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HEATED SAMPLE INJECTION PORT

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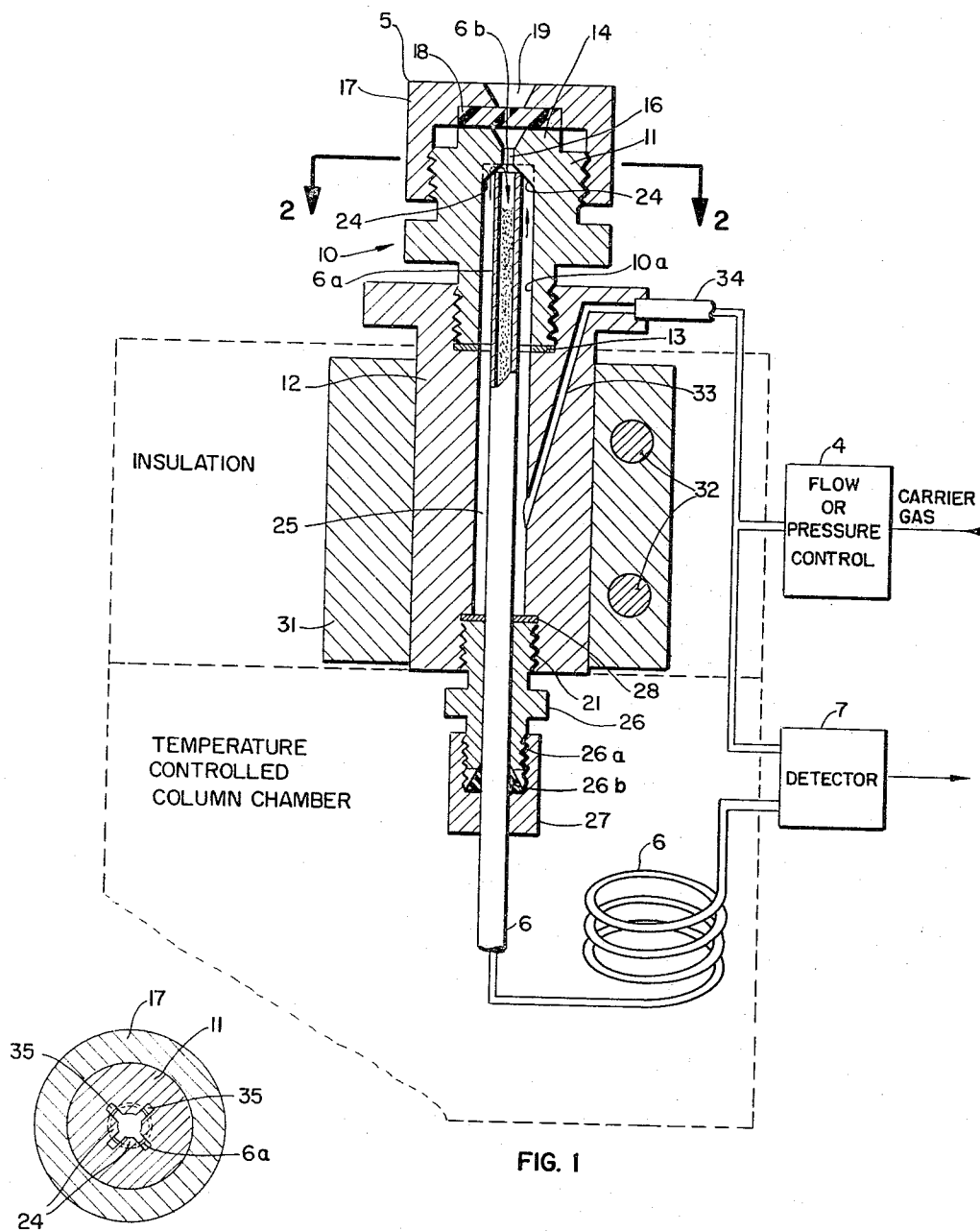


FIG. 1

FIG. 2

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HEATED SAMPLE INJECTION PORT

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1 Claim. (Cl. 73-23.1)

The present invention relates to a sample injection and vaporization port and more particularly to an improved sample injection port for the injection and vaporization of fluid samples for analysis in gas chromatographic apparatus or the like.

In gas analysis apparatus, such as gas chromatography equipment, it is necessary to insert a liquid or gaseous sample into a carrier gas stream for conveyance through the analysis apparatus. In process type chromatograph instruments sampling valves are used for injecting a vaporized sample into the instrument. However, in laboratory type analysis instruments, it is generally considered more desirable to use a syringe injection of the sample product in order to provide greater versatility regarding sample sizes or sample preparation. Because of the nature of the syringe-type sample injection, the syringe-type injection port usually includes some dead volume in the injection region. The sample sometimes becomes trapped in these dead volume regions of the injection port and is not immediately entrained in the carrier gas stream. When such an injection port is used with gas chromatograph apparatus, the portion of the sample trapped in a dead volume region contributes to peak broadening and loss of resolution as the sample constituents are passed through the system.

In addition to the elimination of dead space volume at the point of sample injection, it is desirable in gas chromatograph apparatus to inject the sample mixture and vaporize the mixture as closely as possible to the head of the chromatograph column in order to reduce to a minimum the amount of sample spreading prior to the separation column.

Accordingly, it is an object of the present invention to provide an improved injection port for injecting and vaporizing a fluid sample directly in the head of a chromatograph column.

Another object of the present invention is to provide an improved sample injection port that may easily be used for sample injection into chromatograph columns of varying diameters.

Further objects and advantages of the invention will become apparent as the following description proceeds and the features of novelty which characterize the invention will be pointed out with particularity in the claim annexed to and forming a part of this specification.

In carrying out the objects of the present invention, there is provided a tubular member defining an elongated cavity having an inlet opening covered by a self-sealing cap or rubber septum through which a syringe needle may inject a fluid sample. The head end of a chromatograph column extends through the outlet end of the tubular member so that a space is formed between the inner wall of the tubular member and the column. Clamping means retains the column within the tubular member and seals the space between the column and the tubular member adjacent the outlet opening thereof. A heating means is disposed closely adjacent the tubular member for heating the tubular member and the head end of the chromatograph column. There is provided a guide means adapted to align the end of the chromatograph column with the inlet opening of the tubular member so that a sample may be injected through the rubber cap directly into the column. In order to sweep a sample through the column, there is provided a carrier fluid inlet com-

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municating through the wall of the outer member with the space between the tubular member and the column. At least one opening through the guide means permits carrier fluid to enter the head end of the column to carry on injected sample therethrough. Heat applied to the carrier fluid, in its passage through the outer tubular member and within the space between the column and the outer member, aids in the vaporization of the sample in the head of the column and sweeps it from its point of injection through the column.

For a better understanding of the invention, reference may be had to the accompanying drawing in which:

FIG. 1 is a schematic representation of a gas chromatograph apparatus, illustrating in cross-section the injection and vaporization port of the present invention; and

FIG. 2 is an enlarged cross-sectional view taken along line 2-2 and illustrating the guide means for aligning the chromatograph column.

The gas chromatograph apparatus of the drawing includes a pressure regulator or flow controller 4, a sample injection and vaporization port or device 5, which will be described later, a chromatograph column 6 or system of columns adapted to receive the volatilized sample from the injection apparatus and a detector 7. The flow of carrier gas or fluid, ordinarily helium, although other fluids such as argon, nitrogen, carbon dioxide, hydrogen and even air may be used, is controlled by the pressure regulator 4. At a particular time, a quantity of sample is injected into the injection port 5 and volatilized therein. The sample is then swept by the carrier gas stream from the head end of the column and carried through the column 6. In the chromatograph column, the sample constituents are separated and the detector 7 provides an output indication of the presence of the individual sample constituents as they emerge from the column 6.

The output of the detector may be used as a quantitative measure of the sample constituents. In actual practice, the output of the detector is ordinarily recorded on a recorder instrument (not shown) for subsequent review, although the output may merely be indicated by contemporaneous visual inspection of a meter or other device.

Normally the column is enclosed in a temperature controlled chamber such as is indicated schematically by the broken lines in the drawing. The detector and the column may or may not be enclosed in the same chamber. However, in laboratory instrumentation apparatus, it is sometimes desirable to temperature program the column while it is desirable to maintain the detector at a uniform temperature. Thus, the apparatus is illustrated with the detector 7 separate from the temperature controlled chamber housing the column 6.

The sample injection and vaporization port 5 is preferably mounted in the insulated wall of the column chamber so that it is easily accessible to the operator. While the device is shown in the vertical position in the drawing, it is obvious that it may be disposed in any desired orientation in the apparatus. In the illustrated embodiment of the invention, the port comprises a tubular member, generally designated by the reference numeral 10, which is formed of an inlet end section 11 and a feed-through block 12. The inlet end section 11 is removable from the feed-through block 12 and may be attached to the block 12 by means of mating threaded sections or other suitable means. As shown in the drawing a metal gasket 13 provides a gas-tight juncture between the coupled ends of the block 12 and the inlet end section 11 so that gas may not escape the cavity formed within the tubular member. As will be seen in the drawing, the feed-through block 12 is mounted or disposed within the insulation of the temperature controlled chamber and the feed-through block

is adapted to deliver the sample and carrier gas through the wall of the chamber.

The outwardly extending inlet end section 11 has its upper or remote end thereof partially closed by the end wall 14, which contains a small inlet opening 16 centrally located therein. In the preferred embodiment of the invention, the opening 16 is coaxial with the axis of the tubular member 10. The inlet end section 11 is threaded on its outer surface to receive a retaining cap 17 under which is retained a self-sealing cap or rubber septum 18 of the type commonly employed for sealing an opening to be punctured by a syringe needle. Rubber septum 18 seats against the end wall 14 and is sealed against the inlet end section 11 due to the compression of the threaded retainer cap 17. An opening 19 in the threaded retainer 17 is in alignment with the inlet opening 16 in the tubular member to permit insertion of a syringe needle for injection of a fluid sample through the septum 18.

As will be noted in FIG. 1, the cavity or channel 10a and the outlet opening 21 of the tubular member 10 are relatively larger than the diameter of the column 6. Thus, the head end 6a of the column is extended into the cavity 10a so that the end 6b of the column is disposed against the upper or inlet end of the tubular member 10. In order to align the column 6 with the inlet opening 16, a guide means is provided adjacent the inlet end of the tubular member 10. In the illustrated embodiment of the invention, the guide means comprises a plurality of beveled flange sections 24, which cooperate to form inverted V-shaped centering blocks. The head end 6a of the column is merely forced against the sections 24 and it automatically centers itself coaxially within the tubular member 10. In the preferred embodiment of the invention illustrated the flanged section 24, along with the means supporting the column at the lower end or outlet end of the tubular member, retain the column 6a in a substantially coaxial position and form a space 25 between the column and the inner wall of the cavity 10a. The inlet opening of the column is, therefore, arranged in alignment with the inlet opening 16 in the tube member 10 and the opening 19 in the retainer cap 17 in order to receive a syringe needle injected into the injection port through the rubber septum 18.

While beveled flange sections 24 are shown as the preferred means for guiding or aligning the head end 6a of the column, it will be understood that other structural means are capable of performing this function. For example, the upper end of the tubular member 10 may be made frusto conical in shape so that the side walls thereof divert the column into the proper location. Or, as a further example, the inlet end of the tubular member 10 may have convex curved flanges instead of beveled flanges 24. It is believed that such adaptations are the equivalent of the flange sections 24 illustrated and are within the scope of the present invention.

Means for supporting the column and sealing the space 25 adjacent the outlet end 21 is provided in the form of a threaded tube fitting or plug 26. Plug 26 attaches to the outlet end of the feed-through block 12. In the illustrated embodiment of the invention, the tube fitting 26 is threaded into the outlet end 21 of the tubular member 10 or feed-through block and is adapted at its other end with threads 26a for the attachment of a suitable fitting to clamp the head end of the column into the injection device. In the illustrated embodiment of the invention, the column clamp fitting includes a threaded cap 27 designed to be turned onto the threads 26a of the plug 26 thereby to force a wedge section 26b into a tight engagement around the column 6 and thereby support the column within the outlet end 21 of the member 10. Plug 26 abuts against a metal seal gasket 28 to provide a gas tight closure between the tube member 10 and the plug.

The above-described construction permits easy removal and replacement of column into the device. Merely by loosening the cap 27, the column may be removed and

another inserted until it abuts the upper end or flanges 24 and, thereby becomes properly aligned with the inlet opening 16. The diameter of the column 6 may be varied within the limits imposed by the dimensions of the tubular member 10 and the outlet opening 21 but, of course must be larger than the inlet opening 16. However, if the diameter of the column 6 is varied, it will also be necessary to provide a new plug 26 capable of receiving the diameter of the new column.

In order to vaporize a sample injected through the inlet opening of the device, there is provided a heating means or coil formed within a heating block 31. The heating block 31 is arranged within the insulation of the oven and closely disposed around the feed-through block 12 of the tubular member 10 and vaporization device. Heat is supplied by any suitable means, such as the electrical resistance heating rods 32, or any suitable source of energy such as hot gases or steam whichever is desired. The lower end region of the tubular member 10, the space 25 as well as the column 6 are, therefore, retained at a high temperature for vaporizing a sample injected into the inlet end 6b of the column.

It will be understood that the length of the inlet end section 11 may be increased so that the outer extremities or inlet end of the apparatus may be maintained at a somewhat lower temperature. Thus, for example, the inlet end 11 may be made substantially longer and the head end 6a of the column extended for a greater distance into the cavity 10a thereby moving the inlet opening 16 a greater distance from the direct heat of the heater block 31 to substantially reduce the temperature at the opening.

Carrier gas is conducted into the device to sweep the injected sample from its point of injection and vaporization through the column 6. More specifically, carrier gas is conducted into the apparatus through a port or channel 33 formed through the feed-through block 12. Channel 33 connects with a source of carrier gas through conduit 34 which is directly attached to the feed-through block 12 in a suitable manner, such as by welding or by means of a fitting similar to that used to support the column 6 in the outlet opening 21. It should be noted that the carrier gas stream flows through the channel 33 in the region of the heated section of the feed-through block 12 and is heated during its passage through the block 12 into the space 25 between the chromatograph tube 6 and the wall of the tubular member. During passage through the channel 33, the gas is raised to approximately that of the temperature of the feed-through block 12. Carrier gas entering the space 25 is prevented from escaping through the outlet end 21 of the tubular member 10 by the clamping means in the end of the feed-through block 12. In order to provide a passage for the flow of carrier gas into the inlet end 6b of the column 6, at least one aperture or opening is provided through the guide means in the inlet end of tubular member 10. In the preferred embodiment of the invention, as may best be seen in FIG. 2, a plurality of openings or apertures 35 are formed between the beveled flanges 24 through which carrier gas may flow from the space 25 into the end of the column 6. Carrier gas flowing through the openings 35 sweeps directly through injection end of the column and exposes any sample injected into the column to the heated carrier gas. Thus, as may be seen in the drawing, when a syringe needle is inserted, heated carrier gas flows along the needle in the direction of the injection in order to insure complete and rapid volatilization of all sample injected.

While in accordance with the patent statutes there has been described what at present is considered to be the preferred embodiment of the invention, it will be understood by those skilled in the art that various changes and modifications may be made therein and without departing from the invention, and it is, therefore, the aim of the appended claim to cover all such changes and modifications as fall within the true spirit and scope of the invention.

What is claimed is:

In combination with a chromatograph apparatus having a column adapted to separate a sample mixture carried in a carrier stream through said column and a temperature controlled column chamber, a sample injection port comprising:

a tubular member defining a cavity having inlet and outlet openings, said tubular member being of sufficient size to receive the head end portion of the chromatograph column through said outlet opening thereof with space remaining between the outer wall of said column and the interior of said cavity;

clamping means in the outlet end of said tubular member for retaining said column and sealing said space between the column and the tubular member adjacent the outlet end thereof;

means disposed in said tubular member for aligning the head end of the chromatograph column with said inlet opening in said tubular member;

a self-sealing cap disposed over said inlet opening of said tubular member through which a syringe needle may be inserted into said inlet opening of said tubular member and the head end portion of said chromatograph column;

heating means closely adjacent said tubular member for heating said tubular member and the head end portion of said chromatograph column disposed therein to provide a heating means separate from that of said temperature controlled column chamber in order to establish a temperature at the head of said column which is optimum for sample vaporization;

a carrier gas injection port communicating through said

tubular member with said space between said chromatograph column and said tubular member; and at least one aperture adjacent said inlet opening of said tubular member for the flow of carrier gas from said space between said tubular member and said column into the end of said column for sweeping an injected sample through said column.

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