This invention relates generally to method and apparatus for controlling subatmospheric pressures and more particularly it relates to method and apparatus for introducing samples of gas, vapor or vaporizable material into a mass spectrometer.

The use of mass spectrometers for routine process control has been hindered by the inconvenient and often time-consuming extraction and transportation of the samples from the sample locations to the spectrometer. Additional time and effort has been necessary in spectrometer operation to purge traces of earlier samples and most of this effort was required to be performed manually. If the number of positions to be sampled is considerable these operations may become so uneconomical of labor and time-consuming as to force reliance on other, perhaps less sensitive methods or apparatus. This may occur especially where a process requires rapid and delicate control.

It is one object of this invention to provide method and means for automatically sampling and introducing samples into a mass spectrometer.

Another and somewhat broader object of the invention is to control the pressure at the sample entry into a mass spectrometer.

A general object of the invention is to introduce gasiform samples sequentially into a mass spectrometer without having to break the vacuum therein.

A more general object of the invention is to provide new and improved method and apparatus for controlling gas pressures at relatively high vacuum.

A further object is to provide an economically operated system for rapid analysis by mass spectrometer.

A still further object is to provide means for automatically sampling a gas, vapor or particularly a vaporizable material and introducing a solids-free sample into a mass spectrometer.

An additional object is to provide a mechanically simple, automatic system for purging a mass spectrometer.

Additional objects and the many advantages of this invention will appear hereinafter.

In a broad sense, the heart of our invention is a flow system for controlling the pressure of a flowing gas at a high vacuum, such as at the sample entry to a mass spectrometer. This flow system comprises two flow restrictions positioned in the flow stream on opposite sides of the region in which it is desired to control the pressure. Upstream of the first flow restriction, the pressure is maintained constant at a value substantially more than twice the desired pressure while the pressure downstream of the second flow restriction is kept at any pressure, constant or variable, whose value is always substantially less than one-half the desired pressure. By this relatively simple and very effective device, the pressure in the region between the flow restrictions is maintained constant at a value determined by the highest pressure in the system, that upstream of the first flow restriction.

The invention is illustrated in the accompanying drawings which are to be used for reference with the followingly description with the understanding that it is not intended to limit the disclosure to the details found therein. In the drawings:

Figure 1 is a diagrammatic representation for explaining the theory of operation of the pressure controller provided in accordance with this invention;

Figure 2 is a representation, mainly diagrammatical, of an entire system for sampling a liquid at a number of separate locations and introducing the samples in sequence into the mass spectrometer;

Figure 3 depicts a preferred arrangement of equipment for manifolding mass spectrometer samples in accordance with this invention, and

Figure 4 is a cross-section on line 4—4 of Figure 3 of the sample manifold in accordance with this invention and illustrates a preferred arrangement.

PRESSURE CONTROLLER

This invention employs flow restrictions under pressure conditions that are termed the "critical pressure ratio," a ratio of the downstream pressure on the flow restriction to the pressure upstream thereof. The value of this ratio varies somewhat, but is always approximately one-half; for example, the value of the ratio is about 0.58 for saturated steam and 0.55 for superheated steam. When the ratio is less than about one-half, that is, when the downstream pressure is less than about one-half the upstream pressure, the velocity of the gas through the throat of the flow restriction reaches a maximum value, the speed of sound in the gas in the orifice throat. So long as the critical pressure ratio is not exceeded, the flow through the flow restriction is dependent solely on the upstream pressure and is independent of the downstream pressure. The principle and its application in the present invention will be illustrated with reference to Figure 1. For convenience, flow restrictions to be operated under such conditions are referred to herein as to pass a gas at its acoustic velocity. It should be noted that in this specification and the claims appended, the word "gas" is used to include any gasiform material, and particularly includes vapors.

In Figure 1 there is shown a conduit 10 through which gas is being pumped in the direction indicated by the arrows by a vacuum pump 28. It is desired to control the pressure in the region 22 of the conduit 10. The region 22 is separated from the remainder of the conduit by an upstream orifice plate 12 having an orifice 16 and a downstream orifice plate 14 having an orifice 18. Upstream of orifice plate 12, conduit 10 contains a pressure controller or regulator 26 of any suitable type. There are thus formed in conduit 10, in addition to the region or chamber 22, an upstream chamber 20 between orifice plate 12 and pressure controller 26 and a downstream chamber 24 between orifice plate 14 and pump 28. The selection of these names is based on the direction of flow of a gas through the chambers, as shown by the arrows in the drawing, that is, first through the upstream chamber 20, thence through orifice 16 from chamber 20 into the intermediate chamber 22 where the pressure is to be controlled, thence through orifice 18 from chamber 22 into the downstream chamber.

In accordance with the principles already outlined, when the pressure in upstream chamber 20 exceeds twice the pressure in intermediate chamber 22, the flow of gas through orifice 16 is independent of the pressure in intermediate chamber 22, that is, downstream of the orifice 16. The velocity of the gas in the throat of orifice 16 is the speed of sound in the gas. If the pressure in chamber 20 be maintained constant by the pressure regulator 26, the mass rate of flow into chamber 22 will be constant regardless of the pressure in chamber.
and will be determined by the pressure in chamber 20 for the particular orifice 16. Similarly, when the pump 28 evacuates downstream chamber 24 to a pressure less than one-half that existing in intermediate chamber 22 and the pressure in intermediate chamber 22 is constant, the mass rate of flow through orifice 18 into downstream chamber 24 will be constant. Or, stated in another way, if the mass rate of flow through orifice 18 is constant under these pressure conditions, then the mass rate of flow upstream of orifice 18 in intermediate chamber 22 must be constant. Under these conditions, because there is a constant mass rate of flow through both of the orifices 16 and 18, the pressure in chamber 22 must be constant. The pressure in chamber 22 will remain constant regardless of fluctuations in pressure downstream of orifice 18 caused, for example, by pump 28, or in pressure upstream of the pressure regulator 26.

This principle is especially useful where conditions are such that pressure control is difficult or expensive but another set of conditions may be used that are easier to control. Typical of the difficult conditions is a vacuum, say, of the order of microns or tens of microns, that it may be desired to maintain in chamber 22. Controllers for pressure in this range are not readily available and in any case are exceedingly expensive. Controllers for higher pressures, for example, those that may be maintained in upstream chamber 20, are relatively easily obtainable. It is necessary only to choose orifice sizes that will maintain the required pressure conditions and the desired rate of flow.

The pressure ratios of two or of one-half that are used herein are approximate since the ratios vary somewhat with the gas flowing. In any event it is desirable that the values employed differ substantially from the minimum of two or maximum of one-half. If the ratio used is too close to this limit, a minor pressure fluctuation may cause the limit to be surpassed, reducing the gas velocity in the orifice throat below the acoustic velocity.

**SAMPLING SYSTEM**

Figures 2, 3 and 4 illustrate apparatus for sampling a number of locations in a plant concentrating deuterium in water at a pressure of the order of tens of atmospheres and for introducing the samples at subatmospheric pressure into a mass spectrometer. As shown, there is a conventional mass spectrometer 30 including a magnet 32, analyzer tube 34, vacuum pump 46 and collector 56. The mass spectrometer is connected to the usual preamplifier 38, amplifier 40 and suitable recorder 47.

The mass spectrometer ion source to which the samples are presented is connected by a sample introduction conduit 44 to a conduit 45 in turn connected to the positions to be sampled. The conduit 45 is evacuated by a pump adapted to maintain a high vacuum in conduit 45, such as a diffusion pump 62 backed by a mechanical vacuum pump 63. It is desired to control the pressure in conduit 44 and in the region of conduit 45 about its junction with conduit 44. Orifice plates 56 and 58 are accordingly positioned in conduit 45 spaced on opposite sides of conduit 44 to form a sample chamber 48 as the region in which the pressure is to be controlled. Orifice plate 56 is upstream of conduit 44 with respect to pump 62 while orifice plate 58 is downstream thereof. Sample chamber 48 communicates at its upstream end through a conduit 52 with a sample manifold 50. At its downstream end, sample chamber 48 communicates with pump 62 so that there is formed between orifice plate 58 and pump 62 an evacuable chamber or compartment 64. This arrangement of the manifold 50, sample chamber 48 and downstream chamber 64 connected in series through orifices 56 and 58 is substantially the same as Figure 1; the flow from chamber 48 into the mass spectrometer is small enough not to affect the pressure conditions in the three chambers. In effect the sample flow into the mass spectrometer through conduit 44 is bled as a side stream from the continuous, relatively large flow through manifold 50 and chambers 48 and 64, thus assuring an effective and rapid purge of the sample chamber 48. Now if the pressure in the manifold 50 is maintained constant at more than about two-thirds of sample chamber 48 while downstream chamber 64 is evacuated to a pressure below about one-half the pressure in sample chamber 48, the sample chamber pressure will be constant. It is highly desirable that the volume of conduit 44 be kept at a minimum in order that it be continuously purged. The purge may be accomplished by means of pump 46 or pump 62 and a suitable by-pass conduit.

The sample manifold 50 communicates with a plurality of sources of high-pressure liquid samples through conduits 66 of which three, designated 66a, 66b and 66c, are shown for convenience although any number may be used for sampling purposes depending on the available cycle time and on the difficulty in arranging the conduits in the space available. Preferably the manifold 50 is cylindrical, as shown in Figures 3 and 4 and sample conduits 66 enter the manifold radially. Each conduit 66 contains in series a liquid filter 68, a helical coil 70 of capillary tubing to reduce the liquid pressure and a steam-heated vaporizer 72 in that order from the sampling location. While the coil of capillary tubing is preferred, any type of pressure reducer may be used. Similarly, other heating apparatus that does not alter sample composition may be employed to that end. The preferred liquid filter is of the disc type but other types may be employed. This equipment is necessary in sampling a liquid under high pressure, but all or part may be omitted or other suitable apparatus substituted if the material is inert and under lower pressures, a vaporizable solid or initially a vapor or gas. Such modifications will be apparent to those skilled in the art.

Between the vaporizer 72 and the manifold 50, relatively close to manifold 50, each conduit 66 contains a vapor filter 74 which may be simply a packed bed of glass fiber or of other fibers resistant to temperature used. With water vapor, an acrylic fiber may be used at temperatures of the order of 125° C. Downstream of the filters 74 all the conduits 66 are vented to the atmosphere through a common vent line 76, and an atmospheric condenser may be provided in the vent line to recover a valuable product. The atmospheric vent permits the use of barometric pressure as a reference pressure, as explained hereinafter. Between the vent 76 and manifold 50, each conduit 66 contains a cut-off valve 82 which may be operated by a timer 80. Any suitable automatic sequencing timer may be used with solenoid or diaphragm valves or any other convenient arrangement to open the valves 82 in the proper sequence and admit a sample to the manifold 50. The valves 82 may also be manually operated, if desired. Preferably the portions of the conduits carrying vapor are steam heated and insulated to prevent condensation and adsorption on the walls of the conduits.

Between the valves 82 and the manifold 50 each conduit 66 also contains an orifice 84 which is employed to regulate the pressure in manifold 50. Each orifice 84 is operated in the manner described for orifice 56, that is, if the pressure downstream thereof, in manifold 50, is less than one-half the upstream pressure, the barometric pressure in this case. Since the pressure downstream of orifice 56 in sample chamber 48 is less than one-half that in manifold 50 upstream of orifice 56, the pressure in manifold 50 will remain constant as long as the pressure upstream of orifice 84, barometric pressure, is constant.

In other words, four levels of pressure are maintained in series: barometric pressure upstream of orifice 84, in manifold 50 less than one-half barometric pressure, in sample chamber 48 less than one-half the pressure in manifold 50 and in downstream chamber 64 less than one-half the pressure in sample chamber 48. Barometric pressure is relatively constant, rarely varying more than
about 3% from normal, and the combination of the atmospheric vent 76 and orifices 84 and 56 serves very efficiently to maintain the pressure constant. However, remember for the manifold chamber 50 to maintain its pressure constant and, therefore, to maintain the desired pressure in sample chamber 48, as explained hereinafter. For closer regulation, the vent 76 and orifices 84 may be replaced by any suitable automatic pressure regulator, or alternatively, the pressure may be controlled by a manually operated throttle valve in response to pressure gauge readings.

INTRODUCTION OF A CALIBRATING STANDARD

To maintain the accuracy of mass spectrometer analyses, it is usually necessary to introduce a standard sample periodically for calibration. This may most conveniently be done by treating the calibration as simply another sample to be analyzed and this is the preferred method. As shown in Figure 2, the standard is introduced into the manifold 50 through conduit 66d containing cut-off valve 82d connected to the timer 80. Downstream of valve 82d, conduit 66d contains orifice 84d adapted to be used under critical-pressure-ratio conditions, as described hereinafter. In all respects the apparatus for introducing the standard sample may be the same as that used for routine analysis. However, if, as in the case of heavy water, the standard may be a pure sample whose vapor is easily produced at atmospheric pressure, the barometric vent 76, the filters 68 and 74, and the capillary 70 may be omitted. In the case of heavy water, the standard may be simply produced by evaporation, for example, in a flask 90 fitted with a reflux condenser 100 vented to the atmosphere. The flask may be heated with a suitable heater 102 and is connected in any convenient manner with the standard-sample conduit 66d.

PURGING SYSTEM

A mass spectrometer has a considerable "memory" effect; that is, after one sample is analyzed, its residue in the spectrometer and associated parts interferes with the analysis of subsequent samples and erroneous results may be obtained. This difficulty may be reduced by evacuating the spectrometer and the orifices 84. For reasons to be adverted to later, it is important that the distance from the junction of the tubes 88 and conduits 66 to the manifold 50 be the same in each of the conduits 66, as shown in Figures 3 and 4. Similarly, it is important that the lengths and diameters of all of the tubes 88 be the same. (Some of the conduits 66 and tubes 88 are omitted in Figure 3 for clarity.)

Purge chamber 86 is connected through a conduit 90 to a vacuum pump 92. Between purge manifold 86 and pump 92, the conduit 90 has an orifice 94; an evacuable chamber or compartment 96 is thereby formed between orifice 94 and pump 92. By means of pump 92 the pressure in chamber 96, i.e., downstream of orifice 94, is maintained at a value less than one-half that in purge manifold 86. Thereby, the purge manifold 86 and the sample manifold 50 together with the interconnecting tubes 88 form a single volume delimited by orifices 84, 94 and 56. With the downstream pressures (in sample chamber 48, and in chamber 96 downstream of orifice 94) less than one-half the pressure in this volume and the upstream pressure (barometric pressure) more than twice the pressure in the volume, the upstream pressure determines the pressure in the volume. Again, since barometric pressure is substantially constant, the pressure in the entire volume will be maintained at a substantially constant level although it may vary from point to point because of the pressure drop in the flowing gas, as explained below.

It should be noted here that the pressure in the two manifolds is not affected by the fact that the pressure downstream of one may be different from that downstream of the other, i.e., in chamber 48, and in chamber 96 downstream of orifice 94. For as long as the critical pressure ratio is maintained across the orifices 56 and 94, the manifold pressures are determined by the pressure upstream of orifice 84 only. Moreover, so long as the pressure in the manifolds is constant, the relative mass rates of flow of gas from sample manifold 50 and from purge manifold 86 are determined by the relative sizes of the orifices 56 and 94, respectively.

The purge system also serves another important purpose. Without it, pump 62 would be required to maintain the entire mass of gas flowing from the higher pressures in the sampling conduits 66 into sample manifold 59. On the other hand, in this embodiment, pump 62 is also required to maintain a pressure of a few microns or less. In order to achieve both purposes, flow restrictions of such small size would be required as to cause difficulty in fabrication. However, when the present purge system is used, it handles most of the mass of gas flowing into manifold 59 while only a relatively small mass of gas is withdrawn therefrom by pump 62. For this purpose the purge system may employ a pump with a high mass-flow capacity, for example of the mechanical type, while maintaining a convenient pressure in manifold 50 for withdrawal of the side stream of sample gas. The necessary flow restrictions are then easily fabricated.

OPERATION OF THE SYSTEM

In the operation of the system the pumps are started and the system is evacuated with all the valves closed. Then one of the valves, say 84a, is opened and a liquid sample is drawn from the first sample point into conduit 66a. As the sample flows through conduit 66a, it is initially filtered as a liquid in filter 68a, and the pressure is reduced by flowing through the capillary tubing 70a. The filtered, relatively low-pressure liquid then flows through the heater 72a which converts it to a vapor, and the vapor passes through the vapor filter 74a where any residual solids are removed. This vapor flow is at atmospheric pressure because of the vent 76. The vapor then fills the manifolds 50 and 86 and a minor fraction of it flows through sample chamber 48 whence a small amount of gas is withdrawn into the mass spectrometer for analysis.

When the analysis is complete, as indicated by a steady reading on the recorder 42, valve 84a is closed and valve 84b, the next in the series is opened. A liquid sample is withdrawn from the second sample point. The second sample flows successively through liquid filter 68b, capillary 70b, heater 72b and vapor filter 74b, arriving at valve 82b as a purified vapor at atmospheric pressure.

The flow of vapor is continued to sweep all vestiges of the prior sample from the sample manifold 50, sample chamber 48 and the mass spectrometer. The purge is accomplished by flowing the major portion of the vapor through manifold 50. Actually, all of the vapor flowing in conduit 66b enters manifold 50 except a minor fraction that is diverted through tube 88b into purge manifold 56. Then, except for the small amount withdrawn into sample chamber 48, the remainder of the vapor flowing through conduit 66b sweeps through manifold 50 and flows through conduits 66c, 66d and 66e into the other small diameter tubes 88c, 88d and 88e and into purge manifold 86 whence it is exhausted by pump 92. This arrangement assures that, while only a relatively small quantity of gas or vapor is required for analysis, a relatively large quantity is avail-
able for purging the sample manifold and sample chamber. Also with most of the gas flowing from manifold 50 through conduits 66a, 66c and 66d toward the sample points these conduits are effectively purged and sealed so that any gas leaking through valves 82a, 82c and 82d is prevented from reaching the sample manifold. The volume of gas required for this last purpose is a factor to be considered in determining the number of samples that may be introduced by means of a single system.

SELECTION OF THE PURGE TUBES

As noted before, the sample vapor flowing through one of the charging conduits, such as 66b, is divided among the tubes 88. Except for a minor fraction diverted before reaching manifold 50, all the sample vapor flows into sample manifold 50. The fraction of the sample vapor carried by each of the tubes 88 is determined by the number of sample points. To assure a substantially equal division of the sample vapor among the tubes 88, they should be of equal length and diameter and should have the same position relative to each other in order to establish substantially the same pressure drop across all the tubes. Obviously, in handling a large number of samples, the fraction of the sample vapor diverted through a tube 88 before reaching the sample manifold will be smaller so that the proportion of the sample available for purging. Thus, if there are n sample entry ports into the sample manifold 50, the fraction of gas diverted from a sample stream, such as through conduit 66b, is equal to 1/n. Besides effectively preventing leakage through valves 84 from reaching the sample manifold 50, this system has the principal advantage of requiring no additional valves at all in order to purge the sampling system.

The diameter of the tubes 88 required to effectuate the desired division of a sample may be calculated quite simply. The pressure drop between one of the conduits 66 and purge manifold 86 through one tube 88 carrying 1/n times the flow through conduit 66 (including exit and entrance losses) is set equal to the pressure drop of the remainder of the gas through manifold 50 (including entrance and exit losses). In mathematical symbols, this equality is as follows:

\[
\frac{16W_2^2}{\rho D_2 y} + \frac{16W_2^2}{\rho D_2 y} + 128L_2 W_2 a = \frac{128L_2 W_2 a}{\rho D_2 y} + \frac{128L_2 W_2 a}{\rho D_2 y} + 16W_2^2 + 16W_2^2 - \frac{\rho D_2 y}{\rho D_2 y} - \frac{\rho D_2 y}{\rho D_2 y}
\]

In this equation, \(W\) equals the flow through one tube 88; \(W\) equals the flow into the sample manifold 50, that is, the total flow in a conduit 66 less the amount diverted through a tube 88 prior to reaching the sample manifold or for a tube \(W = (\pi - 1) W\). \(D_2\) equals the diameter of each tube 88; \(D_3\) equals the diameter of each conduit 66; \(L_2\) equals the length of each tube 88; \(L_3\) equals the length of each conduit 66 between its junction with the corresponding tube 88 and its entry into manifold 50; \(\mu\) equals the viscosity of the gas; \(\rho\) equals gas density; \(M\) equals the molecular weight of the gas; and \(g\) is a conversion constant numerically equal to the acceleration due to gravity. It should be noted that to use the foregoing equation, it is necessary that the diameters and lengths of tubes 88 be equal and that the portions of conduits 66 between their junctions with tubes 88 and their entries into manifold 50 be of equal diameters and of equal lengths.

In practice, to assure good distribution of the gas through manifold 50 during the purge and supply portions of the sampling cycle, it is advisable to choose tubes 88 of such diameter that the pressure drop therein is considerably larger, say ten times, than the pressure drop through manifold 50. This factor may be introduced simply by multiplying the right side of the equation thereby.

SPECIFIC EXAMPLE

In a specific embodiment of our invention, the orifice and tube sizes shown in Table 1 were employed: these dimensions were used to extract and analyze samples of water enriched in deuterium. There were eight sampling locations and the samples were initially liquid at about 250 pounds per square inch pressure.

<table>
<thead>
<tr>
<th>Position</th>
<th>Pressure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Upstream of orifice 84</td>
<td>One atmosphere</td>
</tr>
<tr>
<td>Downstream of orifice 84</td>
<td>21.8 mm. mercury</td>
</tr>
<tr>
<td>Sample manifold 50</td>
<td>58.3 mm. mercury</td>
</tr>
<tr>
<td>Sample chamber 68</td>
<td>89 microns</td>
</tr>
<tr>
<td>Chamber 64</td>
<td>2 microns or less</td>
</tr>
<tr>
<td>Purge manifold 86</td>
<td>29.8 mm. mercury</td>
</tr>
<tr>
<td>Outstream 88</td>
<td>1 mm. mercury or less</td>
</tr>
</tbody>
</table>

The response time of this embodiment was about 31 minutes, as measured from the conclusion of one analysis to the conclusion of the next. The measured time included the steps of extracting a sample, flowing the sample through a conduit 66, purging the sample manifold, sample chamber and mass spectrometer and the normal operation of the mass spectrometer. The time was measured for water samples differing in deuterium content by a factor of more than two. The pressure in sample chamber 48 remained constant at 80 microns within three percent.

While our disclosure has been directed to an embodiment in which a mass spectrometer is employed, those skilled in the art will realize that our method and apparatus in its broader aspects are of wider application, particularly wherever a constant but low pressure of the order of microns must be maintained. For example, in any region maintained at a high vacuum into which a gas must be bled at a constant desired pressure, the region may be connected to a conduit between two orifices adapted to pass a gas at its acoustic velocity and the pressure upstream of the first orifice maintained constant at more than twice the desired pressure while that downstream of the second orifice is at less than one-half the desired pressure. An example of such an application is a particle accelerator, or any other device wherein an ion source is employed at high vacuum.

Accordingly, the foregoing description is illustrative of a preferred use of the invention, and it is to be interpreted as illustrative only and not in any limiting sense. Rather, it is intended that the scope of the invention should be delimited only by the scope of the claims hereinafter set forth.

What is claimed is:

1. Apparatus for introducing gas samples into a region of high vacuum from a plurality of sources comprising a sample manifold, a plurality of conduits connecting said
manifold with sample sources, a valve in each said conduit for introducing said samples in sequence, a sample manifold and sample chamber in series, a flow restriction between said sample manifold and said sample chamber, a second flow restriction between said sample chamber and said evacuating means and means for controlling the pressure in said sample manifold at more than twice that in said sample chamber, said evacuating means being adapted to maintain the pressure downstream of said second flow restriction at a value less than about half that in said sample chamber, said flow restrictions being adapted to pass said gas at its acoustic velocity.

2. The apparatus of claim 1 in which the valves are automatic and which includes a timer operatively connected to said valves and adapted to open said valves in sequence.

3. The apparatus of claim 1 including a source of a standard sample for calibrating said mass spectrometer periodically, a conduit connecting said standard source with said sample manifold and a valve in said standard conduit for introducing said standard sample in sequence with the samples to be analyzed.

4. The apparatus of claim 1 adapted for sampling a vapor material comprising in each said conduit a vaporizer upstream of said valve for converting said material to a vapor.

5. Apparatus for removing samples from a plurality of sources of a liquid at high pressure and introducing the samples in the vapor state into a mass spectrometer comprising a mass spectrometer adapted to be maintained in evacuated condition during introduction of the samples, a sample manifold, a plurality of conduits connected to sample manifold with said sample sources, a pressure reducing means, a vaporizing means and a means for controlling in that order from the sample sources, each of said conduits being vented to the atmosphere downstream of said vaporizing means, a flow restriction in each said conduit between the vent and said sample manifold, a sample chamber communicating with said sample manifold and comprising a mass spectrometer adapted to be maintained in evacuated condition during introduction of said sample manifold, for evacuating said sample chamber to cause sample vapor to flow from one of said conduits through said manifold and said sample chamber in series, a second flow restriction between said sample manifold and said sample chamber and a third flow restriction between said sample chamber and said evacuating means, said flow restrictions being adapted to maintain the pressure in said sample manifold at less than one-half the barometric pressure but more than twice the pressure in said sample chamber and to maintain the pressure downstream of said second flow restriction at less than one-half that in said sample chamber, said flow restrictions being adapted to pass the sample vapor at its acoustic velocity.

6. In combination, a mass spectrometer adapted to be maintained in evacuated condition during sample introduction, a sample manifold, a plurality of conduits connecting said manifold with said sample sources, a sample chamber connected to said mass spectrometer and communicating with said manifold, means for evacuating said sample chamber to cause gas to flow through said manifold and said sample chamber in series, a flow restriction between said sample chamber and said evacuating means, pressure regulating means upstream of said first flow restriction adapted to maintain the pressure in said sample manifold at more than twice that in said sample chamber and means for purging said manifold, said sample chamber and said mass spectrometer between sample analyses, said evacuating means and said flow restrictions being adapted to maintain the pressure downstream of said second flow restriction at less than one-half the pressure in said sample chamber, said flow restrictions being adapted to pass a gas at its acoustic velocity.

7. In combination, a mass spectrometer adapted to be maintained in evacuated condition during sample introduction, a sample manifold, a plurality of conduits connecting said manifold with the sample sources, a sample chamber connected to said mass spectrometer and communicating with said manifold, means for evacuating said sample chamber to cause gas to flow through said manifold and said sample chamber in series, a flow restriction between said sample chamber and said manifold, a second flow restriction between said sample chamber and said evacuating means, pressure regulating means upstream of said first flow restriction adapted to maintain the pressure in said sample manifold at more than twice that in said sample chamber, said evacuating means and said flow restrictions being adapted to maintain the pressure downstream of said second flow restriction below one-half the pressure in said sample chamber, evacuating means communicating with said conduits upstream of said flow restriction and said additional flow restriction means between said conduits and the last said evacuating means, the last said evacuating means and said flow restricting means being adapted to maintain the pressure therebetween below one-half the pressure upstream thereof, said first and second flow restrictions and said flow restricting means being adapted to pass a gas at its acoustic velocity.

8. Apparatus according to claim 7 in which the pressure regulating means comprises an atmospheric vent connected to the sample conduits and an additional flow restriction in each of the sample conduits maintaining in each said conduit atmospheric vent and the sample manifold, said evacuating means and said flow restrictions being adapted to maintain the pressure in the sample manifold at less than one-half atmospheric pressure.

9. In combination, a mass spectrometer adapted to be maintained in evacuated condition, a sample manifold, a plurality of conduits connecting said manifold with the sample sources, a sample chamber connected to said mass spectrometer and communicating with said manifold, separate means for evacuating said sample chamber to cause gas to flow through said manifold and said sample chamber in series, a first flow restriction between said sample chamber and said manifold, a second flow restriction between said sample chamber and said evacuating means, pressure regulating means upstream of said first flow restriction adapted to maintain the pressure in said sample manifold at more than twice that in said sample chamber, said evacuating means and said flow restrictions being adapted to keep the pressure downstream of said second flow restriction less than one-half the pressure in said sample chamber, a purging manifold, a conduit communicating between each said sample conduit and said purging manifold and adapted to pass to said purging manifold a minor fraction of the flow through one of said sample conduits whereby the major part of the flow passes into said sample manifold, additional evacuating means for said purging manifold and a third flow restriction between said purging manifold and said additional evacuating means, said additional evacuating means and said third flow restriction being adapted to maintain the pressure downstream of said third flow restriction at less than one-half the pressure upstream thereof, said first, second and third flow restrictions being adapted to pass a gas at its acoustic velocity.

10. The apparatus of claim 9 wherein for n samples each purging conduit is adapted to pass 1/n times the total flow through the sample conduit to which it is connected, whereby the flow into the sample manifold from said sample conduit is (n-1)/n times total flow.

11. In combination, a mass spectrometer adapted to be maintained in evacuated condition during sample introduction, a sample manifold communicating with said mass spectrometer and adapted to be maintained at sub-
atmospheric pressure, sample conduits for delivering a
gas to said manifold and means for purging said mass
spectrometer including a purge manifold, purge conduits
interconnecting said purge and sample manifolds, each
of said purge conduits being adapted to divert directly
into said purge manifold a minor fraction of the gas
flowing from a sample conduit into said sample manifold
whereby the major fraction passes first through said
sample manifold and then through the remainder of said
purge conduits into said purge manifold and separate
means for evacuating said purge manifold.

12. The apparatus of claim 11 in which for $n$ samples
to be flowed into the sample manifold, each purge conduit
is adapted to divert $1/n$ times the total flow of a sample
into the sample manifold whereby $(n-1)/n$ flows first
into the sample manifold.

13. In combination, a mass spectrometer adapted to be
maintained in evacuated condition during sample intro-
duction, a sample manifold adapted to be maintained at
a reduced pressure and communicating with said mass
spectrometer, a sample conduit connecting each sample
source with said sample manifold, a flow restriction in
said sample conduit, means for maintaining a substi-
ately constant pressure upstream of said flow restriction
and means for purging said sample manifold and mass
spectrometer comprising a purge manifold, a purge con-
duit connecting said manifold with each said sample con-
duit between said flow restriction and said sample mani-
fold, said purge conduit being adapted to divert a minor
fraction of the gas flowing through the sample conduit
directly into said purge manifold whereby a major frac-
tion of the gas flows first into said sample manifold,
means for evacuating said purge manifold, a second flow
restriction between said evacuating means and said purge
manifold and adapted to maintain the pressure down-
stream of said second flow restriction below one-half that
in said sample and purge manifolds, said flow restrictions
being adapted to pass a gas to its acoustic velocity.

14. Apparatus for maintaining a desired, relatively low
pressure in a particular volume through which a gas is
flowed comprising a first flow restriction upstream of said
volume, a second flow restriction downstream of said
volume, pressure-regulating means located upstream of
said first flow restriction and comprising an atmospheric
vent and a third flow restriction between said atmospheric
vent and said first flow restriction, an evacuating means
downstream of said second flow restriction adapted to
reduce the pressure there to less than half the desired
pressure, said flow restrictions being adapted to pass said
gas at its acoustic velocity, and said evacuating means and
said flow restrictions being adapted to maintain the pres-
sure between said third and said first flow restrictions at
less than one-half the barometric pressure but more than
twice said desired pressure.

References Cited in the file of this patent

UNITED STATES PATENTS

2,569,032 Washburn ------------ Sept. 25, 1951