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Golovistikov

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[54] **APPARATUS FOR INJECTING A SAMPLE INTO A GAS CHROMATOGRAPH**

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

[51] **Int. Cl.** **G01n 1/28**

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

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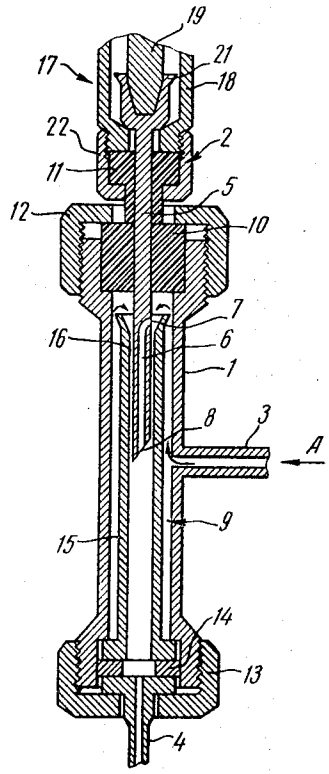
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Primary Examiner—S. Clement Swisher
Attorney—Holman & Stern

[57] **ABSTRACT**

An apparatus for injecting a sample into a gas chromatograph, has a sample evaporation chamber, wherein there is reciprocably received a sample-injecting pipette in the form of a needle with an internal longitudinal capillary passage for accommodation of a sample, having an inlet and an outlet. The inlet of the passage is made through the external side wall of the needle. The sample evaporation chamber is hermetically sealed from the ambient atmosphere with the help of sealing means; it communicates with the supply source of the carrier gas of the gas chromatograph and with the chromatographic column of this chromatograph. The sample evaporation chamber incorporates a member for guiding the carrier gas into the capillary passage of the needle. The sealing means includes a septum made of an elastic material and a sleeve. The septum is received at an end portion of the sample evaporation chamber, where the needle is introduced, whereas the sleeve envelopes the external surface of the needle. The needle is made of a material which can pierce the septum, as a sample is being injected into the sample evaporation chamber.

8 Claims, 6 Drawing Figures



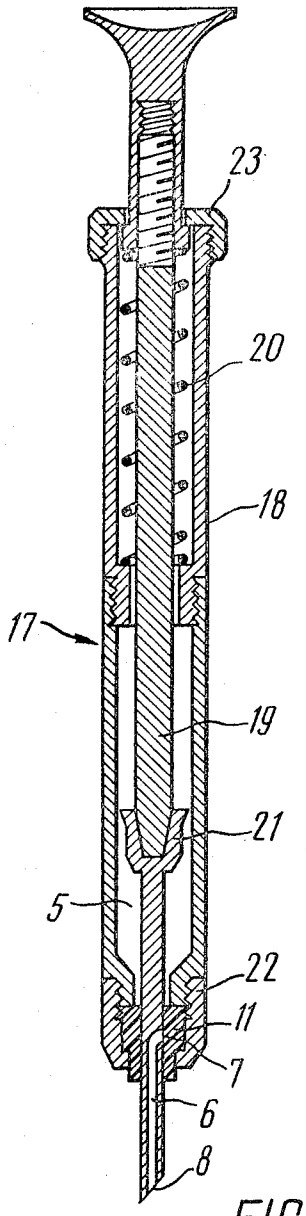

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FIG. 5

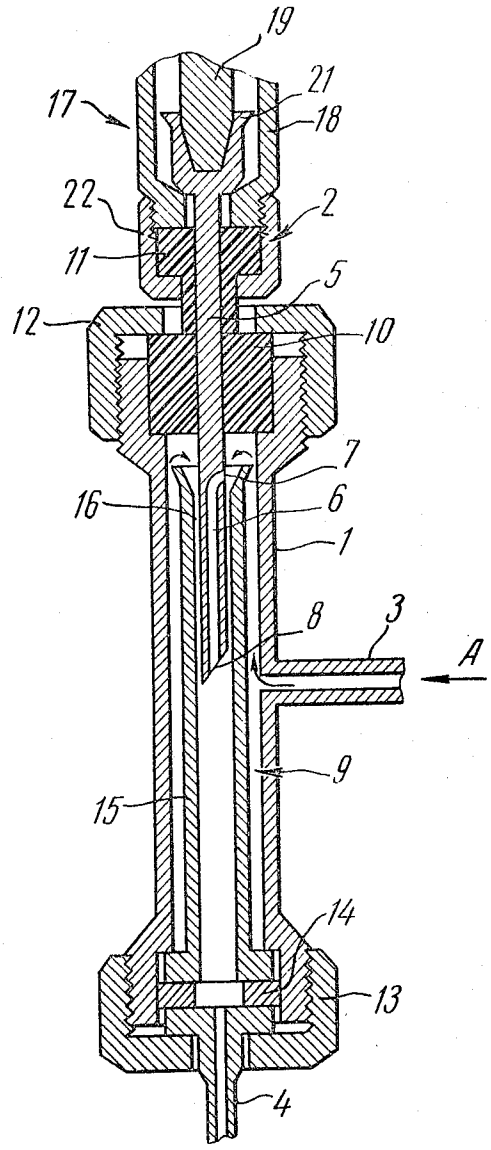

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FIG. 1

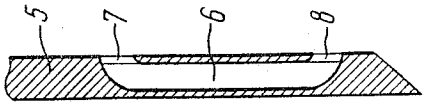


FIG. 6

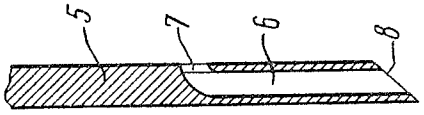


FIG. 4

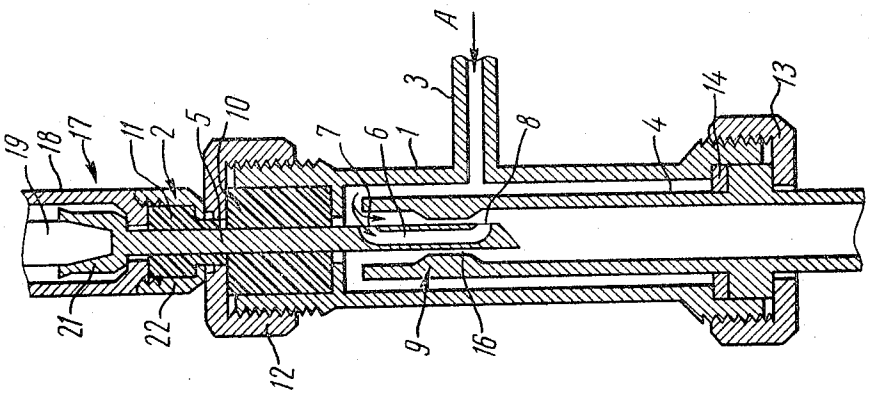


FIG. 3

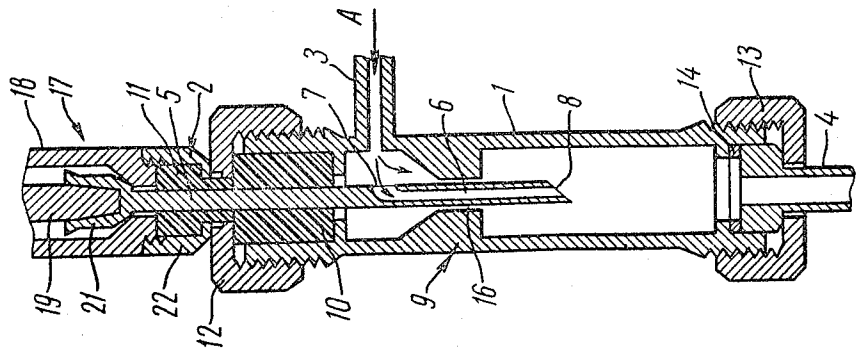


FIG. 2

APPARATUS FOR INJECTING A SAMPLE INTO A GAS CHROMATOGRAPH

BACKGROUND OF THE INVENTION

1. Field of the Invention

The present invention relates to gas chromatography, and, more particularly, it relates to apparatus for injecting a sample into a gas chromatograph, for instance, in cases when a small quantity (from 0.1 μ to several μ) of a liquid or solid sample is to be introduced into a gas chromatograph. The invention can be employed in chemical, petrochemical and food industries, as also in medicine, biology, space research and elsewhere, when the composition of various gases, liquids and solids is to be investigated.

2. Description of Prior Art

There are widely known apparatus for injecting a sample into a gas chromatograph, including a sample evaporation chamber at the end of which there is mounted a septum made of an elastic material, sealing the chamber from the ambient atmosphere. The needle of a microsyringe filled with a sample is introduced into the sample evaporation chamber by displacing the rod of the plunger of the microsyringe. The needle enters the chamber by piercing the septum.

However, despite the wide application of these known apparatus for injecting a sample into a gas chromatograph, they are not free from serious disadvantages.

One of these disadvantages is the presence of the dead volume in the microsyringes employed. In microsyringes of a volume in excess of 10 μ the dead volume is the volume of the needle. In microsyringes of a 1.0 μ and 0.5 μ volume the dead volume is the volume of the space between the internal wall of the needle and the tungsten wire received inside the needle and acting as the plunger. The dead volume is the source of relatively poor reproduction of the analysis performed, particularly when samples less than 1.0 μ in volume are injected.

The presence of the dead volume in the microsyringes employed also brings about the necessity of flushing the syringes to remove the remnants of a sample, such flushing involving removal of the precisely ground-in plungers from their cylinders, which results in excessive wear of the microsyringes and in poor sealing.

Reliable performance of the microsyringes is ensured only when the samples injected with their help are non-aggressive and are not subject to polymerization, decomposition, etc. under the action of elevated temperatures, oxygen and moisture in the ambient air.

With the contacting parts of the microsyringes becoming coated with the products of corrosion, polymerization and decomposition, the syringes are doomed for quick failure.

The abovedescribed disadvantages are obviated in another prior art apparatus for injecting a sample into a gas chromatograph, the apparatus including a sample-injecting pipette having an internal axial capillary passage for accommodation of the sample, the passage having an inlet and an outlet; said inlet is made through an external side surface of the pipette, the pipette being reciprocable in the sample evaporation chamber adjacent to one of the ends thereof, the chamber is hermetically sealed by sealing means and communicates, on the other hand, with a source of the carrier gas of the

associated gas chromatograph, and, on the other hand, with the chromatographic column of this gas chromatograph, the chamber incorporating a member for guiding the carrier gas into the capillary passage of the pipette.

In this known apparatus the sealing means is in the form of a rubber ring with a nut, disposed on an end face of the chamber on the side of the pipette, and a tap disposed within the chamber downwardly from the rubber ring and providing passage of the pipette to the member guiding the carrier gas into the capillary passage of the pipette. The member for guiding the carrier gas into the capillary passage of the pipette is in the form of a seat reducing the cross-sectional area of the sample evaporation chamber. Said member is positioned within the chamber downwardly from the sealing means and from the connection through which the chamber communicates with the carrier gas supply source. The pipette is made of glass, the outlet of the capillary passage thereof being arranged centrally of the pointed end of the pipette, the pipette being so positionable in the seat that the inlet is above the seat and the outlet is beneath it.

However, in this known apparatus also a quantity of ambient air finds its way into the chromatographic column of the gas chromatograph, each time a sample is metered thereinto. This results from the fact that the sealing means include a tap valve and a rubber ring with a nut. When the pipette is removed, the internal space of the sample evaporation chamber intermediate of the tap valve and the rubber ring communicates with the ambient atmosphere. As the pipette is being introduced, the chamber becomes sealed from the ambient atmosphere by the rubber ring with the nut, the tap valve is opened, and the air filling the abovedefined space is forced by the pipette into the chromatographic column.

There are numerous cases when access of ambient air into the chromatographic column is undesirable. The air may contain substances which, particularly when employing highly responsive detectors in the gas chromatograph, might become registered in the chromatogram, and this would result in faulty evaluation of the composition of the sample.

Besides, with the oxygen of the air getting into the chromatographic column including a liquid phase, the phase itself might be oxydized, or else certain components of the sample might be oxydized.

However, the abovedescribed known apparatus can be operated in a manner preventing the access of the ambient air into the chromatographic column, this being achieved by interrupting the flow of the carrier gas for flushing the air from the space between the tap valve and the rubber ring. Such interruption of the flow of the carrier gas through the chromatographic column might lead, when employing a hot-wire detector in the gas chromatograph, to failure of the wires of the detector at high values of working-current; on the other hand, the resumption of the flow of the carrier gas increases the time of the analysis, particularly, when the chromatograph is associated with an automatic gas flow control.

Furthermore, the pipette itself having a volume with the volume of the carrier gas supplied within a unit of time into the sample evaporation chamber, the introduction of the pipette into the chamber brings about a pressure pulse in the gas supply, which affects the uni-

formity of the gas flow through the chromatographic column.

Such variation of the flow rate of the carrier gas yields poorer reproduction of the outcome of the analysis, variation of the sample component retention data, which might lead to obtaining false information concerning the quantitative and qualitative composition of a mixture under test.

Therefore, variations of the parameters of the carrier gas flow through the chromatographic column and access of the ambient air thereinto at injection of a sample with the help of the abovedescribed known apparatus are among the reasons why such prior art apparatus has not found a wide field of applications.

SUMMARY OF THE INVENTION

It is an object of the present invention to provide an apparatus for injecting a sample into a gas chromatograph, which ensures permanency of the established working rate of flow of the carrier gas through a chromatographic column of the gas chromatograph.

It is another object of the present invention to provide an apparatus for injecting a sample into a gas chromatograph, which prevents access of the ambient air into the chromatographic column of this gas chromatograph.

These and other objects are attained in an apparatus for injecting a sample into a gas chromatograph, wherein the sample injecting pipette has an internal axial capillary passage adapted to accommodate therein a sample the pipette having an inlet and an outlet and a needle, said inlet being made through the external side surface of said needle, the needle being arranged for reciprocation within a sample evaporation chamber on a side of one end of said chamber, said chamber being hermetically sealed from the ambient air by sealing means the chamber communicates, on the one hand, with a supply source of the carrier gas of the associated gas chromatograph, and, on the other hand, with the chromatographic column of said gas chromatograph, said chamber incorporating a member for guiding said carrier gas into said capillary passage of said pipette further, in the present apparatus, in accordance with the invention, said sealing means includes a septum made of an elastic material, arranged in the end portion of said sample evaporation chamber, on the side of the introduction of said pipette, the pipette comprising a needle made of a material which can pierce said septum, as a sample is being injected into said sample evaporation chamber; said sealing means further includes a sleeve enveloping the external surface of the needle, so that during reciprocation of the needle said sleeve closes said inlet of said capillary passage of the needle, as said passage passes through said septum, said sleeve being adapted to engage said septum, as said inlet of said capillary passage comes out from said sleeve, in which position said sleeve provides sealing of said apparatus.

Said member for guiding the carrier gas into the capillary passage of said needle may be in the form of a tube arranged within said sample evaporation chamber, so that a capillary annular space is left intermediate of the external surface of said needle and the internal surface of said tube, one end of said tube being mounted adjacent to said chromatographic column, establishing communication between said column and the internal space of said tube, the opposite end of said tube facing said septum, said inlet of said capillary passage of said needle being positioned above said opposite end of said tube, facing said septum.

Alternatively, it is advisable that said member for guiding said carrier gas into said capillary passage of said needle should be in the form of an annular shoulder arranged interiorly of said sample evaporation chamber, so that said inlet of said capillary passage of said needle should be positioned above said annular shoulder, and said outlet of said passage should be positioned beneath said shoulder, there being left a capillary annular space intermediate of the external surface of said needle and said annular shoulder.

It is advisable that said annular shoulder should be arranged on the internal wall of said sample evaporation chamber.

It is also possible to have a tubular member mounted within said sample evaporation chamber, coaxially therewith, said tubular member communicating with said chromatographic column, one of the ends of said tubular member facing said septum, said annular shoulder being arranged on the internal surface of said tubular member.

Preferably, said tubular member is a part of said chromatographic column of said gas chromatograph.

It is advisable that said needle should be associated with a holder assembly including a housing having mounted therein a reciprocable rod connected with said needle, said sleeve being mounted at that end portion of said housing, where said needle projects therefrom.

Said outlet of said capillary passage of said needle may be made through the external wall surface of said needle, beneath said inlet of said passage.

The herein disclosed apparatus for injecting a sample into a gas chromatograph provides for maintaining an established working rate of flow of the carrier gas through the chromatographic column of a gas chromatograph, simultaneously preventing access of ambient air thereinto. It offers reproducible results of sample component retention data, thus improving the accuracy of analysis, as compared with the known prior art devices.

BRIEF DESCRIPTIONS OF THE DRAWING

The present invention will be further described in connection with several embodiments thereof, with reference being had to the appended drawings, wherein:

FIG. 1 illustrates an axially sectional view of an apparatus for injecting a sample into a gas chromatograph, embodying the invention, with the member for guiding the carrier gas into the capillary passage of the needle in the form of a tube;

FIG. 2 is an axially sectional view of the second embodiment of the invention, with the member for guiding the carrier gas into the capillary passage of the needle in the form of an annular shoulder;

FIG. 3 is an axially sectional view of the third embodiment of the invention, with the member for guiding the carrier gas into the capillary passage of the needle in the form of an annular shoulder on the internal wall of the chromatographic column of the gas chromatograph;

FIG. 4 is a longitudinally sectional view of the needle of an apparatus for injecting a sample into a gas chromatograph, embodying the invention;

FIG. 5 is a longitudinally sectional view of the needle of an apparatus for injecting a sample into a gas chromatograph, embodying the invention, associated with a needle holder assembly;

FIG. 6 is a longitudinally sectional view of an alternative embodiment of the needle of an apparatus for injecting a sample into a gas chromatograph, constructed in accordance with the invention.

DESCRIPTION OF PREFERRED EMBODIMENTS

An apparatus for injecting a sample into a gas chromatograph, constructed in accordance with the present invention, is intended for injecting of liquid and solid samples having their volume from 0.1 μl to several μl.

According to the invention, the apparatus in each one of the embodiments described hereinbelow comprises a cylindrical sample evaporation chamber **1** (FIGS. **1**, **2** and **3**) which is hermetically sealed from the ambient air by sealing means **2**. The chamber **1** communicates, on the one hand, with the supply source of the carrier gas of the gas chromatograph (not shown in the appended drawings) through a connection tube **3**, and, on the other hand, it communicates adjacent to one end thereof with the chromatographic column **4** of the chromatograph.

At the other end of the sample evaporation chamber **1** there is slidably and reciprocably mounted thereinside a sample- injecting pipette, or syringe including a needle **5** having an axial capillary passage **6** for accommodation of a sample, the passage **6** having an inlet **7** and an outlet **8**, the inlet **7** being made through the side wall of the needle **5**. The sample evaporation chamber **1** incorporates a member **9** for guiding the carrier gas into the capillary passage **6** of the needle **5**.

The sealing means **2** in the presently described apparatus includes a septum **10** made of an elastic material, e.g. of silicone rubber, and mounted in the end face of the sample evaporation chamber **1** on the side of the needle **5**, and a sleeve **11** also made of an elastic material, e.g. of silicone rubber, sealingly enveloping the external wall of the needle **5**, so that during reciprocation of the needle **5** the sleeve **11** closes the inlet **7** of the capillary passage **6**; further, as the capillary **6** passes within the septum **10**, in the engagement position of the herein disclosed apparatus, said engagement position being the position illustrated in the appended drawings FIGS. **1** to **3**, when the inlet **7** of the capillary passage **6** is outside the sleeve **11**, the latter effects sealing of the apparatus. The needle **5** is made of a material which can pierce the septum **10**, as a sample is being injected into the sample evaporation chamber **1**. In the presently described embodiments of the invention the needle **5** is made of stainless steel. The septum **10** is mounted in the end face of the sample evaporation chamber **1** with the help of a cup-shaped external nut **12**. The chamber **1** is secured to the chromatographic column **4** by mounting means including a nut **13** and a gasket **14**.

As it has been already stated hereinabove, all the abovedescribed features of the apparatus for injecting a sample into a gas chromatograph, constructed in accordance with the invention, are common to all the embodiments of the invention described in the present specification.

Each one of the several preferred embodiments of the invention will be described individually hereinbelow, as far as its specific features are concerned.

Thus, in accordance with the first embodiment (FIG, **1**) which is the simplest in manufacture, the member **9** for guiding the carrier gas into the capillary passage **6** of the needle **5** is in the form of a tube **15** mounted interiorly of the sample evaporation chamber **1** so that an annular capillary space is left intermediate of the external wall of the needle **5** and the internal wall of the tube **15**. The respective end of the tube **15** is secured with the help of the gasket **14** and the nut **13** adjacent to the chromatographic column **4**, so that this open end establishes communication between the column **4** and the internal space of the tube **15**. The opposite end of the tube **15** is facing the septum **10**, so that in the fully introduced position of the needle **5** the inlet **7** of the capillary passage **6** of the needle is positioned directly above the end of the tube **15**, facing the septum **10**. In this embodiment of the invention the outlet **8** (FIGS. **1** and **4**) of the capillary passage **6** of the needle **5** is at a pointed end of the needle **5**, while the inlet **7**, as it has been already described, is made through the external surface of the needle **5**.

To facilitate handling of the apparatus constructed in accordance with the first embodiment of the present invention, the needle **5** (FIGS. **1** and **5**) is mounted in a holder **17**.

The holder **17** (FIG. **5**) includes a housing **18** made up by two separable parts. Mounted for reciprocation inside the housing **18** is a rod **19** associated with a spring **20** biasing the rod from the housing **18**. The needle **5** is secured to the rod **19** by a cup-shaped connection piece **21**. The sleeve **11** is mounted on the end of the housing **18**, through which the needle **5** passes, with the help of a nut **22**. The stroke of the rod **19** should be somewhat greater than the axial height of the septum **10** plus half the axial height of the sleeve **11**. The rod **19** is retained in the housing **18** by means of another cup-shaped nut **23**.

According to the second embodiment of the present invention, the inlet **8** (FIGS **2**, **4** and **5**) of the capillary passage **6** of the needle **5** is arranged in the same manner as in the first embodiment described hereinabove. The same is true of the outlet **7** and of the mounting of the needle **5** in the holder **17**.

However, in the second embodiment the member **9** (FIG. **2**) for guiding the carrier gas into the capillary passage **6** of the needle **5** is in the form of an annular shoulder disposed within the sample evaporation chamber **1**, on the internal wall thereof, so that the inlet **7** of the capillary passage **6** of the needle **5** is above this annular shoulder, the outlet **8** of the passage **6** is beneath this shoulder, and an annular capillary space **16** is left intermediate of the external side wall of the needle **5** and the annular shoulder.

In the third embodiment of the present invention the outlet **8** (FIGS **3** and **6**) of the capillary passage **6** of the needle **5** is arranged similarly to the inlet **7**, i.e. it is arranged in the external side wall of the needle **5**, downwardly from the inlet **7**. Both the inlet **7** and the outlet **8** of the capillary passage **6** being thus made through the side wall of the needle **5**, there is prevented any possibility of the passage **6** becoming clogged with the material of the septum **10**, as the needle **5** is driven to pierce this septum.

In this embodiment the needle **5** (FIG. **3**) is mounted in the holder **17** in the manner similar to that of the first and second embodiments described hereinabove.

Further, in this embodiment a tubular member is mounted interiorly of the sample evaporation chamber **1**, coaxially therewith. This tubular member of the presently described embodiment is a part of the chromatographic column **4** of the associated gas chromatograph. The respective end of the column **4** is facing the septum **10**. The member **9** for guiding the carrier gas into the capillary passage **6** of the needle **5** is in the form of an annular shoulder provided on the internal wall of the chromatographic column **4**. The positioning of this shoulder is similar to that of the annular shoulder described hereinabove in connection with the second embodiment of the invention.

The principle of operation of the herein disclosed apparatus for injecting a sample into a gas chromatograph is the same for each one of the three embodiments described hereinabove. Therefore, it will be sufficient to describe the operation of the apparatus in connection with the first one of the three embodiments described hereinabove, the operation being that of injection of a liquid sample.

Prior to introduction of the needle **5** into the sample evaporation chamber **1**, a sample is collected in the capillary passage **6** of the needle **5**. The rod **19** of the holder **17** is depressed downwardly as far as it will go, for the needle **5** to be driven downwardly so that the inlet **7** of the passage **6** thereof should leave the sleeve **11** and thus become open and accessible from outside. The pointed end of the needle **5** is dipped slightly into the liquid sample, so that the inlet **7** should be well above the liquid level, and the capillary forces make the liquid fill completely the passage **6**. When the rod **19** is released, the needle **5** with passage **6** is returned into the initial position thereof under spring-action, in which the inlet **7** of the needle **5** is closed within the sleeve **11**.

Thereafter the needle **5** with the sample carried thereby are introduced into the sample evaporation chamber, into which the carrier gas is fed continuously through the supply connection **3** in the direction indicated by the arrow line "A." The carrier gas finds its way through the tube **15** into the chromatographic column **4** (small arrow lines inficate the direction of the flow of the carrier gas within the chamber **1** in the appended drawings).

The needle **5** is introduced into the chamber **1** by the needle being driven downwardly to pierce the septum **10**, this downward motion of the needle being continued until the septum **10** engages the sleeve **11**.

This done, the rod **19** is depressed, and the needle **5** is so positioned within the sample evaporation chamber **1**, that the inlet **7** of the passage **6** of the needle **5** is above the adjacent end of the tube **15**, the needle **5** obstructing partly the access of the carrier gas into the tube **15**. The cross-sectional flow passage area of the annular capillary space **16** intermediate of the external side wall of the needle **5** and the internal wall of the tube **15** being substantially commensurate with the cross-sectional area of the capillary passage **6** of the needle **5**, the flow of the carrier gas into the tube **15** is divided into two parts, one of them passing through the passage **6** of the needle **5**, forcing the sample collected therein through the outlet **8** into the tube **15**, wherein the sample evaporates and is carried by the carrier gas into the chromatographic column **4**; the other one of the two parts finds its way into the column **4** through the annular capillary space **16**.

Then the rod **19** is released, and the needle **5** is returned under spring action into the initial position. Thereafter the needle **5** is taken out from the evaporation chamber **1**.

The needle **5** is now ready for collecting another sample, no flushing being needed for the purpose.

As it has been already stated, the operation of the herein disclosed apparatus constructed in accordance with the two other embodiments is similar to the operation of the apparatus constructed in accordance with the first embodiment.

The herein disclosed apparatus fro injecting a sample into a gas chromatograph can be also employed for injection of solid samples. In this case the sample is first reduced to a molten state. Then the pointed end of the needle is dipped into the molten sample. With the needle being adequately heated, the passage therein is filled with the molten sample under the action of capillary forces. The sample thus collected solidifies, after the needle has been removed from the molten sample. As the needle is subsequently introduced into the sample evaporation chamber, under high temperature melting the sample, the carrier gas forces the sample out from the capillary passage, as has been already described.

Viscous samples, as well as water solutions are collected in the capillary passage of the needle under the action of pressure applied to the surface of the liquid into which the pointed end of the needle is dipped, while the other opening of the passage of the needle outside the zone of this pressure. The same technique is used to fill the capillary passage with mercury, when it is necessary to gauge the volume of the passage.

The incorporation of the movable sleeve made from elastic material in the herein disclosed apparatus for introducing a sample into a gas chromatograph ensures that the inlet of the capillary passage of the needle is closed when the sample is being introduced, whereby it becomes possible to perform injection of the sample by driving the needle through septum of the sample evaporation chamber.

The herein disclosed apparatus for injecting a sample into a gas chromatograph (the sample volume being from 0.1 μ to several μ) ensures repetition error not in excess of ± 1 percent. The apparatus can be associated with any kind of a gas chromatograph; the structure of the apparatus provides for maintaining uninterrupted flow of the carrier gas through the chromatographic column and positively prevents any access of air into the chromatographic. It further provides for reproduction of the results of substance retention data, thus stepping up the accuracy of an analysis.

What is claimed is:

1. An apparatus for injecting a sample into a gas chromatograph having a chromatographic column and using a carrier gas, including: a chamber for evaporation of said sample, communicating with and having a supply source of a carrier gas of said gas chromatograph, said chamber being connected with the chromatographic column of said gas chromatograph; a sample-injecting pipette arranged for reciprocation within said sample evaporation chamber, said pipette being disposed adjacent to one end of the chamber, said pipette being in the form of a needle provided with means enabling reciprocatory longitudinal motion thereof; an axial internal capillary passage of said needle provided to accommodate said sample thereinside, said passage

having an inlet and an outlet, said inlet being formed through an external side wall of said needle; a guide member positioned interiorly of said sample evaporation chamber for guiding said carrier gas into the inlet of said capillary passage of said needle; sealing means hermetically sealing said sample evaporation chamber from ambient atmosphere; a septum member of said sealing means, said septum member being made from an elastic material and being mounted in the end portion of said sample evaporation chamber, on the side thereof where said needle is to be inserted and positioned thereinside; a sleeve member forming part of said sealing means and enveloping the external side wall of said needle; said needle being made of a material which can pierce said septum member as said sample is being injected into said sample evaporation chamber, said sleeve enveloping said external side wall of said needle so that during reciprocation of said needle the slleve closes said inlet of said capillary passage of said needle, and, when the needle is passing through said septum member, said sleeve member engages said septum member, said inlet of the needle emerges from said sleeve member towards the guide member, said sleeve member ensuring sealing of said apparatus.

2. An apparatus according to claim **1,** wherein said member for guiding said carrier gas into said capillary passage of said needle includes a tube arranged within said sample evaporation chamber, so that there is formed an annular capillary space intermediate of the external surface of said needle and the internal wall of said tube, one end of said tube being mounted adjacent to said chromatographic column thereby establishing communication between the internal space of said tube with said chromatographic column, the opposite end of said tube facing said septum member, said inlet of said capillary passage of said needle being positioned above said opposite end of said tube, facing said septum member.

3. An apparatus according to claim **1,** wherein said member for guiding said carrier gas into said capillary passage of said needle includes an annular shoulder arranged within said sample evaporation chamber, so that said inlet of said capillary passage of said needle is positioned above said annular shoulder, said outlet of said capillary passage being positioned beneath said shoulder, there being formed an annular capillary space intermediate of the external surface of said needle and said annular shoulder.

4. An apparatus according to claim **3,** wherein said annular shoulder is formed on the internal wall of said sample evaporation chamber.

5. An apparatus according to claim **3,** wherein there is mounted interiorly of said sample evaporation chamber and coaxially therewith a tubular member communicating with said chromatographic column, one end of said tubular member facing said septum member, said annular shoulder being formed on the internal wall of said tubular member.

6. An apparatus according to claim **5,** wherein said tubular member is an integral part of said chromatographic column of said gas chromatograph.

7. An apparatus according to claim **1,** wherein said needle is associated with a holder assembly including a housing having reciprocably mounted therein a rod connected with said needle, said sleeve being mounted at the end portion of said housing, adjacent to said needle projecting therefrom.

8. An apparatus according to claim **1** wherein said outlet of said capillary passage of said needle is made through an external side wall of said needle, beneath said inlet of said capillary passage.

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