Jan. 7, 1964 C. BRUNNÉE 3,117,223

SAMPLE INSERTION VACUUM LOCK FOR A MASS SPECTROMETER

Filed June 9, 1961 2 Sheets-Sheet 1

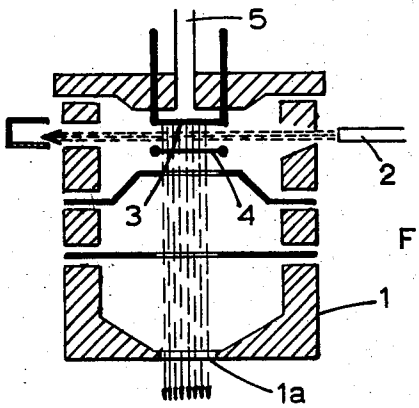


FIG. 1.

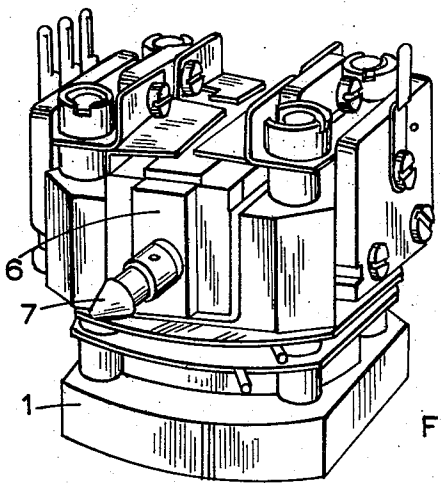


FIG. 2.

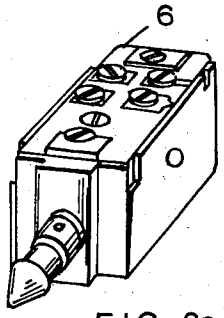


FIG. 2a.

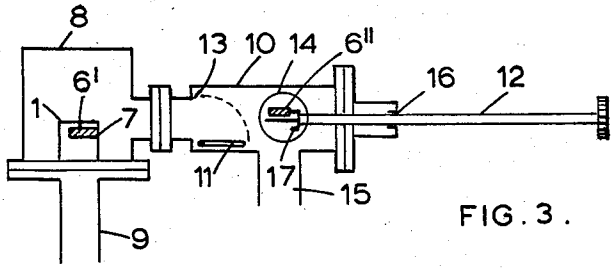


FIG. 3.

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SAMPLE INSERTION VACUUM LOCK FOR A MASS SPECTROMETER

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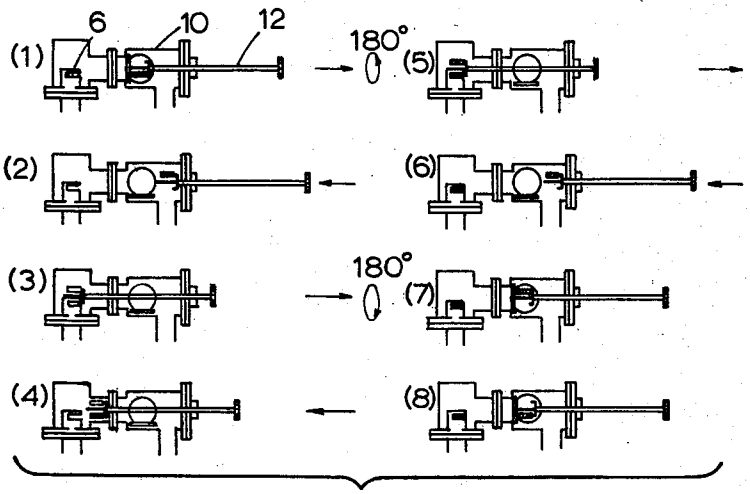


FIG.4

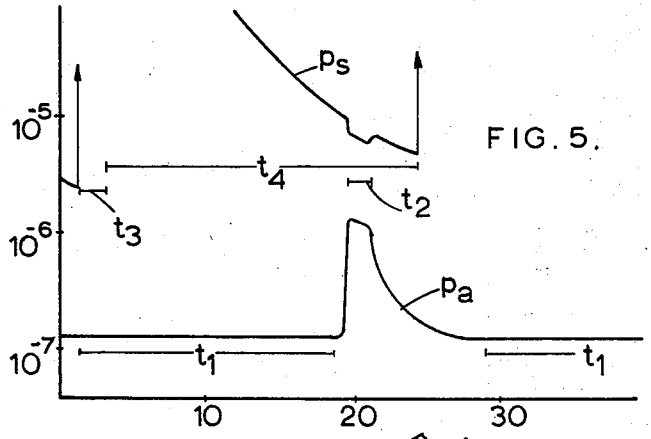


FIG. 5.

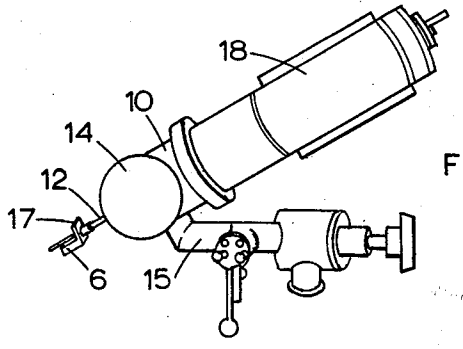


FIG. 6.

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SAMPLE INSERTION VACUUM LOCK FOR A MASS SPECTROMETER

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The present invention relates to vacuum locks and particularly to vacuum locks used in mass spectrometers for changing specimen carriers. In such locks, the specimen carrier is moved by a thrust rod through a valve aperture from the lock chamber into the analyzer, or other vacuum chamber, and subsequently removed. Known vacuum locks of this type have the drawback that the change of specimen has to be effected in two separate operations. After measurement of a first sample is completed, it is first necessary to withdraw the used specimen carrier from the ion source of the mass spectrometer thus exposing the sample to atmospheric pressure, and then to fix a new specimen carrier on the thrust-rod, evacuate the lock chamber again and to reintroduce a new specimen carrier after opening the valve between the evacuated lock chamber and the analyzer chamber.

Vacuum locks are already known in which the change of specimen is effected in a single operation but they involve an undesirably high capital investment, usually cannot be separated from the analyzing head of the mass spectrometer, or only in a complicated manner, and also have the serious defect that the surfaces exposed to atmospheric pressure during the changeover, because of their size, are exposed to an undesirable amount of gas absorption and subsequent escape of gas. Moreover, generally speaking, it is impossible to de-gas the sample carrier in the lock while the ion source is operating.

In accordance with the present invention this drawback is overcome in this way, that in such a vacuum lock, the thrust rod is arranged for the removal of the used sample carrier and the insertion of a new sample carrier in a single operation, i.e. without intermediate ventilation of the lock chamber, by the fact that in a first setting, for example with a rotary movement a setting of zero degrees, it grips the specimen carrier situated in the analyzer and removes it from an operating position, and in a second setting, for example a rotary setting of, 180°, introduces a new specimen carrier into the operating position in place of the one which has been removed.

The thrust rod can also be used with advantage for opening and reclosing the valve between lock chamber and analyzer. Consequently only one bushing is needed for the transmission of the mechanical forces, inserting the sample and actuating the valve.

The invention will be described by way of example with reference to the accompanying drawings, in which:

FIGURE 1 shows the basic structure of a combined thermionic and furnace ion source.

FIGURE 2 is a perspective view of the ion source,

FIGURE 2*a* is a perspective view of the sample carrier removed from the ion source.

FIGURE 3 is a schematic representation of a mass spectrometer with a vacuum lock embodying the invention for the interchange of specimen carriers,

FIGURE 4 shows the mode of operation of the vacuum lock shown in FIGURE 3 in eight different positions,

FIGURE 5 shows the pressure curve in the vacuum lock and in the analyzer when changing samples by the method illustrated in FIGURE 4, and

FIGURE 6 shows the mechanical structure of a vacuum lock embodying the invention.

Referring now to FIGURE 1, this shows the basic structure of the ion source, which is a combined thermionic and furnace ion source and can also be used for the

analysis of gases. The drawing shows the ionization housing 1 with an ion beam and an exit slot 1*a* therefor. The ionization takes place either by ionic bombardment or by thermionization on an incandescent tungsten tape. On the right-hand side of the housing is a cathode 2 for the emission of the electrons and at the top a tape 3 for the surface ionization. Close beside the ionization tape 3 is mounted a second tape 4 by which the solid sample is vaporized. This tape 4 can be replaced by a small evaporator furnace.

The ion source can thus be operated either as a double-tape thermionic source with surface ionization or as a furnace-ion source with electron-collision ionization. The type of operation chosen depends upon the kind of substance to be investigated. As already stated, the ion source can also be used for gas analysis and for this purpose is provided with a glass inlet tube so that, without change of ion source, it is possible also to investigate gases, which may be important for comparative tests.

The analysis of solid substances involves two problems i.e. the change of sample and the contamination of the ion source by the deposit of substance inside the ion source during vaporation. The construction shown takes both problems into account. An ionization box, or speciment carrier, 6 (FIGURE 2) is provided in the ion source for the purpose of ionization. This box, together with ionization tape and evaporator tape or furnace, is arranged to be interchangeable and can be drawn out of the ion source 1 by a pin 7. The electrical leads are automatically connected via contact springs when the box is plugged in. When changing the sample, therefore, only the ionization box is withdrawn from the ion source and a second box, the evaporator tape of which carries the new sample, is plugged into the ion source.

Normally, of course, in changing the sample it is necessary to ventilate the analyzer so that the number of daily analyses is restricted to two or three. In order to avoid this ventilation, a vacuum lock is provided by means of which the ionization box 6 with the sample can be interchanged without ventilating the analyzer.

FIGURE 3 shows how this lock operates and on the left of the figure is the analyzer head 8 with the ion source 1 and the hatched ionization box 6'. The ions are introduced downwards through the entry slot into the separating tube 9. The vacuum in this part is maintained via two exhaust tubes (not shown) of two mercury diffusion pumps. On the right is the actual vacuum lock 10, which is separated from the analyzer 8, 9 by a high-vacuum-tight sealing flap 11.

A thrust rod 12 presses the flap 11 against a Teflon ring 13, so that high-vacuum-tight sealing of the lock-chamber is obtained. The lock chamber is accessible from outside, after the opening of a trap, indicated by a circle 14, and can be evacuated via a valve from a pump. The valve and an oil diffusion pump are connected to the downwardly directed nozzle 15. The thrust rod carries the new ionization box 6'' which is to be introduced into the ion source.

FIGURE 4 shows the individual stages of movement for a change of sample. After the evacuation of the lock 10 (FIGURE 4.1), the thrust rod 12 is first pulled back slightly whereby the flap 11 is automatically opened. The thrust rod is moved by a motor which is automatically controlled by microswitches. The sealing of the slide bushing is effected by a grease-free Teflon sleeve 16. After turning the thrust rod through 180° the position of FIGURE 4.2 is obtained. The rod is now pushed in until, in the position of FIGURE 4.3, a sleeve 17 on the the thrust-rod 12 slips over the pin 7 on the ionization box 6' to be changed and holds it. After its withdrawal and turning of the thrust rod the position of FIGURE 4.4 is obtained.

Pushing the rod 12 forward now brings the box 6'' with

the new sample into the ion source (FIGURE 4.5). Now the rod is completely retracted (FIGURE 4.6) and again pushed slightly forward (FIGURE 4.7) whereby the flap 11 is automatically closed. The change of sample is therefore completed.

After ventilating the lock 10, the lock cover can now be opened, the old sample 6' removed and a new sample inserted in the lock chamber (FIGURE 4.8). During this time and during the evacuation of the lock the mass spectometric measurement is carried out.

Curves p_s and p_a of FIGURE 5 show the variation of the pressure in the lock 10 and in the analyzer 8, 9 when the sample is changed. During the evacuation of the lock in the time interval t_1 a mass spectrometric measurement is carried out. After this measurement is finished, the lock 10 is actuated, the flap 11 opens, and the pressure p_a in the analyzer 8, 9 rises.

Because of the additional pumping capacity through the pumps of the analyzer portion, the pressure p_s in the lock drops a little. After the sample has been changed in the time interval t_2, the flap 11 is closed again, the pressure p_a in the analyzer drops very rapidly and a new measurement can begin. When a measurement begins the lock 10 is ventilated and in a time interval t_3 a new ionization box 6 is inserted in the vacuum lock. The lock is then closed and the pressure p_s, which rose steeply during ventilation, drops, for example, to less than 10^{-5} (mm. Hg) through evacuation until the next ventilation, during a time interval t_4.

There are two interesting details to be mentioned for the use of the lock:

The operation of the lock is automatically blocked as long as the ion source 1 is in operation. Secondly, the evaporator tape or furnace, before it is slipped into the ion source, can be degassed in the vacuum chamber of the lock 10. The ionization box 6 is for this purpose plugged into a support inside the lock 10 in which suitably arranged contacts connect the evaporator tape or the furnace with the filament current regulator forming part of the equipment of the lock. The mechanical design of the lock (without the pumps and motor actuation) is shown in FIGURE 6. In this a drive-and-control device surrounded by a cylindrical casing 18 is coaxially connected to the lock 10, for the automatic carrying out of the process represented in FIGURES 4.1 to 4.8. The whole lock can be heated up to 150° C. and contains no greased packings.

The following five points summarize what may be regarded as particularly advantageous in the described design of solid-substance-ion source and vacuum lock.

(1) The lock mechanism is completely separate from the rest of the mass spectrometer, i.e. the mass spectrometer can also be used without restriction for all measurements after the mounting of the lock.

(2) The investment in apparatus is relatively low. The only additional pumping equipment required is a small oil diffusion pump for the lock, which is connected to the nozzle 15.

(3) It is possible, without reconstruction, to investigate both solid bodies in thermionic or furnace-ion operation and also gases or liquids.

(4) The lock changes the samples in a single operation after the drive-and-control mechanism is switched on. This makes it possible to degas a sample in the lock in the time interval t_1 during the mass spectrometric measurement.

(5) Only the ionization chamber of the box 6 is exposed to the air, i.e. only a part of small dimensions and therefore small surface; the actual ion source 1, on the other hand, remains always in the high-vacuum of the analyzer.

I claim:

1. A vacuum lock for changing a sample in a mass spectrometer, said lock comprising means defining a chamber, said chamber defining an elongated passageway terminating in a first opening, a flap located in said passageway and adapted to open and close said opening, said lock having means defining a second opening for said passageway adapted to be vacuum sealed, a thrust rod having a first end extending into said chamber and a second end located outside of said chamber, said thrust rod being mounted for reciprocal movement relative to a longitudinal axis thereof and for rotational movement about said axis, said thrust rod first end carrying a first means for inserting a first sample in a mass spectrometer and a second means for removing a second sample from a mass spectrometer during a single opening of said chamber to said mass spectrometer.

2. In a mass spectrometer having means defining a vacuum area, the improvement comprising a vacuum lock for changing a sample to be analyzed in said mass spectrometer, said lock comprising, means for removing a first sample contained in said vacuum area and a second means for inserting a second sample in said vacuum area during a single opening of said vacuum area to said vacuum lock.

3. In a mass spectrometer having means defining a vacuum area, the improvement comprising, a vacuum lock for changing a sample to be analyzed in said mass spectrometer, said lock comprising means defining a vacuum passageway terminating in a first opening connecting said vacuum area and said passageway, a valve means adjacent said opening for vacuum sealing thereof, a second opening defined by said passageway adapted to be vacuum sealed, a thrust rod having a first end extending into said passageway, said thrust rod being mounted for reciprocal movement relative to a longitudinal axis thereof and for rotational movement about said axis, said thrust rod first end carrying a first means for inserting a first sample carrier in said vacuum area and a second means for removing a second sample carried from said vacuum area during a period when said valve means is opened thereby allowing communication between said vacuum area and said passageway.

4. The improvement in a vacuum lock as claimed in claim 3, wherein a means for creating a vacuum in said passageway is operatively engaged with said vacuum lock, and said first and second means for respectively inserting and removing sample carriers are symmetrically arranged with respect to said thrust rod axis and are carried by radially extending arms from said thrust rod.

5. A vacuum lock useful for changing a sample in a vacuum area defined by a spectrometer said lock defining a vacuum chamber, said chamber defining plural spaced vacuum sealable openings, an elongated thrust rod mounted in said chamber having a first end carrying first means for removing a sample from a vacuum area upon axial movement of said rod and a second means for inserting a replacement sample in said vacuum area upon rotation of said rod and axial movement thereof, said first and second means being positioned radially of said thrust rod and symmetrically located with respect to an axis of said thrust rod.

6. The vacuum lock of claim 5 wherein said first and second means are located substantially 180° apart and are carried by radially extending arms from said thrust rod.

7. The vacuum lock of claim 5 wherein one of said openings has an adjacent flap means for vacuum sealing thereof.

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