventh conduit means, a second valve connecting said ninth conduit means to said eighth conduit means, and control means to open and close said first and second valves selectively.

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