Oct. 20, 1970 W. T. SWEENEY 3,534,945

INJECTION SYSTEM

Filed July 3, 1968 2 Sheets-Sheet 1

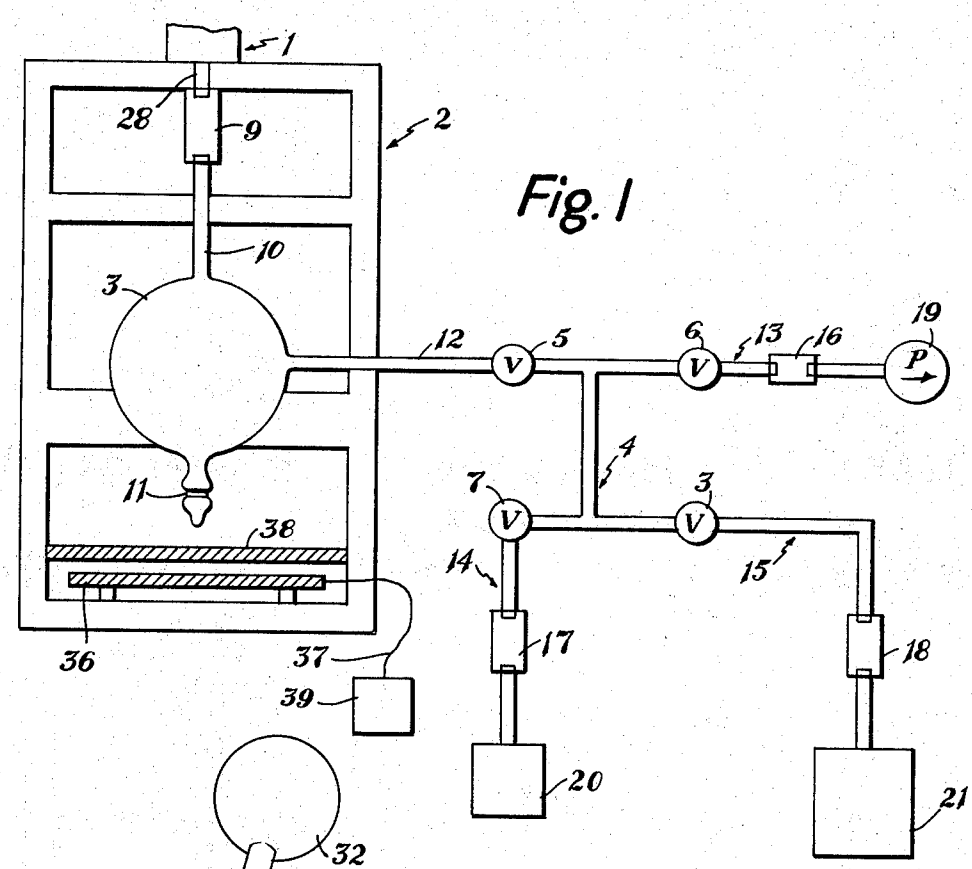


Fig. 1

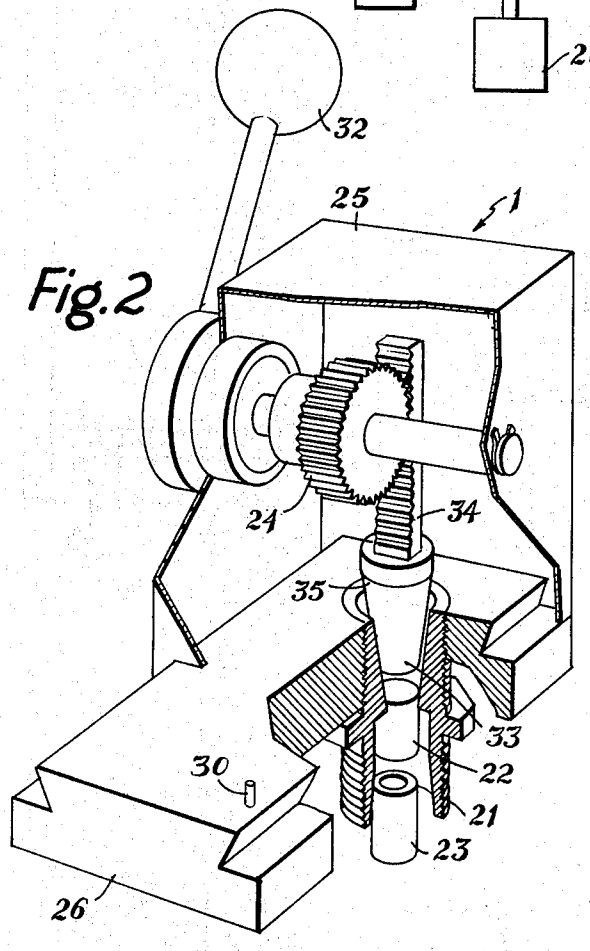


Fig. 2

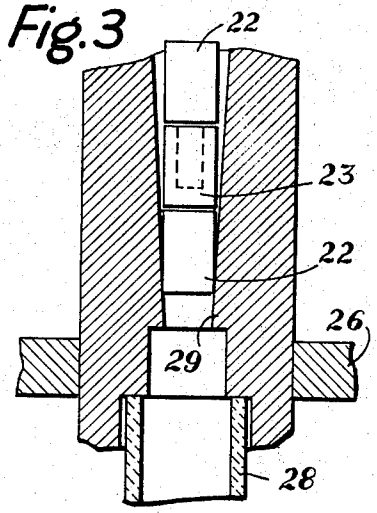


Fig. 3

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INJECTION SYSTEM

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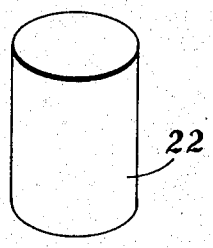


Fig. 4

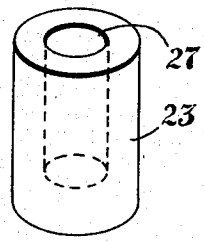


Fig. 5

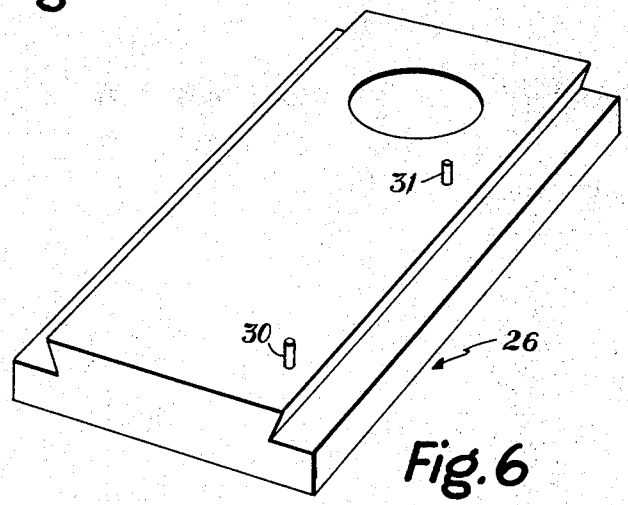


Fig. 6

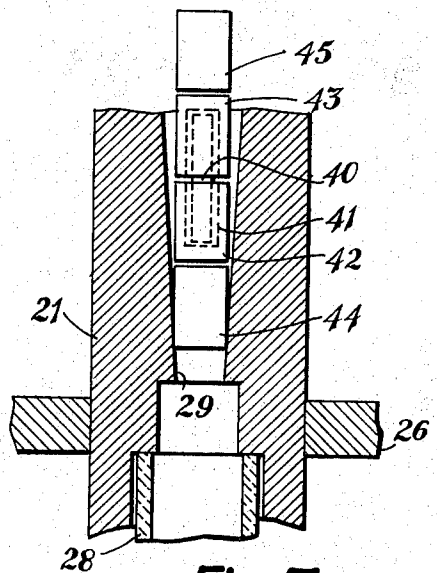


Fig. 7

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3,534,945

INJECTION SYSTEM

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U.S. Cl. 263—1 8 Claims

ABSTRACT OF THE DISCLOSURE

Small amounts of material are introduced into a vacuum-tight enclosure by an enjection system which makes use of a series of slugs, some of which have cavities for receipt of the material to be introduced into the enclosure, and others of which are solid. The slugs are introduced into the enclosure in series through a channel which is just wide enough to permit the slugs to move through it, but which is narrow enough so that pressure between the outer surfaces of the slugs and the inner surface of the channel maintains a vacuum-tight seal. Each cavitied slug containing material to be introduced is flanked by solid slugs in order to preserve the vacuum-tight property of the enclosure.

BACKGROUND OF THE INVENTION

Field of the invention

The invention relates to vacuum-tight systems, and in particular to an injection mechanism for introducing material into a vacuum-tight enclosure, such as the evacuated region associated with a mass spectrometer. However, the invention is not limited to injection into an evacuated region, but includes injection into a pressurized region or indeed injection into an enclosure at atmospheric pressure.

BACKGROUND OF THE INVENTION

Description of the prior art

Many analytical and research instruments used to study organic materials required the sample to be in the vapor phase. Such instruments may operate under low or high static pressures or under flow conditions. Whatever the operating conditions, the introduction of the sample itself should not significantly disturb the operation of the instrument. For example, a mass spectrometer commonly operates at 10^{-6} mm. Hg pressure. Thus during sample introduction the system must not be exposed to atmospheric pressure, for the resultant pressure surge could cause significant damage.

In cases where the sample is naturally in the liquid phase, an inlet system commonly used for a mass spectrometer includes a bulb connected by one line to a vacuum pump and by another line to the ion source of the mass spectrometer. Vapor flow through each line is controlled by means of a valve therein. By means of these valves, the bulb is sealed off from the ion source, and evacuated by means of the vacuum pump. After evacuation of the bulb, the sample in liquid phase is introduced into the evacuated bulb by means of a syringe passed through a rubber membrane or septum connected in some manner to the bulb. The bulb is then heated, so as to vaporize the liquid sample in the bulb. The valve in the line from the bulb to the ion source is then opened, in order to permit the sample to flow in vapor phase towards the ion source. In the line joining the bulb to the ion

source, some type of porous plug or diaphragm with a small hole in it is used to allow the sample in vapor phase to leak at a known rate into the ion source.

This technique suffers from the disadvantage that heat and certain chemicals will decompose the septum or harden it, with the result that either the syringe will not penetrate it or, upon removal, the septum material will not seal up the puncture, thereby causing an air leak. Further, all injected materials must be in the liquid state to be handled by the syringe. Thus a material which is a solid at room temperature must be dissolved in some solvent and then injected. The solvent may inhibit the performance of the instrument. Also, not all materials are soluble in the commonly available solvents.

In the above described arrangement, as in the case of the invention, the sample is introduced directly into the reservoir in nongaseous phase in such a way as not to impair the vacuum, and the sample is vaporized in the reservoir itself. The reservoir is needed in order to provide a controlled supply of vaporous sample and therefore is rather closely connected to the ultimate region of use, such as the ion source of the mass spectrometer, and in general is kept evacuated to about the same degree of vacuum as that in the ultimate region of use.

An alternate system has been used in the prior art in the case of solid samples. In this system, the sample is not introduced directly into the reservoir in the nongaseous phase; instead, the evaporation chamber is separated from the reservoir and is sealed off therefrom by one or more valves until the evaporated chamber has been evacuated and the sample subsequently evaporated. The evaporation chamber may be relatively small, and the sample in solid phase is introduced into the evaporation chamber at atmospheric pressure, without any effort to preserve a vacuum therein. The evaporation chamber is evacuated repeatedly for each new sample. Such a device is generally known as a "hot-box." Both the reservoir and the small evaporation chamber are inside an oven which provides the heat necessary to evaporate the solid sample. One end of a glass neck is connected to the reservoir by means of a valve or stop-cock. At the other end of the glass neck there is a ground-glass joint (either male or female), to which the small evaporation chamber is removably attached after the solid sample has been introduced therein. The air is then pumped away from the evacuation chamber via a suitable connection in the glass neck. After evacuation, the evacuation chamber and the reservoir are heated by means of the oven. When sufficiently hot to generate a vapor pressure of the solid, the valve or stop-cock is opened to allow the vapor into the reservoir. Thus the solid sample is vaporized before it is introduced into the reservoir by providing a small, readily removable evaporation chamber closely connected to the reservoir and heated in a common oven with the reservoir. The portion of the oven housing the small evaporation chamber may be a moveable section which swings out of the way in order to provide ready access to the small evaporation chamber.

This so-called "hot-box" technique is time consuming, has sample contamination problems associated with it, presents difficulty in maintaining the reservoir and small evaporation chamber at equal temperatures while heated by the oven, and is more prone to breakage than the system of the invention. As previously noted, in the system of the invention the solid itself is introduced into the reservoir before evaporation.

SUMMARY

The slug injection system of the invention overcomes all the problems of the prior art hereinabove referred to. In the particular embodiment of the invention described herein (but to which the invention is not limited), namely, its use in connection with a mass spectrometer, the injection mechanism itself is utilized in conjunction with two other main components, namely, an oven and glassware. The glassware includes a glass bulb, manifold, and valves. The injection system itself comprises four main units: (1) slugs; (2) block; (3) baseplate; and (4) injection sleeve. Some of the slugs are solid, and some have cavities for reception of small amounts of material to be injected. All the slugs are adapted to slide through the injection sleeve snugly, and at least one solid slug is in the injection sleeve at all times in order to render the injection sleeve vacuum-tight. The material to be injected is introduced into the cavity of a cavitied slug, and that slug is then introduced into the glass bulb along with its cargo via the injection sleeve, flanked by solid slugs in order to preserve vacuum-tightness at all times. Once in the glass bulb, the material may be vaporized by means of the oven, and the vapor transported to some other region, such as the ion source of a mass spectrometer, via the manifold. The block and base-plate cooperate to provide the mechanism for pushing the slugs through the injection sleeve.

BRIEF DESCRIPTION OF THE DRAWING

The invention may best understood from the following detailed description thereof, having reference to the accompanying drawings, in which:

FIG. 1 is a schematic diagram of an all-glass heated inlet system incorporating one embodiment of the invention;

FIG. 2 is an isometric view of the injection system used in the apparatus of FIG. 1;

FIG. 3 is a longitudinal central section through the injection sleeve of the injection system of FIG. 2;

FIG. 4 is a detail of the view of FIG. 3 and shows a solid slug;

FIG. 5 is another detail of the injection sleeve of FIG. 3 and shows a cavitied slug;

FIG. 6 is a view showing the base plate used in connection with the injection sleeve of FIG. 3; and

FIG. 7 is a view similar to that of FIG. 3 and showing a modification of the embodiment shown in FIG. 3.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to the drawings, the first to FIG. 1 thereof, the total inlet system consists of three main components: namely, the injection mechanism **1**, an oven **2** and glassware. The glassware includes a glass bulb or reservoir **3**, a manifold **4**, and valves **5**, **6**, **7** and **8**. Although the system will be described as having four valves, it will be apparent from the ensuing description that the total inlet system will work with a minimum of three valves.

The oven **2** provides structural support for the injection mechanism **1** and the glassware. It also provides a controlled temperature environment for vaporization of the sample.

The reservoir **3**, within which the sample is vaporized by the heat generated by the oven **2**, is connected by means of suitable tubing **9** and glass **10** to the injection mechanism **1**. Samples to be vaporized are introduced into the reservoir **3** by the injection mechanism **1** to be described in detail hereinafter. At the bottom of the reservoir **3** there is an O-ring connection **11**, which when opened allows access to the contents of the reservoir **3**. The glass bulb reservoir **3** includes an arm **12** which terminates in a glass valve **5** attached to the manifold **4** Additional glass valves **6**, **7**, **8** are attached to the manifold **4** and are connected to external lines **13**, **14**, **15**, respectively, each line including a length of suitable tubing **16**, **17**, and **18**. The external lines **13**, **14**, **15** lead to a vacuum pump **19**, an external inlet system **20**, and an ion source **21** of a mass spectrometer, respectively. In all cases the tubing **9**, **16**, **17**, **18** preferably consists of flexible, heat-shrinkable Teflon tubing, where Teflon is the trade name owned by E. I. du Pont de Nemours and Co., Inc. for tetrafluoroethylene and fluorinated ethylpropylene resins. The use of shrinkable Teflon tubing as joints affords a cushion against both physical and thermal shock. In addition, the tubing may be easily cut so that parts may be removed or changes made.

The valves **5**, **6**, **7**, **8** may consist of a glass ball and socket joint in which the ball is connected to a glass rod containing a magnet. The valve is opened or closed by the action of an external magnet controlled manually by the operator.

The operation of the device shown in FIG. 1 will now be described starting from the point in the cycle at which all valves are closed and the bulb has been evacuated. A sample in liquid or solid phase is injected into the reservoir **3** via the injection mechanism **1** of the invention, and the heat from the oven **2** causes it to vaporize. The valve **5** is then opened, allowing the vaporized sample to enter the manifold **4**. Valve **8** is then opened, and the vaporized sample enters the ion source **21** of the mass spectrometer. After conclusion of the experiment with the mass spectrometer, the entire system may be evacuated by opening valve **6**. After evacuation, valve **5** which connects the reservoir **3** to the manifold **4**, is closed and the reservoir **3** is then ready for injection of another sample. Valve **7** may be used to connect the manifold to any other external inlet system **20** such as a gas inlet system.

The oven **2** may be heated by any of a number of conventional techniques. One such technique makes use of two strip heaters, shown at **36** in FIG. 1, each of which comprises a bar which may be, for example, 8 inches long with a cross section of ¼-inch by ¾-inch. Each bar **36** is a resistance heater, and the current thereto is supplied via leads **37** which may pass through the bottom or sides of the oven **2**. The strip heaters **36** are supported at the bottom section of the oven **2**, and a heat shield **38** is supported above the strip heaters **36** in order to prevent hot spots and thus protect the reservoir **3**. The leads **37** are connected to an external controller **39** which varies the current so as to control the temperature within the oven **2**.

The apparatus shown in FIG. 1 has been described in order to illustrate one possible use for the injection mechanism of the invention. However, it is clearly to be understood that the injection mechanism of the invention is not limited in its usefulness to operation with a mass spectrometer, but will be useful whenever it is desired to transfer liquid or solid samples into an enclosed region, particularly from another region at a pressure different from that existing within the enclosed region.

The injection mechanism of the invention is shown in FIGS. 2 and 3. Referring thereto, the injection mechanism **1** essentially includes an injection sleeve **21** through which a sequence of slugs **22**, **23** is forced by means of a suitable mechanical contrivance such as the rack and pinion gear mechanism shown in FIG. 2. This mechanism **24** is supported on a block **25** which in turn slides on a base plate **26** so as to move the rack and pinion gear mechanism **24** into or out of alignment with the injection sleeve **21**.

The slugs are of two different types, solid and cavitied. A solid slug **22** is shown in FIG. 4 and preferably comprises a solid gas free Teflon cylinder. A representative dimension is 0.250 inch long (plus .025 inch, minus .000 inch) and 0.188 inch in diameter (plus or minus .002 inch). A cavitied slug **23** is shown in FIG. 5; it also preferably comprises a (cavitied) gas free Teflon cylinder and has the same external dimensions as the solid slug **22** but contains a cavity **27**. For the dimensions of

the solid slug previously described, a representative cavity dimension would be .098 inch in diameter (plus or minus .002 inch) and .188 inch deep. The cavitied slug **23** is used to hold the sample. One reason that Teflon is a preferred material for the slugs is that it will flow a very small amount under pressure. Thus if there are any scratches on the surface of the injection sleeve, the slugs will plug them up.

Referring now to FIG. 3, the injection sleeve **21** is supported on the base plate **26** and is aligned to engage the glass line **28** to the reservoir **3**. The sleeve **21** screws into the bottom of the base plate **26** which is fixed to the top of the oven **2**. A cavitied slug **23** containing the sample is inserted into the glass reservoir through the injection sleeve **21**. However, during intervals between injections, the injection sleeve **21** has a solid slug **22** in the tapered neck **29**. This neck **29** is shaped so that no slug will pass freely through it. However, each slug will compress enough so that forced into the bottom portion of the neck **29** it will provide a vacuum or pressure tight seal. Thus during the intervals between injections a solid slug provides this seal.

The base plate **26** provides a platform for the block **25** to slide upon. Referring now to FIG. 6, the base plate **26** contains two poppets **30, 31** which provide forward and backward stops for the block motion. When the block **25** is pushed to the back of the base plate **26** to stop **30**, the upper portion of the injection sleeve **21** is revealed. It will be recalled that a solid slug **22** is already in position within the sleeve **21**. A cavitied slug **23** containing the sample is now placed in the sleeve **21**, followed by another solid slug **22**. The block **25** is then pulled forward to stop **31**.

At this position, the device is now in position to force the sample into the reservoir **3**. The block **25** contains a rack and pinion gear **24** operated by a lever **32**. When the lever **32** is pulled forward, the plunger **33** on the end of the rack **34** moves down. This plunger **33** contacts the upper surface of the top solid slug **22** and forces all three slugs through the injection sleeve **21**. The solid slug originally in the sleeve **21** drops through into the reservoir **3** followed by the cavitied slug containing the sample.

The plunger **33** can only travel a certain distance, such as ½ inch, at which point the shoulder **35** on the plunger **33** bottoms on the base plate **26**. At this point the final solid slug is then in the proper position to provide a vacuum seal and to allow another two slugs, first a cavitied slug **23** and then a solid slug **22**, to be loaded into the sleeve **21** above it. The whole operation takes less than a minute to accomplish and at all times a vacuum tight seal is provided as the slugs pass sequentially through the injection sleeve. Only minute amounts of air trapped between the slugs and the unfilled portion of the cavity in the cavitied slug **23** are allowed to enter the system. Further, the slugs are preferably made of a material such as Teflon which is chemically inert and heat resistant up to 800° F. and will not outgas in a vacuum.

When sufficient slugs have accumulated in the bulb **3**, they may be removed by venting the system to the atmosphere and removing the connection **11** at the bottom of the reservoir **3**. The slugs will then drop through.

More bulky items may be inserted into the reservoir **3** by utilizing a pair of cavitied slugs together with glass capillary tubing. Such an arrangement is shown in FIG. 7. Assuming the previously given dimensions of the cavitied slug, such glass capillary tubing should not have dimensions in excess of 0.376 inch long and .098 inch in diameter. The system should be prepared so that the vacuum seal is provided by the cavitied slug with the cavity facing outward rather than with a solid slug. Then a sample, for example one taken from a liquid chromatography unit contained in suitable glass capillary tubing **40**, is inserted into the cavity **41** in one cavitied slug **42** and capped with a second cavitied slug **43** with opposite orientation. The slug pair **42, 43** enclosing the capillary tubing **40** is then injected in a manner similar to the manner employed to inject the cavitied slug **23** of FIG. 3, forcing the existing slug **44** ahead of it into the reservoir **3**. As before, last to be inserted may be a solid slug, as shown at **45** in FIG. 7, or indeed another cavitied slug with the cavity facing outward. In this way the glass tube **40**, or any other type sample of this nature, may be inserted. Obviously, the size and shape of the sample is limited only by dimensions of the slug and the injection sleeve.

Having thus described the principles of the invention together with illustrative embodiments thereof, it is clearly to be understood that although specific terms are employed they are used in a generic or descriptive sense and not for purposes of limitation, the scope of the invention being set forth in the following claims.

I claim:

1. Injection system comprising, in combination, an injection sleeve having a minimum internal diameter, a multiplicity of cavitied slugs having a maximum external diameter not less than that necessary to provide a vacuum-tight seal during passage through said injection sleeve, a multiplicity of solid slugs having a maximum external diameter not less than that necessary to provide a vacuum-tight seal during passage through said injection sleeve, and mechanical means for overcoming frictional forces between said maximum external diameters and said mnimum internal diameter and thereby forcing said slugs through said injection sleeve.

2. Injection system for introducing small amounts of material into a vacuum-tight enclosure, comprising, in combination, a vacuum-tight enclosure, an injection sleeve supported on said vacuum-tight enclosure and having a channel providing communication between the interior and the exterior of said vacuum-tight enclosure, a multiplicity of solid slugs adapted to fit snugly in said channel, a multiplicity of cavitied slugs adapted to fit snugly in said channel, and mechanical means for forcing said slugs through said channel.

3. A method of injecting small amounts of material into a region, which method comprises introducing small amounts of such material into the cavity of a cavitied slug, flanking said slug with solid slugs, and mechanically forcing said slugs into said region through an injection sleeve into which said slugs fit snugly.

4. System for introducing material in the gas phase into a region, said system comprising in combination an evaporation reservoir, means for heating said reservoir, a vacuum pump, a manifold interconnecting said reservoir, pump and evacuated region, valves adapted to shut off said interconnections, and an injection system for introducing small amounts of said material in a non-gaseous phase into said reservoir, said injection system comprising, in combination with said reservoir, an injection sleeve supported on said evaporation reservoir and having a channel providing communication between the interior and the exterior of said reservoir, a multiplicity of solid slugs adapted to fit snugly in said channel, a multiplicity of cavitied slugs adapted to fit snugly in said channel, and mechanical means for forcing said slugs through said channel.

5. Injection system in accordance with claim **2**, wherein said mechanical means includes a plunger having a shoulder adapted to abut the external extremity of said channel so as to limit the inward motion of the plunger and thus prevent the plunger from forcing the final slug through the channel.

6. Injection system in accordance with claim **2**, wherein said mechanical means includes a rack and pinion gear mechanism.

7. Injection system in accordance with claim **1**, wherein said slugs comprise tetrafluoroethylene and fluorinated ethylpropylene resins.

8. A method of injecting small amounts of material into an evacuated region sealed by an interinjection seal

slug, which method comprises introducing small amounts of such material into a capillary tube to be held between and within the opposed cavities of a pair of cavitied slugs, and mechanically forcing said slugs into said evacuated region through an injection sleeve into which said slugs fit snugly, thereby ejecting said interinjection seal slug, the capillary, and the foremost cavitied slug, while leaving the hindmost cavitied slug as a replacement interinjection seal slug.

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JOHN J. CAMBY, Primary Examiner

U.S. Cl. X.R.

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