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3,475,950

CHROMATOGRAPH SAMPLE SYSTEM

Filed Feb. 14, 1966

2 Sheets-Sheet 1

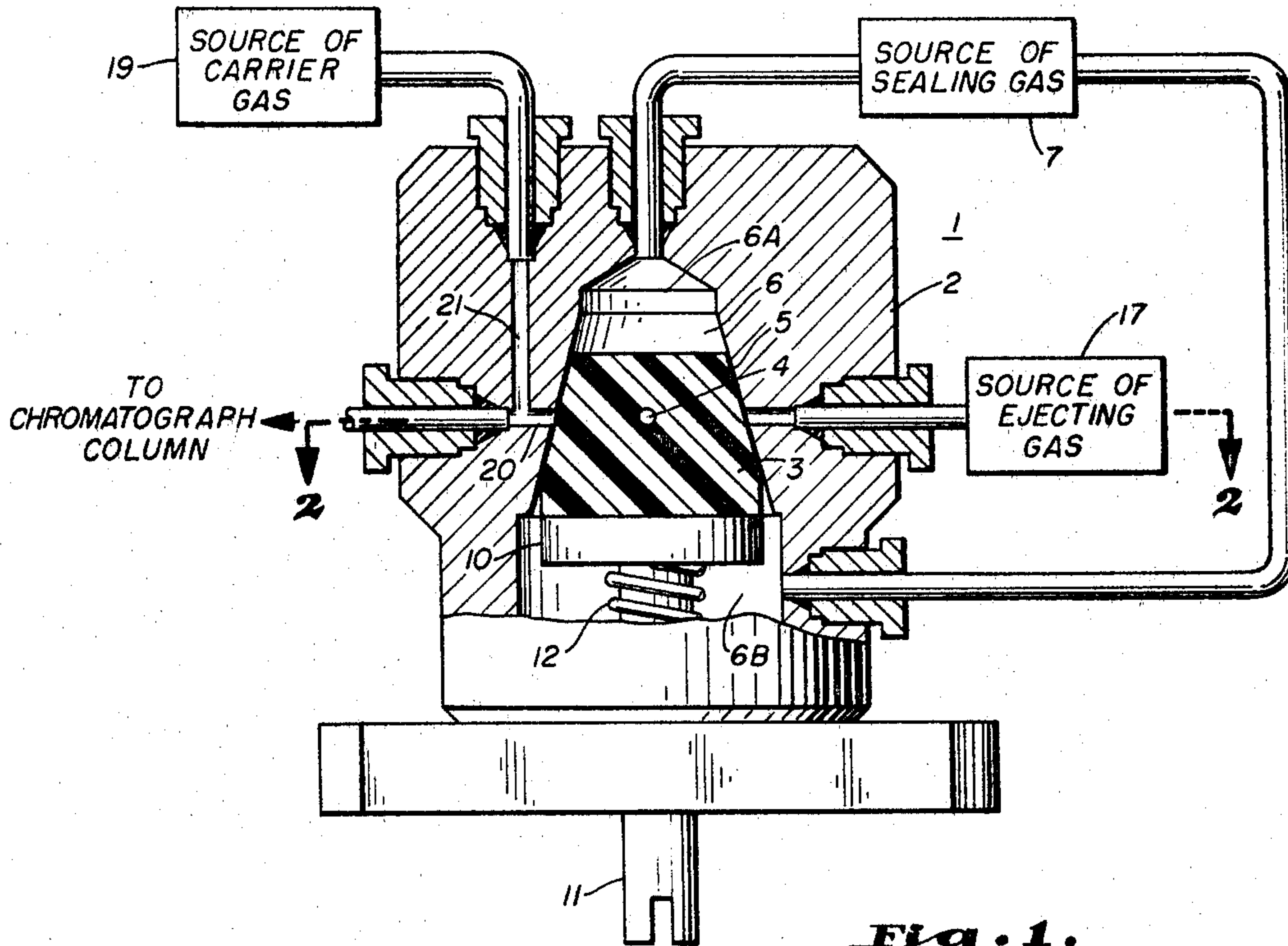


Fig. 1.

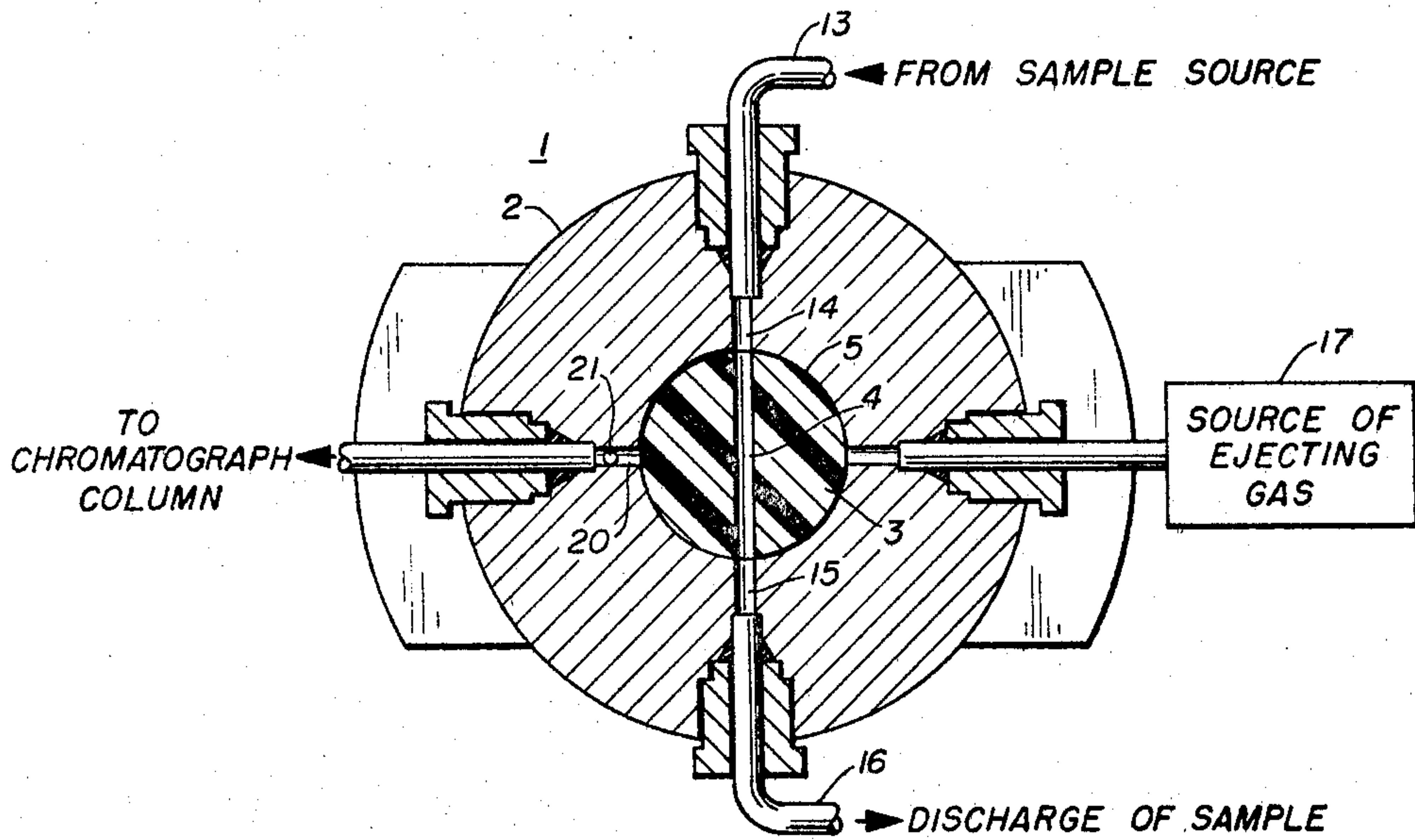


Fig. 2.

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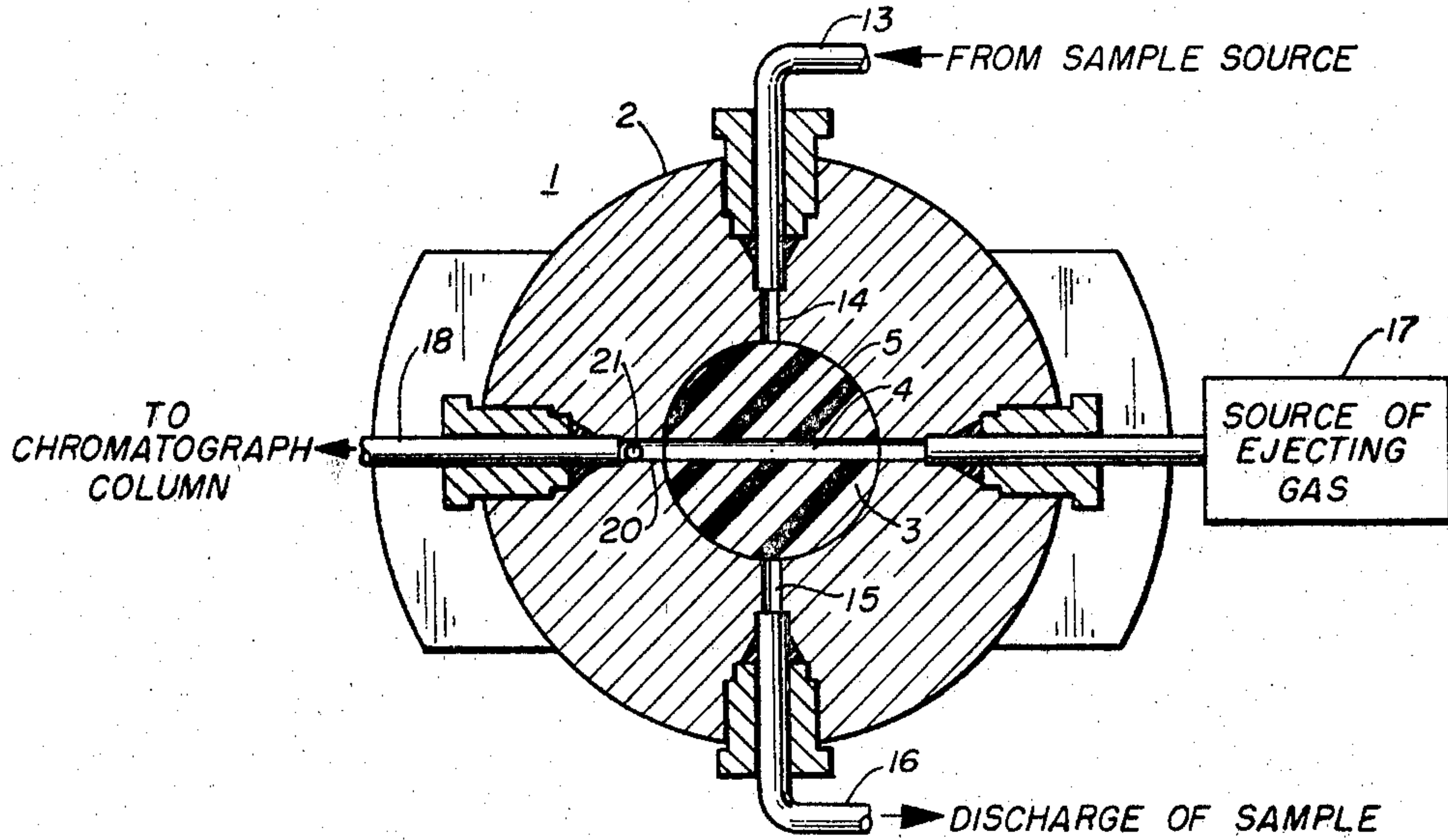


Fig. 3.

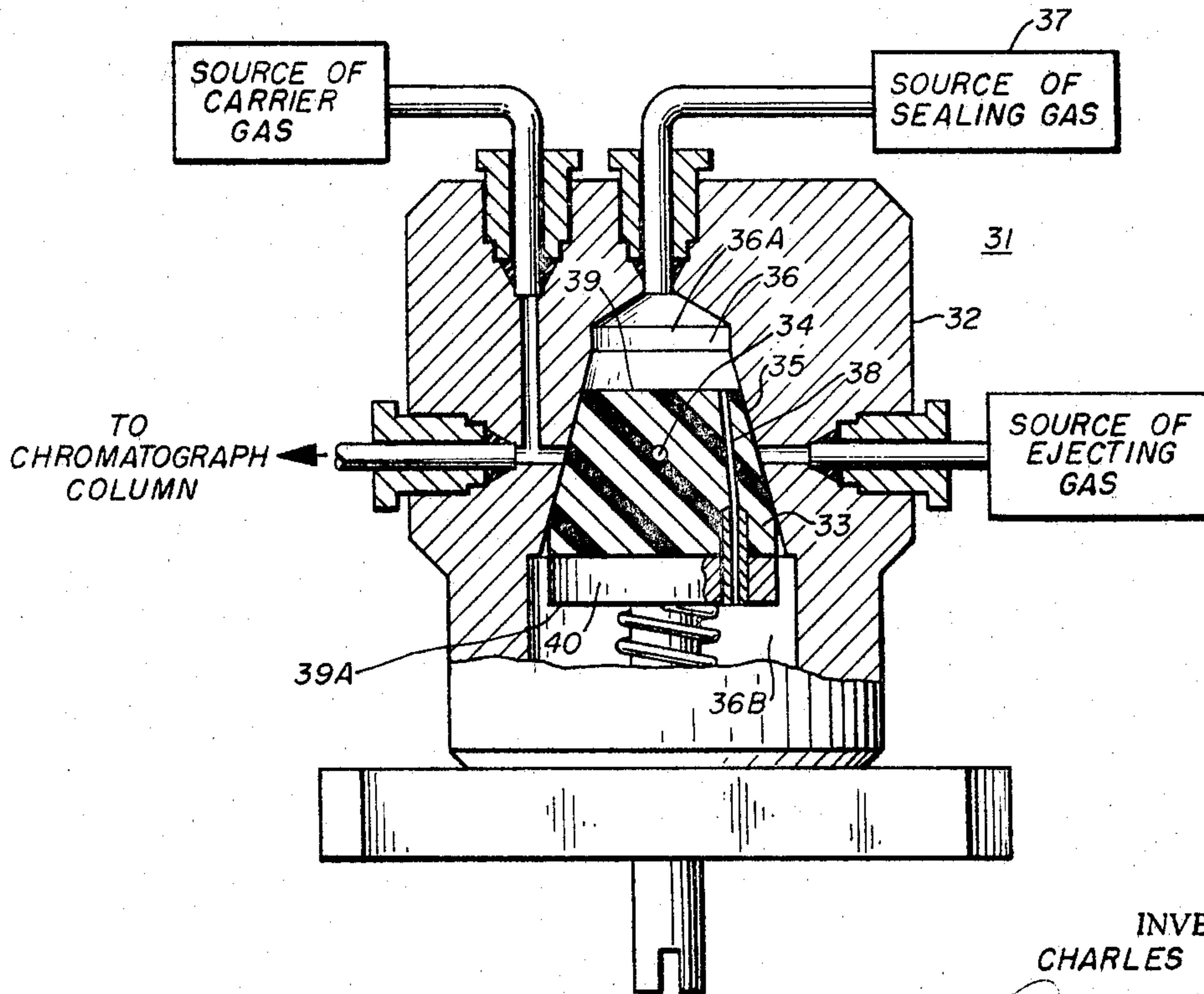


Fig. 4.

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CHROMATOGRAPH SAMPLE SYSTEM
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U.S. Cl. 73—23.1 **2 Claims**

ABSTRACT OF THE DISCLOSURE

The movable element of a valve has a passage in which a high pressure liquid sample is inserted and transferred to a chromatographic column. A source of sealing fluid is connected to the valve to apply the high pressure fluid to the sealing surface about the valve passage to prevent leakage of the sample.

The present invention relates to a method and apparatus of inserting a sample of high pressure liquid into a chromatograph column. More particularly, the invention relates to providing a pressure system for a sample transfer valve which will effectively control a liquid sample transferred by the valve from a source of sample to a chromatograph column.

One of the difficult problems in the art of chromatography is the transfer of a prepared sample of high pressure liquid into a chromatograph column. The nature of the chromatograph columns make it necessary to supply small volumes of high pressure liquid samples. It is extremely difficult to effectively isolate small sample volumes of high pressure liquid with mechanical valving structures and transfer this sample of liquid into a stream of carrier gas for entry into the column. The mechanical elements of valving structure are subject to wear with the development of subsequent leaks.

The principal object of the present invention is to apply sufficient fluid pressure across the sealing surface of a valve element which is about the ends of a sample-containing passage in the valve element while the transfer of the sample to connection with a chromatograph column is being made.

Another object is to provide for ejection of the high pressure liquid sample from the valve passage without interruption of the flow of this sample from the passage into the chromatograph column.

The present invention contemplates providing a plug valve for transferring a high pressure liquid sample. A valve passage in the plug is supplied the sample of high pressure liquid for chromatograph analysis. While the sample is held in the plug passage, a separate source of pressure is maintained on the sealing surface of the plug; the pressure being at least equal to that of the sample to thereby militate against leakage of the sample from the passage. The pressure on the sealing surface is maintained while the plug is positioned to connect the plug passage to the column.

The invention further contemplates a source of fluid for ejecting the sample from the plug passage. The source of ejection fluid is sized so it will eject the sample from the plug passage without interruption of its continuous flow into the column.

Having generally described the invention, it will now be explained in more detail by reference to the accompanying drawings showing preferred embodiments of it and in which;

FIG. 1 is a partially sectioned side elevation of a high pressure liquid entrance system for a chromatograph in which the present invention is embodied;

FIG. 2 is a sectioned plan view of FIG. 1 along lines 2—2;

FIG. 3 is a view similar to FIG. 2 but with the plug of the valve positioned to eject the liquid sample into the column; and

FIG. 4 is a view similar to FIG. 1 but with a valve plug arranged with an internal passage to develop the balancing pressure on the plug.

The broad scope of the invention

The problem is one of obtaining a very small volume of high pressure liquid and ejecting it into a stream of carrier gas for conveyance into, and through, a chromatograph column. A small passage can be formed in a valve plug for the liquid sample. The sample can be trapped in this volume. However, in present practice, as the plug is positioned, the slightest wear, or imperfection, results in an escape of some part of the liquid sample. The difference in pressure between that of the sample in the plug passage and ambient pressure on the other side of the sealing surface has been great enough to permit the escape of some of the sample. The chromatograph analysis has, heretofore, been inconsistent and inaccurate under these circumstances.

The present invention simply eliminates the pressure differential. A fluid pressure is generated on the sealing surface surrounding the plug passage to reduce its pressure differential with the sample until no leakage of the sample takes place. This pressure is maintained on the valve plug while it is positioned and no sample can escape.

The general system embodying the invention

FIGS. 1—3 illustrate one embodiment of the invention in a system including a valve 1 with its housing 2 connected to the necessary sources of fluids to be passed into a chromatograph column for analysis. Plug 3 of the valve has a passage 4 which receives the sample of high pressure liquid to be analyzed. The sealing surface 5 of plug 3 extends about the ends of passage 4, sealing against the walls of housing cavity 6.

Housing cavity 6 has a volume 6A on one side of plug sealing surface 5 and volume 6B on the other side of the plug sealing surface. Source 7 simultaneously develops a fluid pressure in housing volumes 6A and 6B great enough to retain the fluid sample in plug passage 4, effectively isolated. The plug can then be rotated to the position disclosed in FIG. 3 and the sample properly ejected from passage 4 and into the column.

Under this concept, the sealing of the high pressure liquid sample is not solely dependent upon the effectiveness of the mechanical seal between the walls of plug 4 and the walls of housing cavity 6. The pressure applied to each end of plug 3 accomplishes what mechanical sealing has failed to do in this art.

Details of FIGS. 1—3

Plug valves are well-known devices. The employment of Teflon plastic as the movable element of such valves is also well-known. The self-lubricating qualities of this material are generally understood and appreciated. Plug 3 is shown here as mounted on the end of a metallic base 10 and turned by shaft 11 while spring-urged with spring 12 into sealing engagement with the walls of cavity 6. These parts are precision made in order to maintain alignment between plug passage 4 and passages through the walls of housing 2.

FIGS. 2 and 3 show plug passage 4 rotated from the position at which it is filled with a sample to a position at which that sample is ejected from passage 4 and into a chromatograph column. The sample source is not shown. It is only required for conduit 13 be disclosed, conducting the sample to housing passage 14. From passage 14, the

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high pressure sample flows into plug passage 4. In actual practice, a stream of the sample liquid is flowed through passage 4, housing passage 15 and is discharged through conduit 16. When experience dictates the sample flowing through plug passage 4 is representative of the liquid to be tested, plug 3 is rotated to connect plug passage 4 as shown in FIG. 3. Gas from source 17 then ejects the liquid sample from plug passage 4 and into conduit 18 for entrance into the chromatograph column not shown.

A source of carrier gas is indicated at 19. This source is connected to housing passage 20 so it will convey the ejected sample into, and through, the column. Housing passage 21 is shown in simple T connection to passage 20, flowing the carrier gas into passage 20.

The pressure of the carrier gas source is only slightly above atmospheric. Therefore, it is possible that the ejecting gas from source 17 could push the sample from plug passage 4 so fast that its pressure would cause a part of the sample to flow up housing passage 21 for a finite distance. This "side-track" of a portion of the sample interrupts the smooth flow desired for the sample into the column, causing double indications of the same components.

To avoid this interruption in flow, the quantity of ejecting gas should be fixed at a value which will cause all the sample to flow past housing passage 21 smoothly, without interruption. A volume of ejecting gas could be isolated between valves and released when plug passage 4 is positioned as shown in FIG. 3. The result would be a smooth, continuous, uninterrupted flow of the sample into the column.

Preferred embodiment

FIG. 4 represents a more simple and efficient way to apply the sealing gas pressure to the sealing surface of a plug valve. Valve 31 has a housing 32 similar to the structure of FIG. 1. Plug 33 has a passage 34 for the high pressure liquid sample. The sealing surface 35 of plug 33 extends about the ends of passage 34, sealing against the walls of housing cavity 36.

Housing cavity 36 has a volume 36A on one side of plug sealing surface 35 and volume 36B on the other side of the plug sealing surface. Source 37 simultaneously develops pressure in housing volumes 36A and 36B through passageway 38 in plug 33.

Passageway 38 extends from plug face 39 to plug face 39A and equalizes the pressures of 36A and 36B. The passageway can be formed as shown. A pin from base 40 extending into the body of plug 33 is disclosed with an axial bore to which a passage in the plastic body of plug 33 connects. However, specifically disclosed here, the concept is to have this passageway 38 equalize the sealing pressure of source 37 at faces 39, 39A, or in volumes 36A and 36B, placing the complete sealing surface about sample passage 34 under the sealing pressure.

From the foregoing it will be seen that this invention is one well adapted to attain all of the ends and objects hereinabove set forth, together with other advantages which are obvious and which are inherent to the method and apparatus.

It will be understood that certain features and sub-combinations are of utility and may be employed without reference to other features and sub-combinations. This

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is contemplated by and is within the scope of the claims.

As many possible embodiments may be made of the invention without departing from the scope thereof, it is to be understood that all matter herein set forth or shown in the accompanying drawings is to be interpreted as illustrative and not in a limiting sense.

The present invention having been described, what is claimed is:

1. A system for injecting a sample of high pressure liquid to be analyzed into a chromatographic column, including:

a valve housing having a cavity whose walls comprise a sealing surface for a valve plug,

a valve plug positioned in the housing cavity to bring its outer surface into sealing engagement with the cavity walls,

a passage formed in the plug to extend to the sealing surfaces of the cavity and plug walls,

a first passage in the housing which is located to register with the plug passage in a first of the positions to which the plug is rotated,

a source of high pressure liquid sample communicated with the first passage to conduct a sample of the liquid into the plug passage when the plug is in the first of its positions,

a second passage in the housing which is located to register with the plug passage in a second of the positions to which the plug is rotated,

means for ejecting the sample from the plug passage into the second housing passage,

a source of carrier gas at a pressure substantially less than that of the sample connected to the second housing passage,

a chromatographic column connected to the second housing passage to receive the sample and carrier gas for analysis of the sample, and

a third passage in the housing located to supply a fluid pressure at least as great as that of the sample to the sealing engagement between the outer surface of the plug and the cavity walls to prevent escape of high pressure liquid from the plug passage before transfer of the sample to the column.

2. The system of claim 1 having a passage system including the third housing passage arranged to apply the fluid pressure to both sides of the valve plug in the housing cavity to balance the forces on the plug from the fluid pressure.

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