United States Patent

[11] **3,578,969**

[72] Inventor **Walter Proskauer**
San Francisco, Calif.

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[73] Assignee **Electronic Associates Inc.**
Long Branch, N.J.

[54] **SOLID SAMPLE INLET SYSTEM FOR A MASS SPECTROMETER**
8 Claims, 6 Drawing Figs.

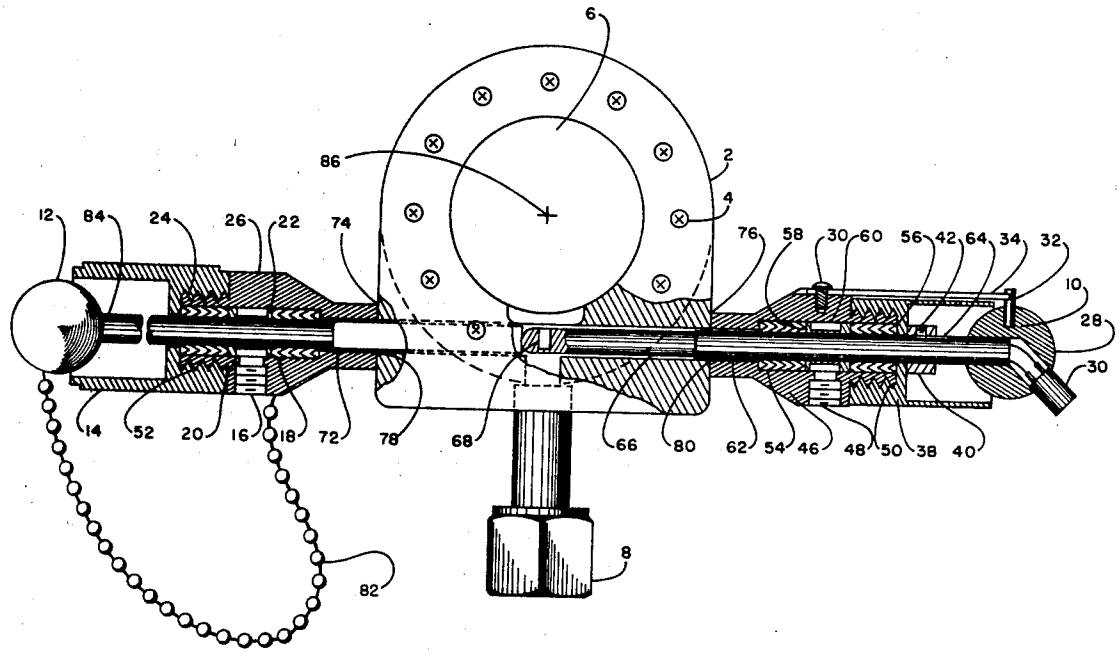
[52] **U.S. Cl.** **250/41.9,** 13/31, 250/49.5
[51] **Int. Cl.** **H01j 39/34**
[50] **Field of Search** ..... 250/41.9 (S), 41.9 (ISB), 49.5 (2); 13/31

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Primary Examiner—James W. Lawrence
Assistant Examiner—A. L. Birch
Attorneys—Edward A. Petko and Robert M. Skolnik

ABSTRACT: A system is disclosed for injecting solid material into an evacuated chamber without disturbing the pressure therein. Two rods are slidably mounted for extension into the chamber via sealed apertures at opposite ends thereof. A larger diameter rod may extend through the chamber and out the opposite ends. A smaller diameter rod carrying the solid material is interfit within the larger diameter rod. The larger rod is withdrawn as the smaller one is inserted.



3 Sheets-Sheet 1

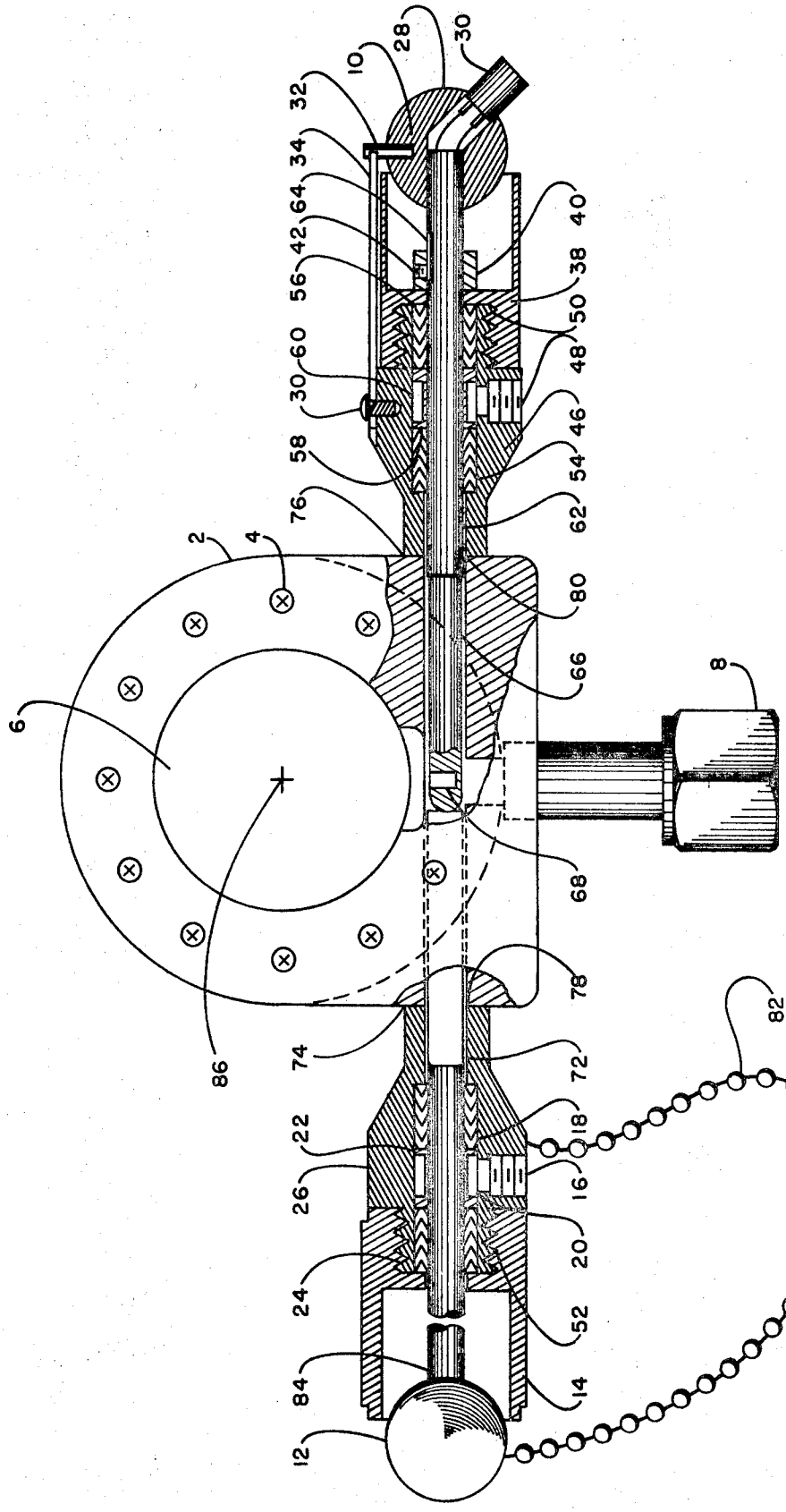


FIGURE 1

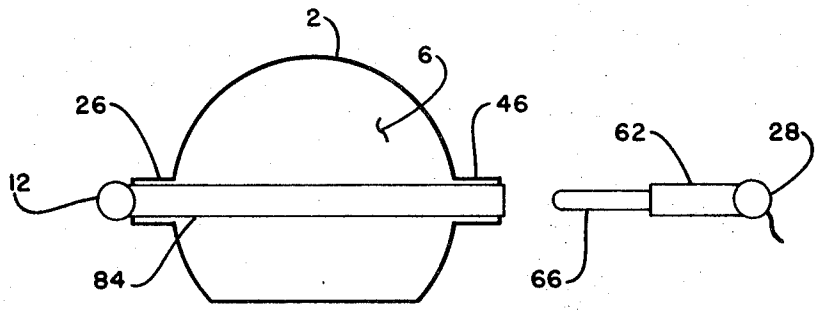


FIGURE 2A

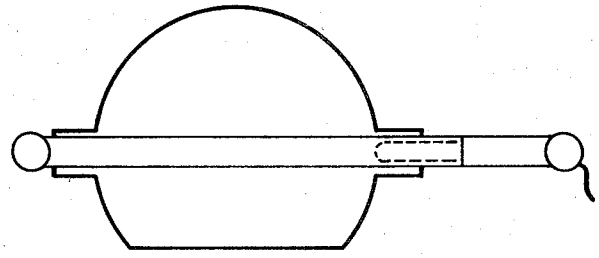


FIGURE 2B

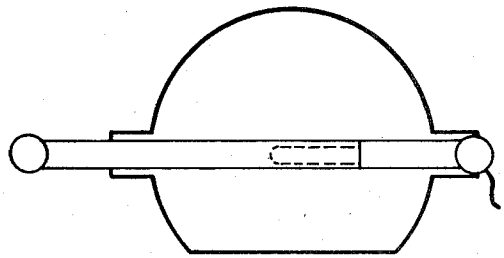


FIGURE 2C

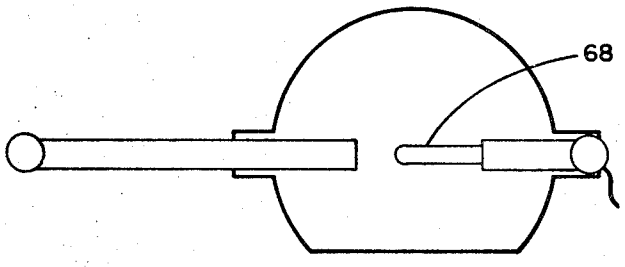


FIGURE 2D

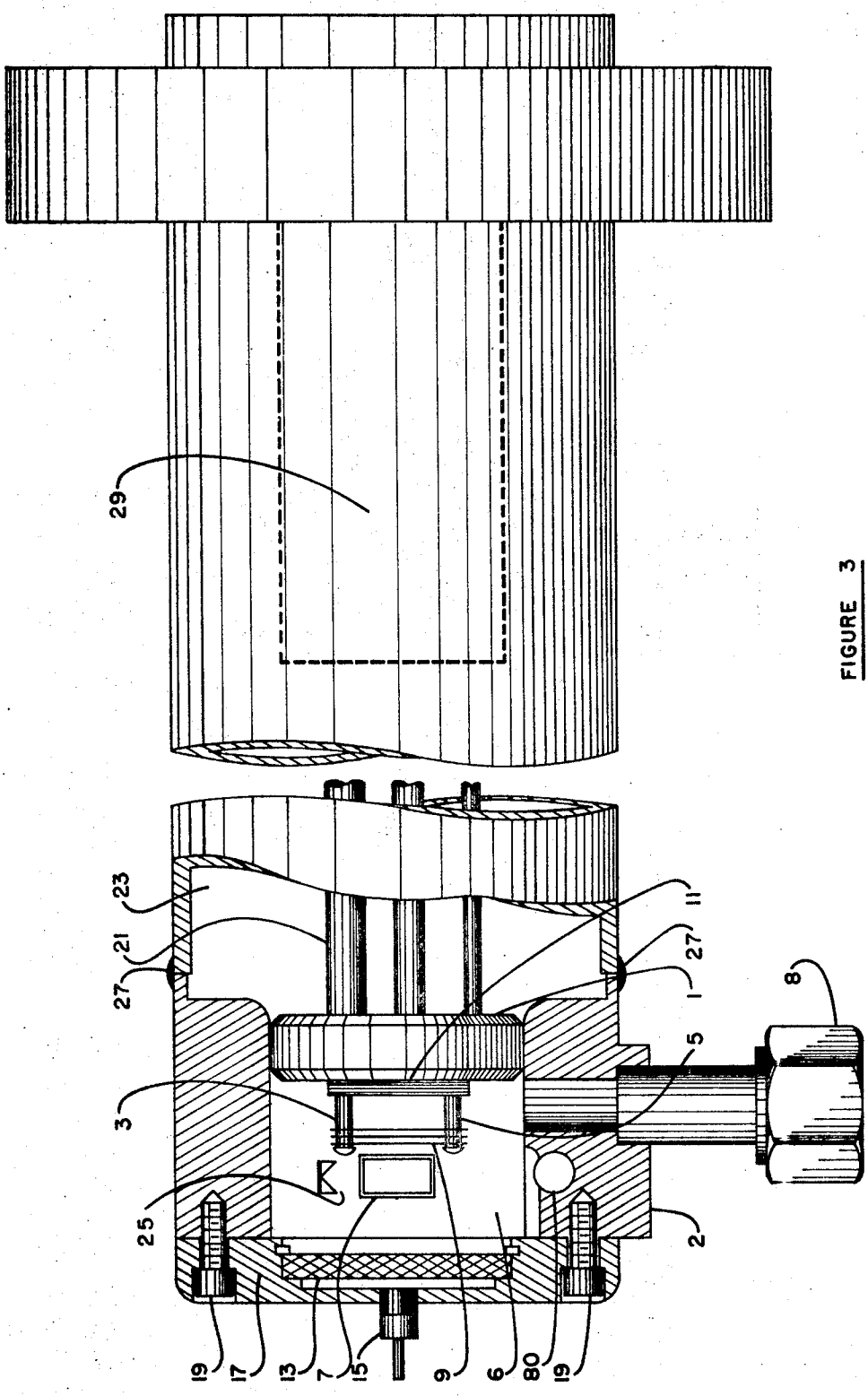


FIGURE 3

SOLID SAMPLE INLET SYSTEM FOR A MASS SPECTROMETER

This invention relates to a solid sample inlet system for injecting solid materials into an evacuated chamber without disturbing the internal pressure in the chamber.

The solid sample inlet system of the present invention is designed for use in the ionizer section of a quadrupole mass analyzer or any other type of mass spectrometer. Quadrupole analyzers or spectrometers have been described in the prior art and particularly in U.S. Pat. No. 2,939,952 to Wolfgang Paul et al., where a controlled varying voltage is used in conjunction with a fixed frequency in performing a mass spectrum analysis. Such analyzers may be used in measuring the composition of chemical substances and are comprised primarily of an ionizer, a quadrupole section, and an ion detector. Generally, the substance to be analyzed is introduced into the ionizer as a vapor at low pressure. A portion of the atoms or molecules which make up the chemical substance are ionized by electron bombardment or other means, and these ions are then accelerated and focused into the quadrupole section as an ion beam. The ion beam is filtered by permitting only those ions having a specific value of charge-to-mass ratios to pass through the quadrupole section. Those ions which are able to pass through the quadrupole section are then collected by the ion detector which may be an electron multiplier or a Faraday Cup.

The output current produced by the ion detector is a measure of the number of atoms or molecules in the ion beam which have a particular charge-to-mass ratio. The specific detected charge-to-mass ratio is determined by the values of the scanned or controlled varying RF voltage and the related DC voltage which are applied to the electrodes of the quadrupole section. The RF and DC voltages applied to the rods, at any given time determine the mass number of the ions being passed by the filter and the detector current indicates the ion abundance of that mass number. A display of the quantitative abundance of such detected ions as a function of atomic mass may be conveniently presented on conventional types of display instruments such as oscilloscopes, *x-y* recorders, or strip chart recorders.

Optimum performance of a quadrupole mass analyzer requires the maintenance of very low pressures in the ionizer, analyzer and detector sections. The highest pressure than can be tolerated in the ionizer is 10^{14} Torr, while the optimum pressure in the analyzer section is 10^{17} Torr or less. A torr is defined as the pressure required to support 1 mm. of mercury at 0° C. and at standard gravity.

Because of these high vacuum conditions, it is extremely difficult to introduce substance samples into the ionizer section without disturbing the vacuum conditions in the quadrupole. Such problems are more easily avoided where the substance to be analyzed is injected in gaseous form. To change to another substance, a valve is closed sealing the quadrupole and a new source of gaseous substance is connected. The valve is then opened and the analysis proceeds. Solid samples necessitate brining the quadrupole up to atmospheric pressure, dismantling the ionizer, placing the sample in the ionizing region and evacuating the system. This procedure consumes time that would otherwise be spent in the analysis function.

Mechanisms have been conceived involving a series of vacuum locks for sliding a sample into a first chamber, bringing the chamber to the pressure of the ionizer, opening the chamber, and moving the sample into position. These mechanisms are characterized by their complexity in construction, operation, and the probability of operator error.

The present invention provides a mechanism for easily injecting solid samples into the ionizer section without disturbing the vacuum conditions therein, and without the need for a series of valves and complex mechanical construction.

The invention employs two interfitting tubular members, each capable of lateral motion. A first large diameter member has a length extending completely across the ionizer section. The second, smaller diameter member has a length such that it extends into the ionizing region of the ionizer section. The system operates by placing the solid sample in the smaller diameter member, inserting the larger diameter member in the ionizer completely across the ionizer section, inserting the smaller diameter sample carrying member in the larger diameter one, and withdrawing the larger diameter member from the ionizer. This results in the solid sample being accurately positioned in the ionizing field without having disturbed the vacuum condition in the ionizer.

It is an object of the present invention to enable injection of substances into an evacuated space without disturbing the vacuum conditions.

Another object of this invention is the provision of means to inject substances in solid form to be analyzed by a quadrupole mass analyzer.

A further object of the present invention is the provision of a solid sample inlet system with which many different substances can be analyzed in an expeditious manner without disturbing the vacuum conditions in the analyzer.

These, as well as further objects and advantages of the present invention will be apparent from a reading of the following specification and drawings in which:

FIG. **1** is an end view, partially in section of the solid sample inlet system of the invention;

FIGS. **2A—2D** are a representation of the steps involved in manipulating the invention, and

FIG. **3** is a side view, partially in section, of a quadrupole mass analyzer adapted for use with the invention.

FIG. **1** is an end view of the solid sample inlet system according to the invention. In FIG. **1**, reference numeral **6** denotes an evacuated chamber formed by manifold **2**, which manifold may be affixed to another vacuum chamber by any means. Chamber **6** represents the ionizer section of a quadrupole mass analyzer but could be any area where a vacuum is maintained.

Chamber **6** is evacuated by a pumpout port shown generally at **8**. Port **8** is, in practice, connected to a vacuum pump. The details of the pump and connections thereto form no part of the present invention and will not be further described. Suffice it to say that such pumps are known and are commercially available.

Manifold **2** has apertures **78** and **80** formed therein. The configuration of these apertures is chosen to match the configuration of moveable rods **62** and **84**. In practice, these rods are cylindrical in shape but any desired configuration may be employed.

The location of aperture **78** and **80** in manifold **2** relative to the axial center **86** of the chamber **6**, may or may not be important depending on the particular application of the inlet system. In the quadrupole mass analyzer, it has been found advantageous to inject the sample orthogonally to the axial center line of the ionizer.

As the solid sample is heated, the molecular beam so generated will traverse the ionizing region at right angles to the desired ion beam and be intercepted by the ionizing electron beam. The molecules which are hit by electrons will be ionized while those which are not will continue in their original direction of travel and be collected by a relatively cool surface of an excess sample collecting plate, not shown. This reduces contamination in the analyzer section since unionized molecules are limited from entering.

Supports **26** and **46** are affixed to manifold **2** over the apertures **78** and **80** respectively as by welding shown generally at **74, 76**. Supports **26, 46** are identical in construction. Rods **62** and **84** are slidably mounted within suitably apertured portions of the supports.

Supports **26** and **46** also house a pressure sealing arrangement comprising elements **18, 22** and **24** for support **26**; and elements **54, 56** and **58** for support **46**. The sealing arrangement consists of two teflon glands separated by a stainless steel spool. This sealing arrangement ensures removal of unwanted injected gases since the spools **22** and **58** form spaces **20** and **60** respectively, which spaces are maintained at a lower pressure via pump ports **16** and **48**.

Support 26 has a cap 14 threadedly engaged thereto via screw threads 52. This cap provides access to the sealing structure when it is unscrewed while providing an excellent seal when tightened on the support.

Shaft 84 is provided with a handle 12. Chain 82, is connected between handle 12 and the manifold 2 to prevent accidental withdrawal of shaft 84 from the confines of the evacuated area, thereby preventing an undesired venting of the system.

Thus, it will be seen that shaft 84 may be moved in and out of the chamber 6 through the gland pressure sealing arrangement.

Shaft 62 serves as the solid sample carrying probe. More particularly, shaft 62 has a smaller diameter portion 66 having a cup 68 formed in its tip. Portion 66 also contains a heating element for any substance located in the tip. Power for the heater is supplied via electrical wires 70 which are located within the rod 80 and terminate in handle 28 at an electrical connector 30.

In order to ensure that the tip of the sample probe is oriented in the proper direction, arm 34 and pin 32 are provided. More particularly, arm 34 is affixed at one end to support 46 by way of a screw 30. Pin 32 permanently affixed to handle 28 engages a slot in arm 34. Thus, by aligning the pin 32 with the slot in arm 34, cup 68 is maintained in an upright position.

Rod 62 has an indented portion 64 located near the handle. This indented portion mounts a collar 40 which is affixed to the shaft by screw 42. The collar provides an adjustable stop for the sample probe, thereby ensuring that cup 68 will be correctly aligned relative to the lateral and longitudinal axes of the chamber 6.

It is to be noted that the diameter of rod 84 is the same as the diameter of the rod portion 62, while heater portion 66 is of a smaller diameter sufficient to enable the insertion of portion 66 within rod 84.

Cap 38 is threadedly affixed to support 46 by screwthreads 50. This cap has the same function as the cap 14 described above.

FIGS. 2A—2D show the sequence of operations involved in locating a solid sample within an evacuated chamber using the present invention. Like reference numerals denote like parts throughout the drawings.

FIG. 2A shows the initial position of rod 84 and sample probe 62. Rod 84 extends through chamber 6 to support 46. The gland seals in supports 26 and 46 prevent any loss of vacuum in this position. In FIG. 2B, the small diameter portion 66 of probe 62 is shown interfit with the larger diameter rod 84. The abrupt change in diameters between portion 66 and 62 serve to limit the engagement between rod 84 and portion 66.

In FIG. 2C, sample probe 62 is moved into position pushing rod 84 out of the support 46. Again, the gland seals prevent loss of vacuum in this position.

Finally, rod 84 is withdrawn from engagement with probe 62 which results in the precise positioning and uncovering of cup 68 within the vacuum chamber.

The above sequence of steps are reversed to withdraw the sample probe from the chamber.

FIG. 3 shows the ionizer section of a quadrupole mass analyzer adopted for use with the solid sample inlet system of the present invention.

Manifold 2 has aperture 80 therein as described in connection with FIG. 1. The ionizer consists of a Faraday cage 7 mounted above the aperture 80. A ceramic bushing 1, separates the ionizer section from the analyzer section shown generally at 23. The bushing 1 mounts on the quadrupole rods 21, whose function is completely described in the aforementioned Paul et al. patent, and in commonly assigned copending applications, Ser. Nos. 486,660 now abandoned and 486,703, now U.S. Pat. No. 3,457,404 both filed Sept. 13, 1965. The bushing 1 also provides a restriction in pumping speed between chamber 6 and analyzer section 23. This restriction results in a difference in pressure between ionizer section 6 and analyzer section 23 ensuring a greater probability of ionization.

In addition to serving as a support for the rods, bushing 1 also has clearance holes 11 for the ionizer leads. Spacers 3 and 5 support the lens structure 9 which focuses the beam of ions developed by the cage 7 and a filament 25.

Cap 17 is affixed to manifold 2 by screws 7 and 19 to permit easy access to the ionizer. Cap 17 also contains a heater 13 and necessary electrical connections 15 for the heater.

The entire manifold assembly 2 is affixed to the quadrupole section 23 as by welding shown generally at 27.

The substance to be analyzed is thus injected into the ionizer through aperture 80, formed into an ion beam by cage 7 and filament 25 and formed into the quadrupole section via electrode lens 9.

The ions which traverse the analyzer section are detected in an electron multiplier 29. The output current of the multiplier is a measure of the presence in the sample of ions of particular charge-to-mass ratios.

It is to be noted that, when used in the quadrupole, it is advisable to make the cup 68 of the sample probe 62 removable. This is necessary because of possible reaction between the material of the cup and the sample in the cup. This is accomplished by making the tip of the probe threadedly engage the heater portion 66.

The multiple aperture system of FIG. 1 may be utilized to provide a quick change capability in the analysis of solid and gaseous samples. For example, more apertures may be provided in manifold 2 having valves, etc. connected thereto. Various samples could thus be connected to the analyzer as desired.

I claim:

1. An apparatus for injecting material into an evacuated space without disturbing the vacuum conditions therein comprising,

an evacuated chamber having at least two aligned apertures thereon,

orificed sealing and support means affixed to said chamber over each of said apertures,

a first elongated member of length sufficient to pass through said apertures and orificed sealing means,

a second elongated member having a material carrying portion capable of interfitting within said first elongated member, whereby said material may be inserted in said chamber by interfitting said first and second members and withdrawing said first member as said second member is inserted.

2. A system for injecting solid material in an evacuated chamber without disturbing the vacuum conditions therein, comprising a chamber having at least two apertures aligned on opposite sides thereof,

support means affixed over each of said apertures, said support means having two orifices of different respective diameters formed therein,

pressure sealing means mounted within the larger diameter orifice, said sealing means having an aperture therein aligned with the smaller diameter orifice,

a first hollow elongated member mounted on a first of said support means for movement within said orifice and said sealing means said member being of sufficient length to pass through both said apertures,

a second elongated member having a cup portion for carrying solid material and capable of interfitting within said first member mounted on the other of said support means for movement within said orifice and said sealing means so that when said first member traverses both apertures, said second member may be inserted into said first member and, thereby inserted into said chamber as said first member is withdrawn.

3. The system of claim 2 wherein said sealing means includes first and second glands separated by a spool, said spool providing a space between said sealing means and said larger diameter orifice, whereby said sealing means can be main-

tained at a desired relative pressure to the pressure within said chamber.

4. The system of claim 2 wherein said first elongated member is affixed to said support to prevent accidental withdrawal of said member from said chamber, and

said second elongated member is limited for slidable movement within said chamber so that said cup is properly located therein.

5. The system of claim 2 wherein said second elongated member includes a heating element for said cup portion whereby said material may be brought to a desired temperature.

6. In a quadrupole mass analyzer having an ionizer, a filter section and an ion detector, a system for injecting solid samples therein comprising,

an ionizer including a chamber and means to generate an electron beam and to focus an ion beam,

first and second aligned apertures on said chamber.

orificed sealing and support means affixed to said chamber, over each of said apertures,

a first hollow elongated member capable of extending within both said first and second apertures and slidably mounted in one of said orificed means,

a second elongated member having a sample cup formed in

one portion thereof and a heater for said cup, slidably mounted in the other of said orificed means, said heater and cup portion being capable of interfitting within said hollow first member,

whereby said cup may be inserted into said chamber as said first member is withdrawn, and solid material in said sample cup produces a molecular beam when heated,

said apertures being located such that said electron beam, said molecular beam and said ion beam are mutually orthogonal.

7. The injection system of claim 6 wherein each of said sealing and support means includes a member having large and small diameter orifices therein, and a seal mounted in said large diameter orifice and including first and second gland means separated by a spool member,

said spool providing a space thereby permitting said seal to be maintained at a desired pressure relative to the pressure within said chamber.

8. The inlet system of claim 6 wherein said first elongated member is affixed to said chamber to prevent accidental withdrawal therefrom and said second elongated member includes stop means ensuring accurate positioning of said cup in said chamber.