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VACUUM LOCK SOLID SAMPLE SOURCE DEVICE FOR MASS SPECTROMETERS

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FIG. 2.

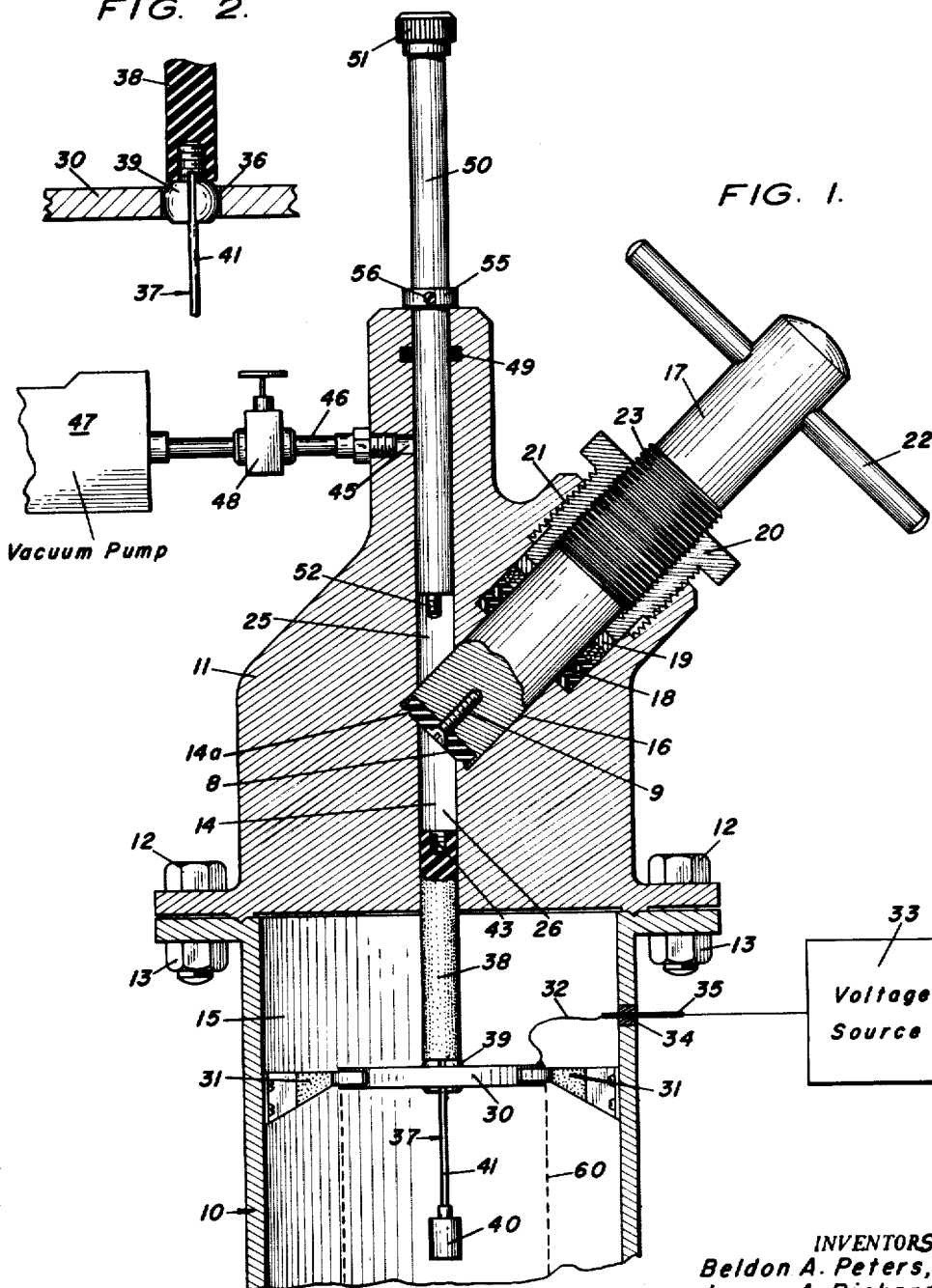


FIG. 1.

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VACUUM LOCK SOLID SAMPLE SOURCE DEVICE FOR MASS SPECTROMETERS

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This invention is directed to apparatus for replacing solid samples in a vacuum lock mass spectrometer.

Briefly, the invention comprises a housing provided with a closure member having a passage therein extending between the interior and exterior of the housing, valve means positioned on the closure member for closing off the passage intermediate its length thereby isolating the interior from the exterior of the housing, sample holder means positioned within the housing, stationary supporting means mounted in the housing and insulated therefrom adapted to support and electrically connect with the sample holder means, an electrical conductor extending from the supporting means adapted to connect the sample holder means with a high voltage source, sample replacement means sealingly slidable in the passage engageable with the sample holding means for removing and replacing samples, and vacuum source means communicating with the passage adapted to apply a vacuum to the passage and associated housing.

In mass spectrometers currently in use, measurements made on gas or liquid samples are not difficult. However, making mass spectrometer measurements on solid samples is more complex because the spectrometer tube must be open to the atmosphere for removing and replacing the solid samples. Opening of the spectrometer tube to the atmosphere results in long delays while the vacuum is again reduced to a sufficiently low value to permit the mass spectrometer measurements. It may take hours to obtain a proper vacuum after exposure of the tube to atmosphere whereas it only takes minutes to actually change the samples. Further, the "memory" effects of absorbed gases can be removed only after prolonged vacuum pumping and may be present at all times.

An object of this invention is to provide apparatus to permit changing of solid samples without breaking the vacuum in the spectrometer tube. By not breaking the vacuum, the time consumed for each sample analysis is drastically reduced and permits analysis of solid samples on a routine basis whereas, previously, these samples were examinable only on a research basis.

Another object of this invention is to provide apparatus wherein the spectrometer tube high vacuum region is sealed firmly and positively from the atmosphere. Maintaining a high vacuum region is of fundamental importance in obtaining the very high vacuum required for accurate analyses. This invention eliminates materials of questionable high vacuum properties such as rubber O-rings and this device does not depend on a sliding vacuum seal with its inherent questionable high vacuum properties.

A further object of this invention is to provide apparatus wherein a large number of interchangeable sample holders may be easily fabricated. This results in a reduction in the time required for changing samples and a simplification of the changing procedure.

Another object of this invention is to provide apparatus wherein the sample can be changed without moving or disturbing any of the electrical connections. The design

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of the metallic support plate into which the sample holder fits is such that the electrical connections can be made to the sample holder without incorporation in the movable sample holder itself.

Referring to the drawings:

Fig. 1 illustrates a partly schematic vertical sectional view of the device.

Fig. 2 illustrates an enlarged fragmentary sectional view of the sample holder assembly and plate of Fig. 1.

Referring to the drawings in greater detail wherein identical numerals designate identical parts, numeral 10, Fig. 1, designates a fragmentary portion of a mass spectrometer tube or vacuum housing. The vacuum housing 10, preferably cylindrically configured, is provided with a closure member 11 secured thereto by means of a plurality of bolts 12 and nuts 13. Closure member 11 is provided with an elongated passage 14 extending from the interior 15 of the housing to the exterior thereof. The passage 14 has formed thereon a valve seat 14a intermediate the length thereof. A second passage 16 extends from passage 14 adjacent valve seat 14a to the housing exterior. A valve stem 17 is arranged in passage 16 for movement therein. Suitable packing means 18 are provided adjacent passage 16 for sealingly engaging valve stem 17. The packing means 18 is provided with retaining lugs 19 and 20. Lug 20 is screw threadedly connected to the walls of passage 16 as at 21 and the valve stem 17 screw threadedly connects with lug 20. A sealing gasket 8 is secured to the inner end of valve stem 17 by means of screw 9. A handle 22 is provided on valve stem 17 whereby the valve stem is moved inwardly and outwardly on screw threads 23, thereby opening and closing passage 14.

Passage 14 is divided into two regions 25 and 26 when the sealing gasket 8 of valve stem 17 is seated on valve seat 14a.

A metallic, preferably cylindrical support plate 30 is rigidly mounted within housing 10 and may be secured to housing 10 by means of one or more insulated support rods 31 extending between the support plate 30 and the housing 10. An electrical conductor 32, 35 connects support plate 30 to a source of high voltage 33 located outside of housing 10. Conductor 32, 35 extends through housing 10 and is insulated therefrom by means of insulation 34.

As seen more clearly in Fig. 2, support plate 30 is provided with a central opening 36 through which extends a sample holder assembly, generally designated 37. The sample holder assembly 37 includes an insulated shaft 38 to which is connected a spring plug 39 which is in turn connected to a sample holder capsule or container 40 by means of a rod 41. As shown more clearly in Fig. 2, spring plug 39 compresses slightly and wedges in opening 36 and electrically connects support plate 30 and sample holder or container 40. The insulated shaft 38 insulates plate 30 from the closure member 11 of housing 10. This shaft 38 extends partially into passage 14 when the sample holder assembly 37 is in operating position as shown in Fig. 1. The upper end of insulated shaft 38 is provided with a threaded indentation 43 adapted to threadedly engage with a shaft 50.

The region 25 of passage 14 has connected thereto a vacuum passage 45 to which is connected a conduit 46 which in turn communicates with a vacuum pump 47. A valve 48 is provided in conduit 46 for controlling the vacuum in passage 14. The upper end of passage 14, above passage 45, is provided with an O-ring seal 49.

The shaft 50, provided with a knurled end 51, is slidably extensible in passage 14. O-ring 49 sealingly engages shaft 50 thereby preventing fluid flow through passage 14 when shaft 50 extends below the O-ring 49. Shaft 50 is also provided with a threaded member 52 at

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its lower end which is engageable with the threaded indent 43 in insulator shaft 38 noted supra.

The upper end of closure member 11 is provided with a collar 55 which is provided with a set screw 56 adapted to engage and secure shaft 50 in any desired position.

As shown in Fig. 1, the apparatus is ready for analysis of a solid sample held in sample holder 40. Valve 48 is normally maintained open. To change the samples, valve 48 is closed and the valve stem 17 is withdrawn by turning handle 22. Withdrawing valve stem 17 communicates regions 25 and 26 of passage 14. When these passages are completely open to each other the set screw 56 is loosened in collar 55 permitting shaft 50 to slide in passage 14. Shaft 50 is then moved downwardly and is threadably engaged into the indent 43 in shaft 38 by means of threaded member 52 on shaft 50. Shaft 50 is then withdrawn from passage 14 thereby removing simultaneously therewith the sample holding assembly 37 including insulated shaft 38, metallic spring plug 39 and sample holder 40. Then, shaft 50 is moved upwardly in passage 14 until the sample holder 40 is above valve stem 17. Valve stem 17 is then closed and the shaft 50, insulated shaft 38, spring plug 39 and sample holder 40 are completely withdrawn from passage 14. This removal permits air to enter region 25 and passage 45 leading to valve 48. After the sample has been changed in sample holder 40, shaft 50 and the sample holder assembly 37 are reinserted into region 25 of passage 14. The valve 48 is then opened and region 25 is evacuated by means of vacuum source 47. The O-ring 49 prevents fluid leakage around shaft 50. When the vacuum in region 25 becomes sufficiently low the valve 48 is closed, the valve stem 17 is opened and the shaft 50 is moved downwardly until the spring plug 39 is engaged in opening 36 in the metallic support plate 30. The shaft 50 is then unscrewed from the insulated shaft 38 and withdrawn to the position shown in Fig. 1. Collar 55 is then fixed by means of set screw 56 to shaft 50 thereby preventing atmospheric pressure from pushing shaft 50 downwardly. The valve stem 17 is then closed which seals gasket 8 to valve seat 14a providing a vacuum seal for the high vacuum regions 26 and 15 and isolating these high vacuum regions from possible leak points in O-ring 49. As an extra precaution, valve 48 may remain open during the process of changing the samples thereby maintaining region 25 evacuated.

In the analysis, high voltage from the high voltage source is fed to sample holder 40 through conductor 32 and support plate 30. Analysis is made in the conventional manner in an ion source 60.

While we have illustrated our invention with a crucible type solid sample holder 40, the apparatus of the invention is applied with equal ease to the filament type and to other solid sample types.

Having set out fully the objects of our invention and having fully described the apparatus and operation thereof, we desire to claim:

1. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, a removable sample holder assembly including a container member, a metallic spring plug and an insulator member positioned in said housing, a support plate engageable with said spring plug rigidly mounted in and electrically insulated from said housing, said plate having an opening through which said sample holder assembly extends, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a voltage source, a shaft sealingly slidable in said first passage adapted to engage with said insulator member, 75

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a third passage in said closure member extending between said housing exterior and said first passage, said exterior end of said third passage being adapted to communicate with a controlled vacuum source and the inner end of said third passage communicating with said first passage between said valve seat and said housing exterior.

2. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, removable sample holder means positioned within said housing, a support plate engageable with said sample holder means rigidly mounted in and electrically insulated from said housing, said plate having an opening through which said sample holder means extends, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a voltage source, a shaft sealingly slidable in said first passage adapted to engage with said sample holder means, a third passage in said closure member extending between said housing exterior and said first passage, said exterior end of said third passage being adapted to communicate with a controlled vacuum source and the inner end of said third passage communicating with said first passage between said valve seat and said housing exterior.

3. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, a removable sample holder assembly including a container member, a metallic spring plug and an insulator member positioned in said housing, stationary supporting means mounted in said housing and insulated therefrom adapted to support and electrically connect with said metallic spring plug, an electrical conductor extending from said supporting means through said housing adapted to electrically connect said supporting means to a voltage source, a shaft sealingly slidable in said first passage adapted to engage with said insulator member, a third passage in said closure member extending between said housing exterior and said first passage, said exterior end of said third passage being adapted to communicate with a controlled vacuum source and the inner end of said third passage communicating with said first passage between said valve seat and said housing exterior.

4. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member having a passage therein extending between the interior and exterior of said housing, a valve means positioned in said closure member for closing off said passage intermediate its length thereby isolating the interior from the exterior of said housing, a removable sample holder assembly including a container member, a metallic spring plug and an insulator member positioned in said housing, a support plate engageable with said spring plug rigidly mounted in and electrically insulated from said housing, said plate having an opening through which said sample holder assembly extends, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a voltage source, a shaft sealingly slidable in said passage adapted to engage with said insulator member, and vacuum source means communicating with said passage adapted to apply a vacuum to said passage.

5. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending

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between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, sample holder means positioned within said housing, stationary supporting means mounted in said housing and insulated therefrom adapted to support and electrically connect with said sample holder means, an electrical conductor extending from said supporting means adapted to connect said sample holder means with a voltage source, sample replacement means sealingly slidable in said first passage engageable with said sample holding means for removing and replacing samples, and vacuum source means communicating with said first passage adapted to apply a vacuum to said first passage.

6. A vacuum lock solid sample device for mass spectrometers comprising a housing including a closure member having a passage therein extending between the interior and exterior of said housing, valve means positioned on said closure member for closing off said passage intermediate its length thereby isolating the interior from the exterior of said housing, sample holder means positioned within said housing, stationary supporting means mounted in said housing and insulated therefrom adapted to support and electrically connect with said sample holder means, an electrical conductor means extending from said supporting means adapted to connect said sample holder means with a voltage source, sample replacement means sealingly slidable in said passage engageable with said sample holding means for removing and replacing samples, and vacuum source means communicating with said passage adapted to apply a vacuum to said passage.

7. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, a removable sample holder assembly including a container member, a metallic spring plug and an insulator member positioned in said housing, a support plate engageable with said spring plug rigidly mounted in and electrically insulated from said housing, said plate having an opening through which said sample holder assembly extends, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a voltage source, a shaft slidable in said first passage adapted to engage with said insulator member, a third passage in said closure member extending between said housing exterior and said first passage, said exterior end of said third passage being adapted to communicate with a controlled vacuum source and the inner end of said third passage communicating with said first passage between said valve seat and said housing exterior, and sealing means positioned adjacent said first passage between the inner end of said third passage and the exterior of said housing adapted to seal the annulus between said shaft and the wall of said first passageway.

8. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, removable sample holder means positioned within said housing, a support plate engageable with said sample holder means rigidly mounted in and electrically insulated from said housing, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a

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voltage source, a shaft sealingly slidable in said first passage adapted to engage with said sample holder means, a third passage in said closure member extending between said housing exterior and said first passage, said exterior end of said third passage being adapted to communicate with a controlled vacuum source and the inner end of said third passage communicating with said first passage between said valve seat and said housing exterior.

9. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member, a first passage in said closure member extending between the interior and exterior of said housing, a valve seat arranged in said first passage, a second passage in said closure member extending between said valve seat and the exterior of said housing, a valve stem sealingly movable in said second passage, a removable sample holder assembly in said housing, a stationary supporting means mounted in said housing and insulated therefrom adapted to provide support for said sample holder assembly and to electrically connect with said sample holder assembly, an electrical conductor extending from said supporting means through said housing adapted to electrically connect said supporting means to a voltage source, a shaft sealingly slidable in said first passage adapted to engage with said insulator member, a third passage in said closure member extending between said housing exterior and said first passage, said exterior end of said third passage being adapted to communicate with a controlled vacuum source and the inner end of said third passage communicating with said first passage between said valve seat and said housing exterior.

10. A vacuum lock solid sample source device for mass spectrometers comprising a housing including a closure member having a passage therein extending between the interior and exterior of said housing, valve means positioned in said closure member for closing off said passage intermediate its length thereby isolating the interior from the exterior of said housing, a removable sample holder assembly positioned in said housing, a support plate engageable with said sample holder assembly and electrically insulated from said housing, said plate having an opening through which said sample holder assembly extends, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a voltage source, a shaft sealingly slidable in said passage adapted to engage with said insulator member, and vacuum source means communicating with said passage adapted to apply a vacuum to said passage.

11. A vacuum lock solid sample device for mass spectrometers comprising a housing including a closure member having a passage therein extending between the exterior and interior of said housing, a valve means positioned in said closure member for closing off said passage intermediate its length thereby isolating the interior from the exterior of said housing, a removable sample holder assembly positioned in said housing, a support plate engageable with said sample holder assembly and electrically insulated from said housing, an electrical conductor extending from said plate through said housing adapted to electrically connect said plate to a voltage source, a shaft sealingly slidable in said passage adapted to engage with said insulator member, and vacuum source means communicating with said passage adapted to apply a vacuum to said passage.

12. A vacuum lock solid sample source device for mass spectrometers comprising a housing, a first passage in said housing extending between the interior and exterior thereof, means arranged in said first passage adapted to close off and open fluid communication through said passage, a second passage in said housing extending between said means and the exterior of said housing, sample holder means positioned within said housing, stationary supporting means mounted in said housing and insulated therefrom adapted to support and electrically connect with said sample holder means, an

electrical conductor extending from said supporting means adapted to connect said sample holder means with a voltage source, sample replacement means sealingly slidable in said first passage engageable with said sample holding means for removing and replacing samples, and vacuum source means communicating with said first passage adapted to apply a vacuum through said first passage.

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