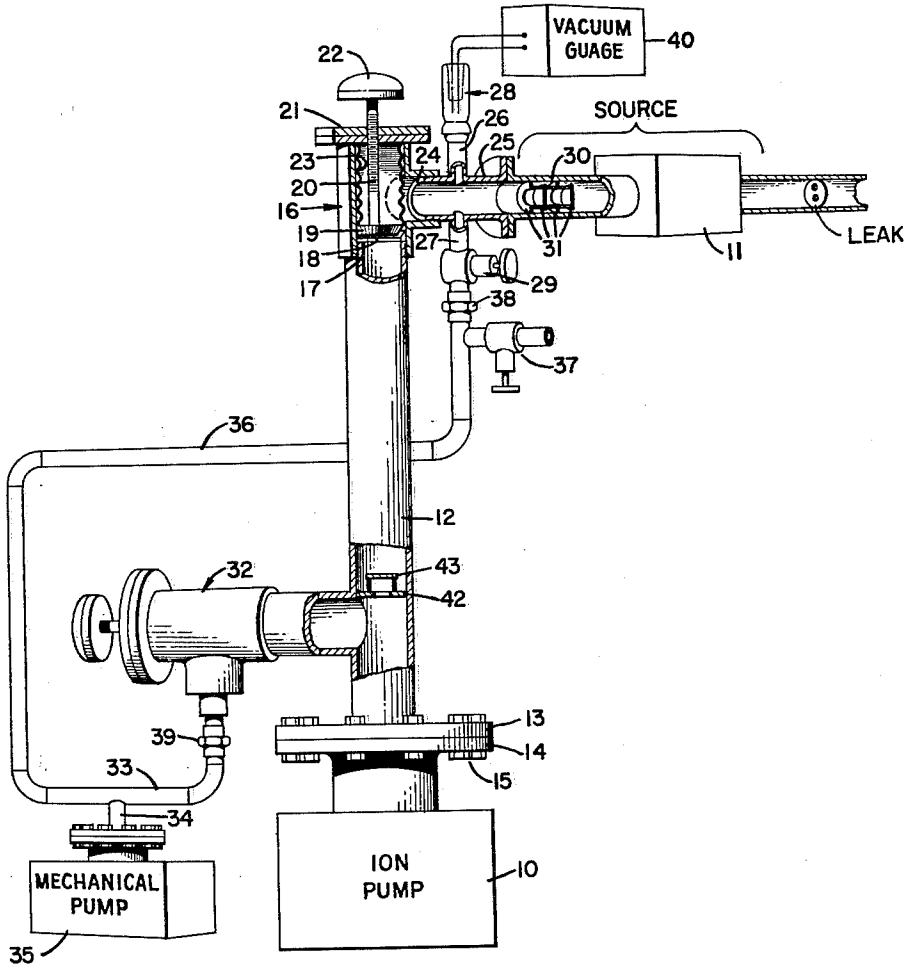
Aug. 21, 1962 E. W. BOYER ET AL 3,050,622

METHOD AND APPARATUS FOR CONNECTING A GETTER-ION PUMP TO AN ANALYTICAL MASS SPECTROMETER

Filed Oct. 3, 1960



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United States Patent Office 3,050,622 Patented Aug. 21, 1962

3,050,622

METHOD AND APPARATUS FOR CONNECTING A GETTER-ION PUMP TO AN ANALYTICAL MASS SPECTROMETER

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Filed Oct. 3, 1960, Ser. No. 59,870
6 Claims. (Cl. 250—41.9)

This invention relates generally to the adaptation of an ion vacuum pump to an analytical mass spectrometer and in particular to the hardware used to connect the ion vacuum pump to the analytical mass spectrometer.

This application refers to the novel construction of hardware for connecting an ion pump to an analytical mass spectrometer. The method for operatably connecting an ion pump to an analytical mass spectrometer is described in greater detail in an application entitled "Method and Apparatus for Operating An Analytical Mass Spectrometer with a Getter-Ion Pump," by Ernest W. Boyer, filed concurrently with this application and assigned to the same assignee.

Since the development of the analytical mass spectrometer the diffusion pump has been the source for obtaining the high vacuum required for its proper operation. While the diffusion pump has been more than adequate in obtaining a satisfactory vacuum for the proper performance of the spectrometer, the industry has been plagued with the length of time that the pump requires to obtain an adequate vacuum, especially with condensable light hydrocarbon. Maximum or minimum nitrogen levels in the cold traps fail to solve the problem. Even when a programmed nitrogen level was used, the solution was far from satisfactory. The problem is further compounded by a compound such as ethane which is condensed to a volatile liquid under trap conditions and is in equilibrium with cold trap and source region. Under these conditions, it causes a variable background which can not be eliminated by simply subtracting the background as measured before recording a sample spectrum.

The ion pump, on the other hand, permits the elimination of the troublesome cold trap and thus permits direct coupling between the ion pump and source. The application of an ion pump to a mass spectrometer is not new; however, the use was limited solely to a spectrometer which was analyzing simple systems. Since the ion pump is species selective, that is, will pump one entity faster than another, it was determined to be impossible to apply the pump to an analytical mass spectrometer that obviously requires the pumping of widely different compounds. The molecules having different sizes and atomic structures would be operated on by the electronic stream of the pump differently. The pumping rate would, of course, be determined by not only the molecular size, that is, how large an area the molecule presented to the electronic stream, but also the electronic configuration. However, the ion pump creates new problems since it is species selective in its pumping rate. Thus, the ion pump will pump various entities at a different rate, thereby upsetting the ratio of the entities at the source and resulting in errors in the analysis of the mixtures under test. The ion pump has a further difficulty in that if the entire system requires venting for any reason, such as maintenance on the source, the system must be opened to the atmosphere. When the system is again evacuated, the ion pump is roughed to approximately 20 microns; when this pressure is reached, the power supply to the ion pump is energized. The nature of the pump is such that at operation at these pressures (about 5 microns) the pump heats up. At elevated temperatures, for some reason not completely understood, chemical entities previously

pumped are released from surfaces within the pump. These chemical entities so released can migrate to and contaminate the spectrometer source. These deposited entities will result in erroneous analysis of samples under test.

Entities, as used in this specification, shall mean any particles or combination of particles (molecules, atoms, etc.) in a charged or uncharged state.

It is, therefore, an object of this invention to disclose apparatus for connecting an ion vacuum pump to an analytical mass spectrometer.

It is a further object of this invention to provide unique apparatus that will isolate the mass spectrometer from the electronic pump during the initial start-up of said pump.

It is a still further object of this invention to prevent contamination of the analytical mass spectrometer source from material degassed or emitted from the pump during the start-up cycle.

It is another object of this invention to disclose a method of connecting the ion pump so that it will never become vented during the routine maintenance or use of the equipment, thus prolonging the life of the pump.

It is a further object of this invention to reduce pump-down time required for the analytical mass spectrometer after venting of the source.

It is a still further object of this invention to provide apparatus that will permit connection of an electronic pump to the usual analytical mass spectrometer in service today and further prevent interference of the ion pump magnet with th magnetic field of the analytical mass spectrometer.

This invention features hardware that is particularly adapted to connect an ion pump to an analytical mass spectrometer source and comprises a first pipe which has a flange on one end which is adapted to mate with the ion pump output and an isolation valve on the other end which is adapted to isolate the ion pump when the source is purged. A second pipe connects the isolation valve with the mass spectrometer source. Connected to both the first and second pipes and communicating therewith are two valves which may be jointly or selectively connected to a mechanical vacuum pump. When the source must be purged, the isolation valve is closed; the valve in the second pipe is opened and connected to the mechanical pump thereby permitting evacuation of the pipe prior to reopening the isolation valve. The valve connected to the first pipe, when connected to the mechanical pump, provides a method for evacuating the pipe prior to the operation of the ion pump.

The invention further features a method for eliminating secondary emission originating in the pump from migrating to the source by mounting the first and second pipes at right angles, thus the right angle bend tends to ground secondary emission particles leaving the ion pump, thereby preventing said secondary emission particles from entering the source and upsetting sample portions under test.

This invention also features baffles added to the line near the pump to aid in minimizing secondary emission from the pump after evacuation has occurred.

Further objects, features, and advantages of the invention will become apparent from the following description and claims when read in view of the accompanying drawing which is a partial sectional drawing of the hardware used to couple the ion pump to the spectrometer source.

Referring to the drawing, an ion pump 10 is shown connected to an analytical mass spectrometer source 11 through air-tight hardware which comprises a cylindrical tube 12 which has a flange 13 adapted to mate with an output flange 14 of the ion pump. Flanges 13 and 14 are held together by any suitable means such as a plurality

of bolts 15. A high vacuum valve 16 is connected to the end of cylindrical tube 12 opposite flange 13 by any suitable means such as shoulder 17. High vacuum valve 16 may be any suitable valve capable of maintaining an air-tight seal during operation in either a closed or open position. The type of valve 16 used is commonly available and briefly incorporates a valve seat 18, a valve head 19 adapted to seal the end of cylinder 12 when the valve head and seat are mated. A threaded valve stem 20 passes through valve end 21 and has one end rotatably attached to valve head 19 and the other end rigidly attached to a suitable handle or nob 22. In order to maintain an adequate seal at all times between the valve end 21 and valve head 19, a bellows 23 is sealably attached to the top of the valve and to the periphery of the valve head. Thus, any air escaping into the inner portion of the valve around the valve stem threads is confined within the bellows and does not enter the inside of cylindrical tube 12.

The output 24 of valve 16 is connected to a cylindrical portion 25. Portion 25 includes a pair of pipes 26 and 27, respectively, attached to its periphery. The first input 26 is connected to a vacuum gauge measuring means 28 and the second input 27 is connected to a valve 29. The output of cylindrical portion 25 is connected through a cylindrical portion 30 to spectrometer source 11. For convenience, cylindrical portion 30 is here shown to contain a labyrinth comprising a plurality of baffles 31 supported within cylindrical portion 30 by any suitable means. Also connected to cylindrical portion 12 is a second valve 32 which is shown to be a valve similar to valve 16 and, therefore, will not be described. The size of valve 32 permits rapid evacuation of the chamber within cylindrical tube 12; however, it is obvious that other types of valves may be used for either valve 32 or valve 16 providing they are sufficiently air-tight during their operation in either the open or closed position. Connected to the outlet of valve 32 is a pipe 33 which is connected to the exhaust exit 34 of a mechanical pump 35. A pipe 36 is connected between the outlet of valve 29 and exhaust exit 34 of mechanical pump 35. A valve 37 is connected between pipe 36 and the air to permit venting of the spectrometer source in the event that work must be done on the unit when the mechanical pump is connected. Mechanical pump 35 and pipe 36 may be disconnected by including a disconnect 38 and 39, thereby freeing the pump for other uses in the laboratory. Annular rings 42 and diaphragm 43 are rigidly attached within cylindrical tube 12 and aid in the elimination of secondary emission entities.

Operation

Referring to the drawing, with valve 16 in an open position ion pump 10 evacuates cylindrical tube 12, cylinder portions 25 and 30, and spectrometer source 11; both valves 32 and 29 under normal operation are operated in a closed position thereby preventing the entry of air into the evacuated system. Inserted within tube portion 30 is shown a plurality of baffles or diaphragms 31. These baffles show one method of increasing the source conductance so that the pump can not pump each species in the analytical spectrometer source at a different rate. Other methods of controlling the source conductance are possible, as for example, a plurality of right-angle bends. When for some reason the source must be shut down and the system vented, valve 16 is closed. This will permit continuous operation of pump 10, and will maintain cylindrical tube 12 in a highly evacuated state, thereby greatly reducing the time required to pump-down the system when the work on the source is completed. If mechanical pump 35 is connected in the manner shown in the drawing, that is, pipe 36 is connected to valve 29, valve 37 must be opened in order to let air into the system. Once the work is completed, valve 37 is closed and the mechanical pump energized. Operation of the mechanical pump will then partially evacuate the spectrometer source and cylinder portions 25 and 30, thus clearly reducing the work required by the ion pump, and likewise, greatly increasing the speed that the system can be totally evacuated once the ion pump is connected to a spectrometer source. When vacuum gauge 40 indicates that the vacuum is as well as can be obtained by the mechanical pump, valve 29 is closed and valve 16 opened. The remaining air in tube portions 25 and 30 and spectrometer source 11 will be brought to operating vacuum. If for some reason the ion pump must be changed or replaced thereby requiring that cylindrical tube 12 be purged, rapid evacuation is obtained by connecting mechanical pump 35 through pipe 33 to valve 32. When valve 32 is opened and mechanical pump 35 actuated, the system is rapidly brought to a near operating rate by the mechanical pump. When this occurs, valve 32 is closed and the ion pump energized and the system brought rapidly to its operating vaccum.

Valve 16, while operating as a valve, also presents a unique feature not apparent. When, for example, the analytical mass spectrometer has been used to test samples of hydrocarbon and ion pump 10 has been used for a period of time, hydrocarbon molecules will become imbedded in the titanium surface on the inside of ion pump 10. If for some reason, as previously explained, the ion pump is disconnected or cylindrical tube 12 is filled with air, these molecules must then be evacuated when the pump is again re-energized. When the pump is initially started, the elevated temperatures which develop cause a release of entities previously pumped which may migrate up cylindrical tube 12 and back to source 11. This emission or migratory molecules if permitted to reach source 11 would obviously introduce errors during analysis. Further secondary emission entities released by the pump during normal operation would enter the source and contaminate same if the pump and source were in "line of sight" with each other. This, however, is eliminated in our unique construction by placing cylinder 25 at right angle to cylinder 12. Thus, as secondary emission entities leave ion pump 10, they will be inhibited from entering cylinder 25 by the right-angle bend. Thus, as the molecules travel up cylindrical tube 12, they will strike valve head 19 causing them to become grounded or deflected and will migrate back to pump 10. It is obvious that if valve 16 is not included in the system that a baffle or diaphragm or series of annular rings or baffles could be inserted within cylindrical tube 12 and accomplish the same results. Further, cylindrical tube 12 may be bent at right angles and thereby result in the grounding or deflecting of the charged or uncharged entities, the end result being, of course, to prevent the stray entities from migrating back to the spectrometer source and causing an interference in analysis.

While annular ring 42 and diaphragm 43 are shown in cylindrical portion 12, they may be eliminated if valve 16 provides sufficient isolation for the sample under test. If valve 16 is eliminated, rings 42 and diaphragm 43 would be necessary to eliminate the secondary emission entities. Rings 42 and diaphragm 43 combine to form a deflection means such that entities traveling from the ion pump back toward the source will strike the diaphragm or rings and be grounded or deflected from their straight-line path resulting in their being drawn back to the pump rather than migrating on toward the source.

This invention has described in detail the unique hardware used to connect an ion pump to an analytical mass spectrometer; however, the particular hardware can be readily adapted to other uses. For example, it provides an effective method of connecting an ion pump to an electron microscope or to any mass spectrometer or time-of-flight mass spectromer since in all cases the unique problems presented by the ion pump are present.

Valve 16 will provide an easy method for preventing the ion pump from being exposed to the atmosphere, thereby

extremely prolonging its life. Further, the special valves **29** and **32** provide an additional method of extending the life of the ion pump by permitting evacuation of the system prior to operation of said pump.

While the preferred embodiments disclose circular tubing, it is obvious to one skilled in the art that other forms and shapes of tubing may be employed without departing from the spirit and scope of this invention and that the principle of operation of the apparatus regardless of its general physical appearance would represent the true invention.

Although this invention has been described with respect to particular embodiments thereof, it is not to be so limited, as changes and modifications may be made therein which are within the spirit and scope of the invention as defined by the appended claims.

We claim:

1. Apparatus for retaining a vacuum on a mass spectrometer tube, comprising:

an ion pump;

pipe means connecting the ion pump to the spectrometer tube; and

electrically conductive obstruction means in the pipe means electrically grounded to the spectrometer tube and positioned to prevent line-of-sight communication between the ion pump and the spectrometer tube, whereby

charged entities tending to migrate from the ion pump to the spectrometer tube are grounded by the obstruction means and are drawn back to the ion pump

to prevent erroneous operation of the spectrometer tube.

2. Apparatus as defined in claim 1 wherein the obstruction means comprises a bend in the pipe means.

3. Apparatus as defined in claim 1 wherein the obstruction means comprises a baffle assembly.

4. Apparatus as defined in claim 1 wherein the obstruction means comprises a valve interposed in the pipe means.

5. Apparatus as defined in claim 4 characterized further to include a mechanical pump, and a second valve connecting the mechanical pump to the pipe means between the first mentioned valve and the spectrometer tube, whereby the spectrometer tube and the pipe means between the spectrometer tube and the first mentioned valve may be evacuated by the mechanical pump upon closure of the first mentioned valve while continuing the operation of the ion pump.

6. Apparatus as defined in claim 5 characterized further to include a third valve connecting the mechanical pump to the pipe means between the ion pump and the first mentioned valve, whereby the mechanical pump may be used to selectively evacuate the pipe means between the ion pump and the first mentioned valve prior to start-up of the ion pump.

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