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ASTRONAUT BREATH ANALYZER

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ABSTRACT

A miniature vacuum system, comprising a molecular leak from atmospheric pressure, an ion pump, and a quadrupole mass spectrometer has been designed for use as a breath analyzer by an astronaut. It is to fit under his chin, inside his helmet. The most critical elements of the system, a molecular leak and an associated ion pump of small dimensions, have been assembled and have demonstrated capability to work together in a satisfactory manner. The leak utilizes an electro-polished tungsten needle in a gold foil seat. A large, mechanical actuator has been used to adjust the flow rate at values below 5×10^{-7} torr-liters/second. A thermally adjusted actuator, the size of a pencil eraser, has been designed and fabricated but not assembled. The external dimensions of the ion pump, including the magnetic field structure, are those of a 1-inch right circular cylinder. The quadrupole mass spectrometer has rods 2 inches long, with hyperbolic contours defined by $r_0 = 0.075$ inches. When excited by a laboratory oscillator at 6 megahertz, it provides unit resolution with flat topped peaks. An experimental assembly of the leak, 5-inch heated inlet tube, ion source, quadrupole and laboratory auxiliaries demonstrated a negligible lag time between the change of the composition of the gas at the inlet and the onset of the instrument response. The near completion of the step change (to within 10 percent) in composition of inlet gas in 120 milliseconds indicates a "time constant" of 52 milliseconds based on an exponential decay.

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INTRODUCTION

This report describes the work done by Earth Sciences on Contract NAS9-8371 with Manned Spacecraft Center, Houston, Texas. Technical monitoring of this work was accomplished by Paul W. Schlottman. As indicated by the Work Statement, this project concerns the development of a mass spectrometer breath analyzer system to be worn under the chin by an astronaut while on a mission in space.

A knowledge of the compositional change in an astronaut's breath between inhale and exhale is of great value to the medical people who are observing the physical condition of the man in space. This is of particular importance if it is obtained on a real-time basis and is supplemented by the measurement of the rate at which breath gas is flowing. The flow rate instrumentation is beyond the scope of this endeavor. From these combined data, the respiratory quotient is readily obtained.

The concept of sampling and analyzing the breath gas with a self-contained instrument which is small enough to fit essentially under the chin of the astronaut, within the helmet, is a most challenging one. The device to accomplish this task consists of a gas inlet system, a mass spectrometer, and a vacuum pump. Each of these "building blocks" of the system must be miniaturized without appreciable sacrifice in performance if the goals are to be achieved. Auxiliary electronic components are to be carried in a back-pack.

Even though the mass spectrometer must be miniaturized beyond current state-of-the-art, the problems associated with the introduction and removal of the breath sample with miniaturized "leak" and pump combination is most acute. Laboratory apparatus for this function is huge compared to the constraints of this endeavor.

WORK STATEMENT

Phase A1, Leak

- Item 1. Study means of devising miniature needle valve.
- Item 2. Make experimental model, with no regard for refinement of actuator mechanism.
- Item 3. Attach to ultra-high-vacuum system with slow pumping speed and make experiments on valve operation.
- Item 4. Optimize valve parameters (exclusive of motion producing devices).
- Item 5. Design, devise and fabricate miniaturized valve actuator.
- Item 6. Attach miniaturized valve and actuator system to vacuum apparatus and observe its performance.
- Item 7. Deliver five leak valves with actuators for performance tests.

Phase A2, Ion Pump

- Item 1. Investigate the utility of various permanent magnetic materials for producing a magnetic field of 0.1 to 0.2 teslas in a small volume suitable for a Penning Discharge.
- Item 2. Design a self-contained magnetic circuit for producing the appropriate magnetic field for the Penning Discharge.
- Item 3. Design and fabricate small Penning Discharge structures and experimentally determine their pumping capabilities as functions of pressure, magnetic field strength, cell geometry and applied potential.
- Item 4. Choose most promising combination of geometry, magnetic field, and potential and build model.
- Item 5. Select titanium sublimator most suitable for use with pump model built in Item 4.

Item 6. Optimize parameters of power applied to sublimator to achieve maximum deposition of titanium with minimum (average) power.

Item 7. Add titanium sublimator to pump model.

Item 8. Perform experiments to determine operating characteristics of pump model described in Item 7.

Item 9. Make two ion pump assemblies and integrate with two of the leak valves to form self-contained system. These will be delivered to MSC for evaluation.

Phase B, Ion Source

Item 1. Study operating characteristics of filaments of various configurations and materials in oxygen-rich environments.

Item 2. Based on theoretical studies made previously, design miniature ion source which incorporates:

- a. Multiple filaments
- b. Differential pumping
- c. High production efficiency for well collimated ion beam
- d. Low production rate of photons which can reach detector.

Item 3. Fabricate and assemble ion source of Item 2.

Item 4. Attach ion source to system which includes small quadrupole, gas inlet, and pumping system similar to that proposed for use on astronaut.

Item 5. Perform experiments on assembly (Item 4) to demonstrate degree to which goals have been achieved.

Item 6. Optimize parameters.

Item 7. Prepare refined ion source for attachment to miniaturized quadrupole mass spectrometer of Phase C.

Phase C

Item 1. Design, fabricate and assemble a miniature quadrupole mass analyzer to match the ion source developed in Phase B.

Item 2. Integrate ion source, quadrupole, and a secondary emission multiplier into an assembly of minimum volume.

Item 3. Energize assembly with laboratory electronics (supplied by Earth Sciences) and make experiments to explore the relationship between performance capabilities and the power level of the excitation of the rod system.

Item 4. Review findings with Manned Spacecraft Center and mutually decide the preferred combination among the variables of Item 3.

Item 5. Design, fabricate and test flight-compatible electronics, guided by choices made in Item 4.

Item 6. Attach system comprising the ion source, quadrupole, and the detector to a laboratory vacuum apparatus (supplied by Earth Sciences). Using laboratory type gas inlet system, observe the performance of the mass spectrometer system to breath gas.

Item 7. Replace the laboratory vacuum and inlet systems with those developed in Phases A and B and repeat the experiment described in Item 6, using electronics of Item 5.

Item 8. Deliver to Manned Spacecraft Center the operating instrument used in Item 7.

SYSTEM GOALS AND CONSTRAINTS

The most obvious constraint is that of size, because of the severe limitation of space under the chin (and ears) of a man for an apparatus of this complexity. The space factor is further complicated by the voltage limitation placed on the wires which interconnect the back-pack with the other parts, since this necessitates the use of both dc and ac potential converters adjacent to the mass spectrometer assembly.

The system response is to be the same for all four of the gases - water vapor, nitrogen, oxygen, and carbon monoxide. This is most difficult to obtain, because the polar water vapor molecule has such a strong affinity for surfaces. Unless very special precautions are taken, an undesired time lag exists between the change in the water vapor concentration in the atmosphere and the arrival of the signal at the detector.

It is desired that the response of the entire system to a step change in the composition of the atmospheric gas be nearly completed in 100 milliseconds from the time of the onset of the change. This fast response is difficult to obtain with laboratory apparatus where pumps of large capacity are available. The very limited speed of the tiny pump is a very severe handicap in this instance. To display this change in concentration for all four gases, the height of each of four mass peaks must be observed many times during the 100 milliseconds of the change in output signal. It is anticipated that the outputs corresponding to each gas composition will be observed every 5 milliseconds.

TEST APPARATUS

A flexible vacuum system was set up for measuring the flow of gases through the experimental leak valve assemblies and for observing the speed of the ion pump models. This system is shown in Figure 1.

The system is evacuated from atmospheric pressure by a chilled zeolite sorption pump. Continued pumping to low pressures is provided by the 30 liter/second DriVac ion pump which can be valved off following the baking of the system. At no time is the system exposed to vapors from oil pumps (roughing or fine pumping).

Gas flow rates are monitored by observing the pressure differences across an orifice of known conductance. Quadrupole residual gas analyzers are used to measure the pressure differences. The quadrupoles are calibrated by admitting a fixed quantity of argon to the system and observing the resultant change in the current from the Bayard-Alpert ionization gauge in the absence of any ion pump operation. The ion gauge and the RGA's are operated at very low electron currents to minimize their pumping action. Further, the significance of their pumping action is demonstrated by observing the readings of the others when they are turned off in sequence.

The variable magnetic field is provided by a movable electromagnet. Following the system bake, the magnet is rolled in place, surrounding the test Penning cell. The field produced by this magnet is much more uniform than that provided by the self-contained magnetic structure described later.

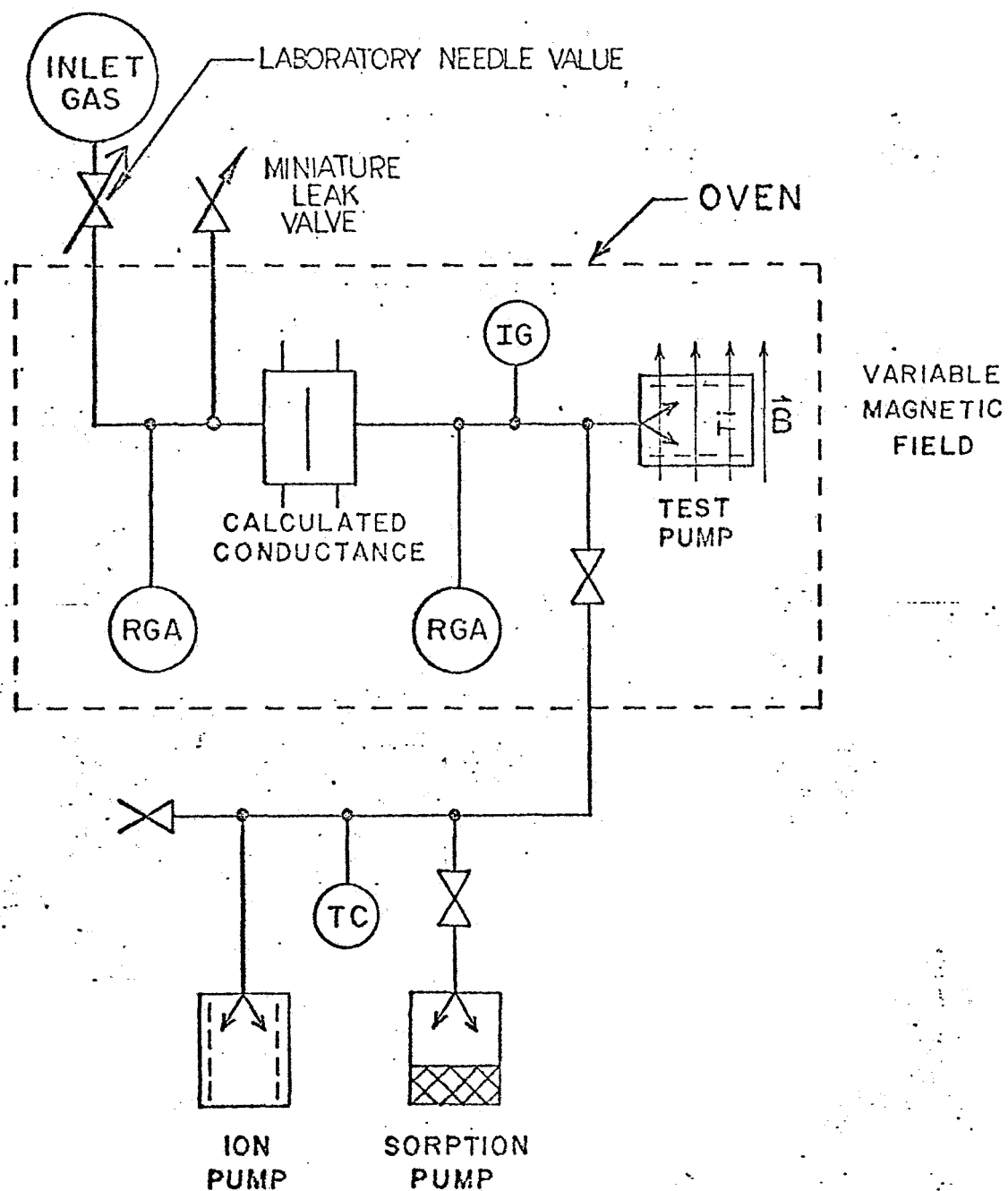


FIGURE 1
PUMP TEST SYSTEM

THE LEAK

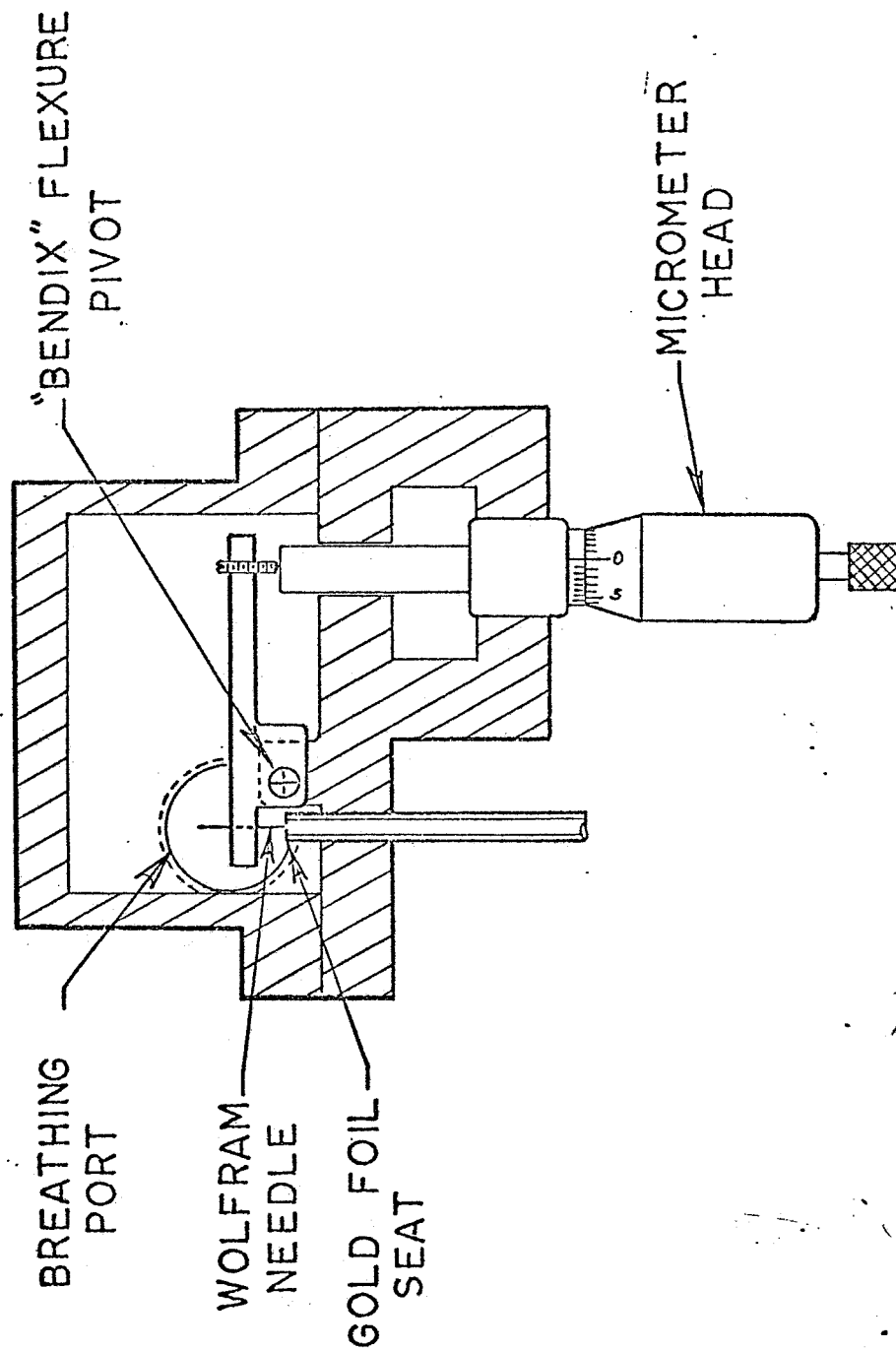
DISCUSSION

At the onset of this program, the leak was considered to be the most difficult part of the whole task. Because of the very limited capacity of the ion pump, the flow rate through the leak is extremely small. The goal that this flow be of molecular nature, and not viscous, places further constraints on the design. A capillary tube, for instance, will not meet this requirement for molecular flow.

Conductances on the order of 10^{-10} liters/second are required. When sampling from atmospheric pressure, this is given by an aperture area of 10^{-11} cm². If the conductance is a round tube of diameter D and length L, the area of the cross-section varies as L/D for constant conductance. Thus, it is evident that the conditions of molecular flow limit the ratio of L/D to such values that the cross-sectional area remains minute. The diameter becomes comparable to a large virus molecule and consequently is vulnerable to plugging.

TEST MODEL

Of the various devices which might be used to form a miniature gas leak, we chose to try first the microneedle and seat arrangement. This follows a suggestion made by MSC. The needle is an electro-polished tungsten wire; the seat is 1-mil thick gold foil. In the laboratory model, emphasis was placed on the needle and seat combination and no attempt was made to miniaturize the actuator. The arrangement used is shown in Figure 2. The pivot for the needle is placed in the plane of the foil to minimize the sidewise motion of the needle in the seat. Further, the pivot is a "flexure" with no sliding parts. Relative motion between the needle and the seat is provided by the micrometer screw through the linkage with a mechanical advantage of about six. The adjustment



LABORATORY LEAK

FIGURE 2

of this unit is particularly sensitive to extremely small motions of the screw. Consequently, an unillustrated refinement was added. An arm about 1.8 inches long was attached to the micrometer screw. This arm was spring loaded against a 6-32 screw. In pushing against this arm, the screw opens the leak. The spring closes it, but only to an adjustable stop. This arrangement prevents the inadvertent insertion of the needle too deeply into the foil.

The initial operation of the needle to create the leak is very critical. With the aid of a microscope to locate the needle, it is clamped in the arm with its axis perpendicular to the plane of the foil and its tip just touching the gold seat. The tube from the leak is attached to a helium mass spectrometer leak detector. With the needle and seat immersed in an atmosphere of helium, the tedious job of forming the leak is begun. The procedure consists of alternately advancing the needle a very small distance, then withdrawing it. When the needle ruptures the diaphragm, a leak of helium into the leak detector is noted on the withdrawal. The first piercing of the foil provides a leak of more than ample conductance. Reinsertion of the needle into the foil closes the leak effectively.

PERFORMANCE

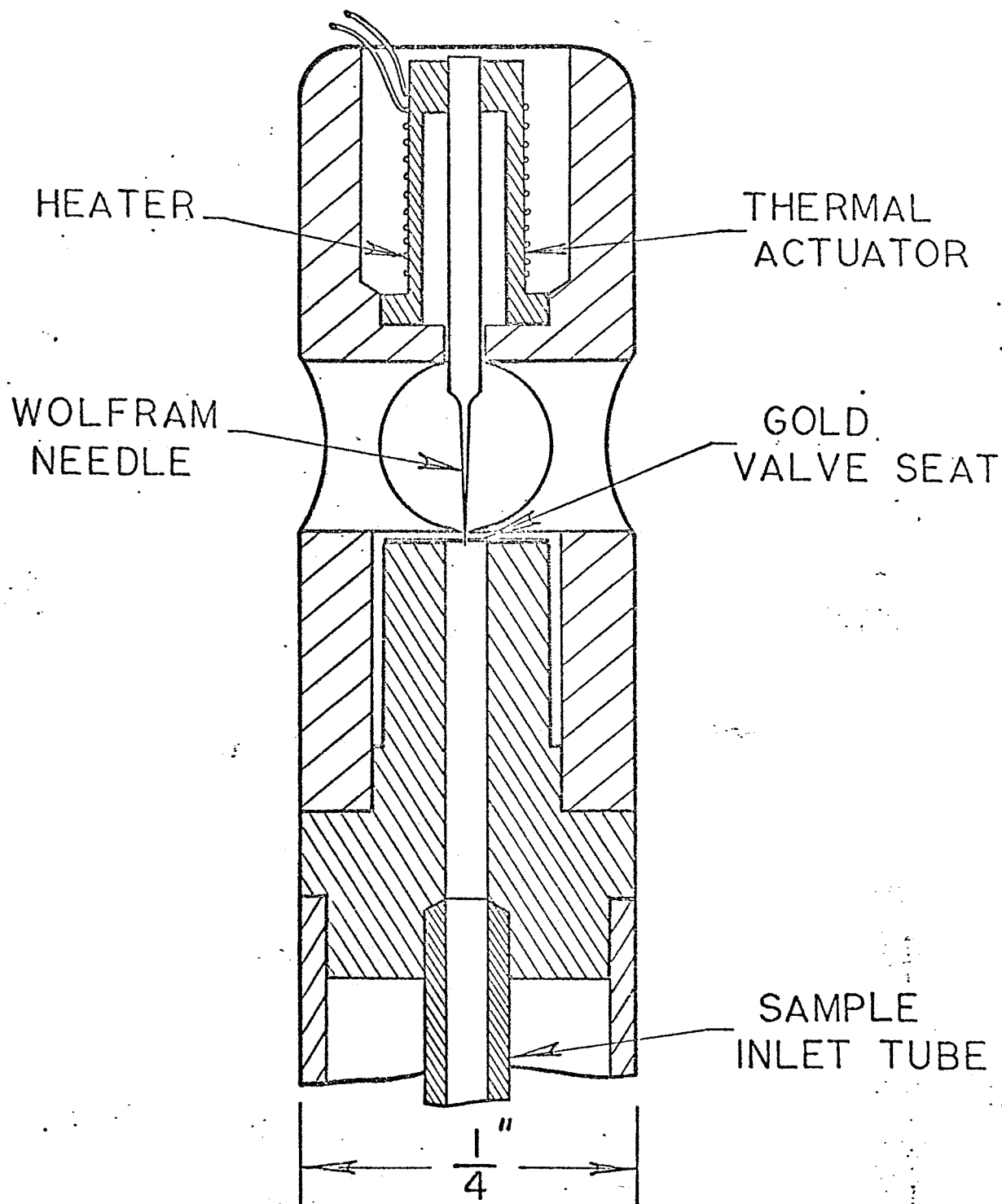
Leak assemblies were tested by using the helium leak detector and many were capable of adjustment in the range of 1 to 2×10^{-7} torr-liters/second. One assembly was successfully operated at a temperature of 160°F for 18 hours and gave a stable flow rate of 2×10^{-7} torr-liters/second, nitrogen equivalent.

A single leak assembly was attached to the test system and performed continuously for 11 weeks. During this period, it was easily adjusted to obtain flow rates from 1×10^{-7} to 9×10^{-7} torr-liters/second. Occasionally, the leak closed of its own accord, but it never opened spontaneously. A simple manual adjustment restored the leak to its normal operation.

An experimental leak assembly was hand carried to Manned Spacecraft Center in November 1968. There its performance was observed, using a magnetic mass spectrometer to observe the respiratory components of interest. The leak demonstrated its molecular flow capability through the appearance of the water vapor peak before the CO_2 peak during the exhale portion of the respiratory cycle.

FLIGHT MODEL

A flight model of the molecular leak and actuator was designed and fabricated. It is to be operated by differential expansion of the needle support structure by the application of electrical power. A schematic of this leak is shown in Figure 3. The needle and seat are identical to those of the laboratory model.



MINIATURE LEAK
FIGURE 3

THE PUMP

DISCUSSION

In order to fit within the available space, the ion pump used with the breath analyzer must have dimensions of "about an inch." This volume must contain the magnetic field-producing structure, including any magnetic shielding which may be required to contain the field.

The pumping speed one might anticipate from a pump of this size can be estimated from the speeds of commercial ion pump designs. A typical 30 liter per second pump uses 36 cells in a field of about 0.2 tesla (2 kilogauss). The excitation potential is usually 8 kilovolts. The small structure can support a field of only about 0.1 tesla, which works at a correspondingly lower potential. The lowered magnetic field and voltage result in a speed of about 8 liters per second for the 36 cells, as evidenced by the very early diode pumps (VacIon). This is 0.22 liter per second per cell. These cells are 0.5 inch in diameter, 0.75 inch long. The pump under consideration is about the same diameter, but half the length. A consideration of the potential distribution in this cell under the influence of high electron space charge leads to the conclusion that the ends of the cells do not contribute as heavily to the pumping process as do the center portions. This accounts for the difficulty people have had in scaling down the geometry of the commercial ion pumps. The maximum speed expected from the cell of the miniature ion pump thus becomes less than 0.1 liter per second.

During a study of the characteristics of the Penning Discharge, the potential on the axis of the device was found

to be essentially that of the cathode⁽¹⁾. Further, the volt-ampere relationship of the discharge proved to be nearly independent of the presence or absence of a post on the axis of the device between the two cathodes.

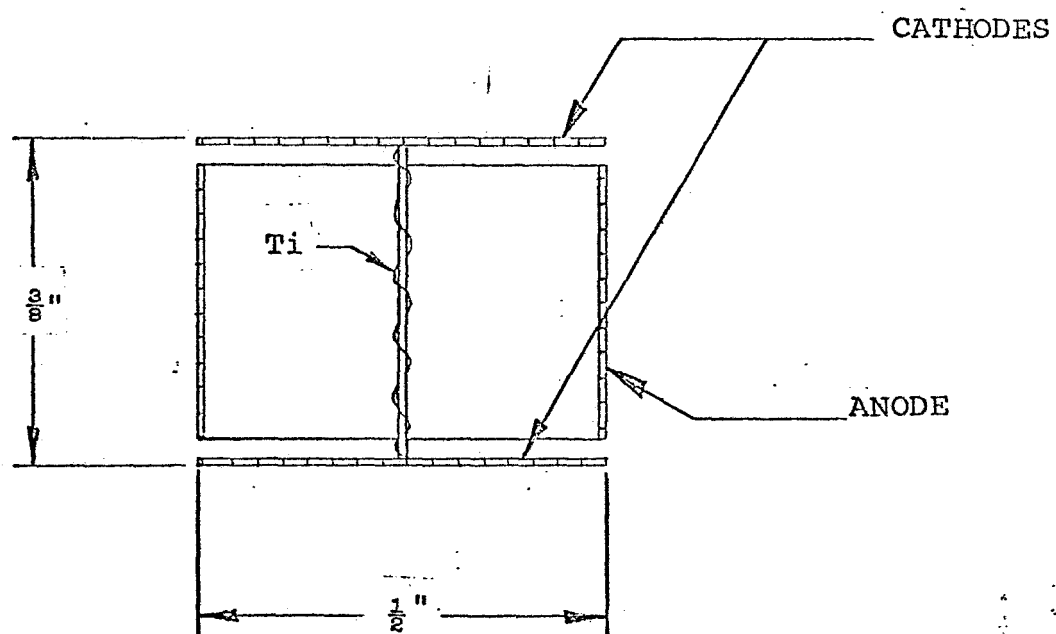
During these early experiments, the ionic pumping of noble gases (argon) by the cell was shown to be very greatly enhanced by the evaporation/sublimation of titanium onto the active cathode surfaces during the running of the discharge⁽²⁾. That the gettering action of the freshly deposited titanium surfaces effectively pumps the chemically active gases has been known for many years. Hence the fact that the deposition of titanium on the inner surfaces of a Penning Discharge cell greatly enhances its pumping capabilities for all gases is well established.

DESIGN OF CELL

The problems associated with the development of a miniature getter/ion pump consist primarily of finding the best parameters for titanium deposition and determining the most appropriate combination of magnetic field and discharge potentials for the Penning Discharge for this very unique application.

The design of the miniature pump centers around the small Penning cell shown in cross-section in Figure 4. Its working volume is a right cylinder 1/2 inch in diameter and 3/8 inch long. The center post contains a titanium wire for sublimation wrapped around a supporting wire of tantalum.

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- (1) W. M. Brubaker, Space Charge and Potential Distributions in the Penning Discharge, Proceedings 20th Annual Conference Physical Electronics, MIT, 1960
 - (2) W. M. Brubaker, A Method for Greatly Enhancing the Pumping Action of a Penning Discharge, Sixth National Symposium on Vacuum Technology, 1959.



ACTUAL SIZE

Figure 4. Miniature Ion Pump

TEST RESULTS - MINIATURE CELL PERFORMANCE

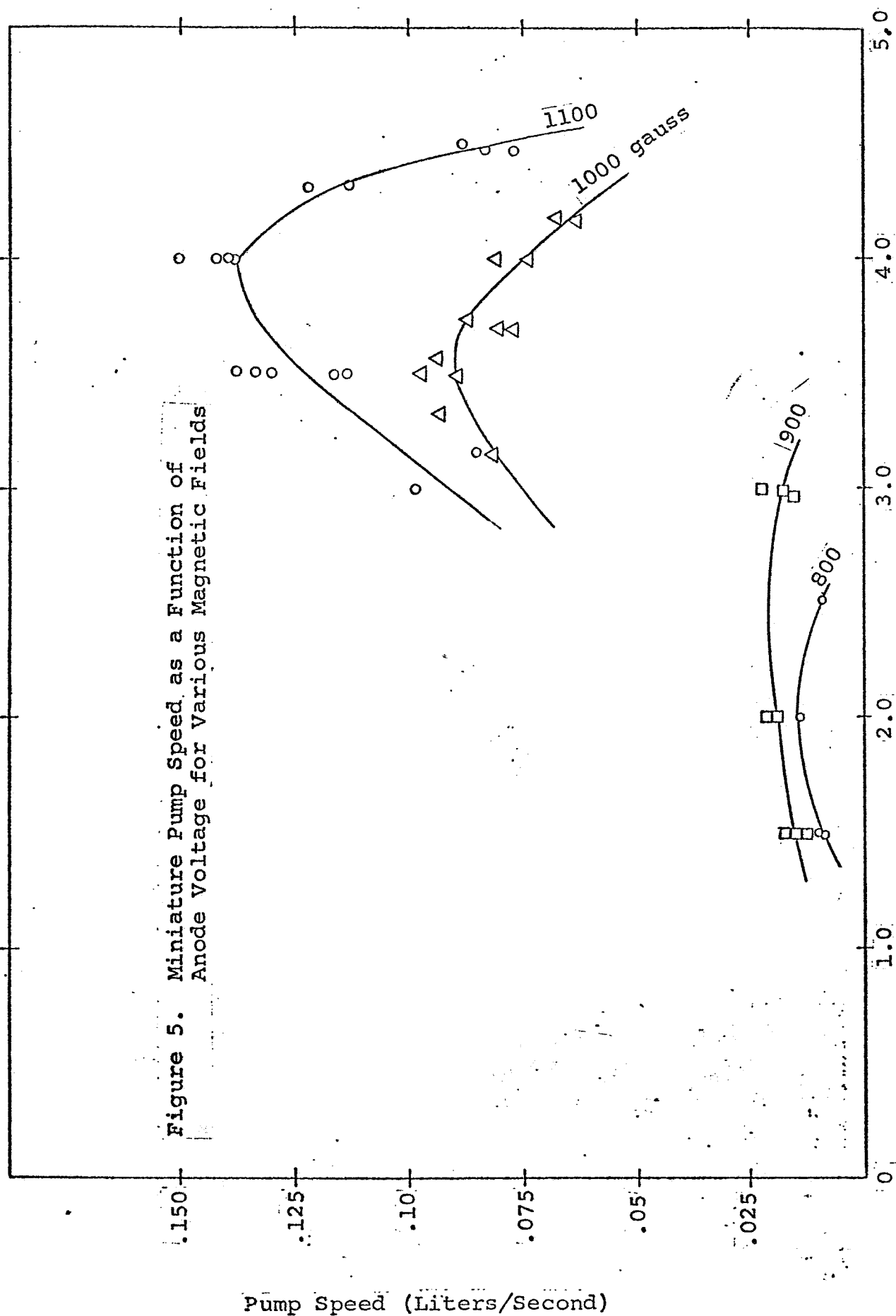
Tests of the miniature cell in the electro-magnet were performed over a period of 20 weeks. During this time, the cell was continuously under a load of nitrogen gas ranging from 1×10^{-7} to 5×10^{-6} torr-liters/second. A miniature leak assembly was used for 11 weeks of this period. It successfully provided flow rates in the 10^{-7} range. Pumping speeds (without sublimation) as a function of anode voltage, for various magnetic field strengths, were determined. These data are summarized in Figure 5. Note that the potential which produces optimum pumping speed varies approximately as the square of the magnetic field strength. Also, the speed of the pump is seen to fall appreciably for fields of less than 1,000 gauss.

SELF-CONTAINED MAGNETIC FIELD - DESIGN AND TEST RESULTS

A magnetic field-forming structure which fulfills the necessary field strength and size requirements is shown in Figure 6. It utilizes two cylindrical Indox V ceramic magnets 1/2 inch in diameter and 3/16 inch long to produce a field of approximately 1,000 gauss in a 0.4 inch gap. The magnets and pump electrodes are held in place by a cylindrical "pill-box" which completes the magnetic circuit between the two magnets and also serves to shield the rest of the system from the magnetic field. The outer dimensions of the "pill-box" are 0.925 inch in diameter by 1 inch long. Holes and slots in the side of the "pillbox" provide for electrode leads and pumping.

The complete pump, with the Penning cell inside the magnetic structure, is shown in Figure 7. Tests of the pump were conducted over a 2-week period and the results are shown in

Figure 5. Miniature Pump Speed as a Function of Anode Voltage for Various Magnetic Fields



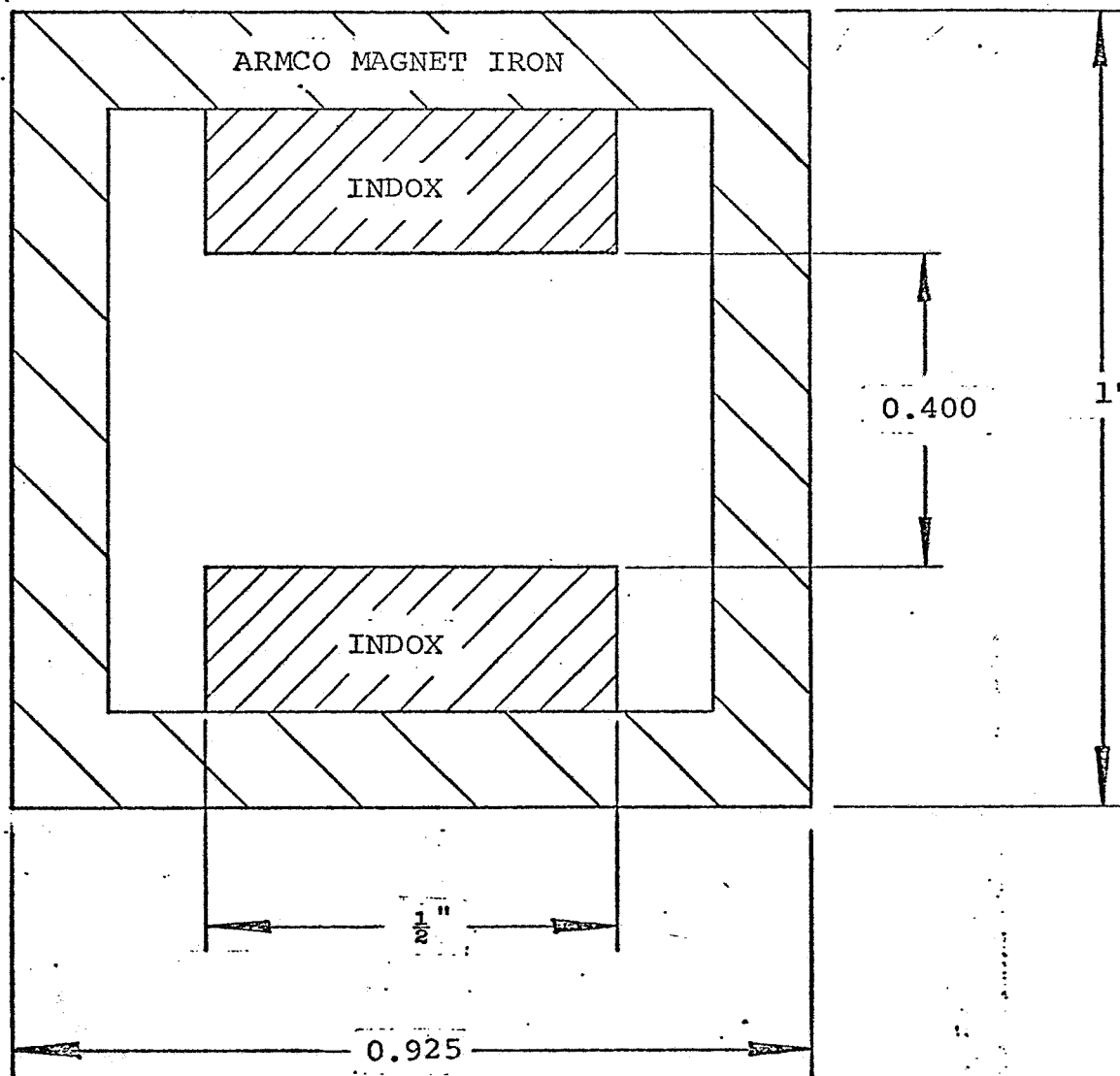
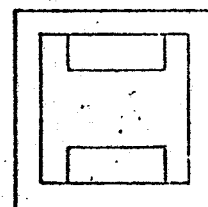
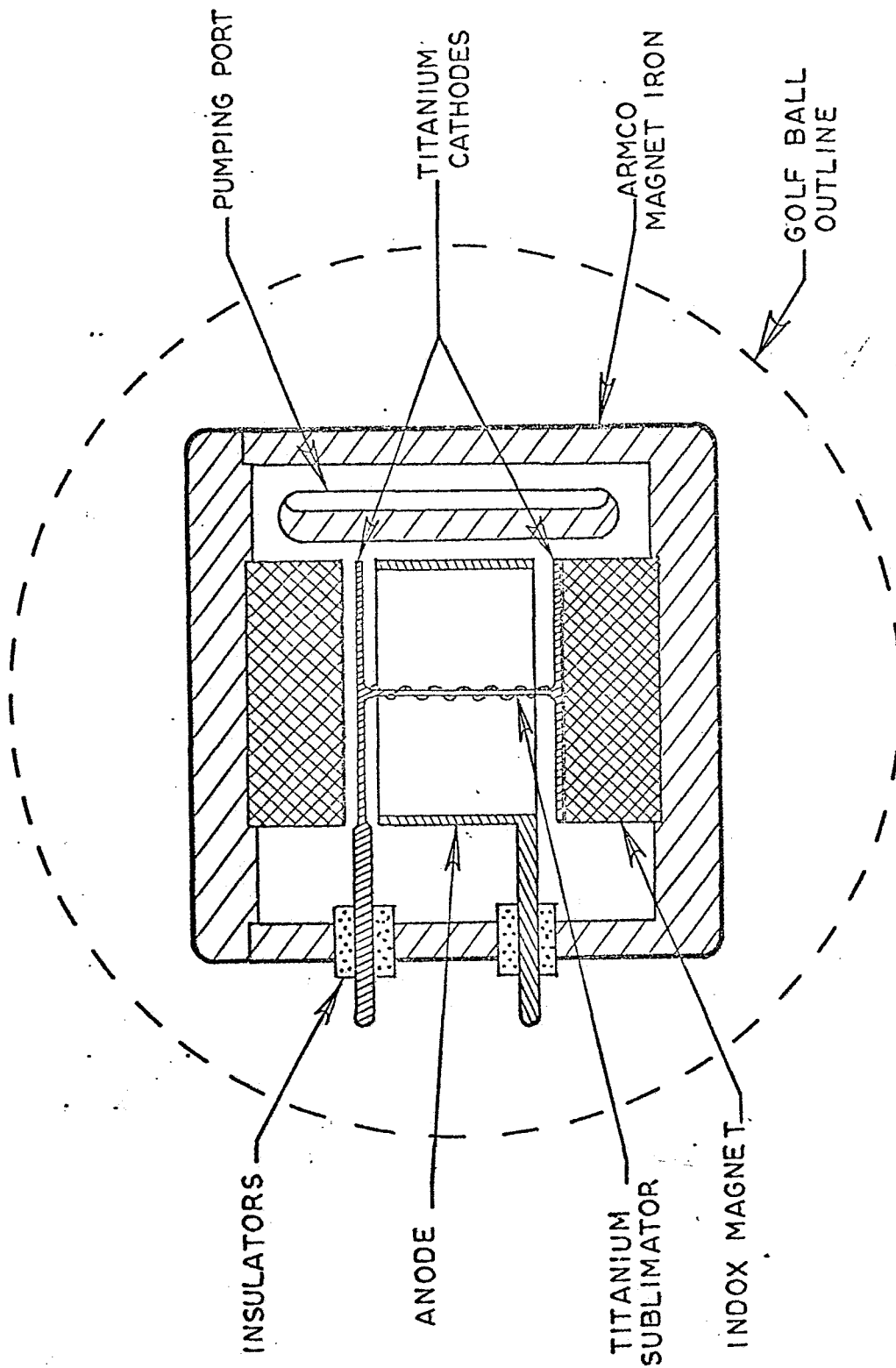


Figure 6. Ion Pump Magnet





MINIATURE ION PUMP AND SUBLIMATOR
FOR ASTRONAUT BREATH ANALYZER

FIGURE 7

Figure 8 along with the data obtained from the tests of the Penning cell in the uniform field of the electromagnet. A maximum speed of 0.14 liters per second occurs at an anode voltage of 2.5 kilovolts. This equals the best speed attained by the prototype pump when operated in a uniform field of 1,100 gauss. The pump current at 3×10^{-6} torr is approximately 2 microamps, giving a total power dissipated by the pump of less than 6 milliwatts.

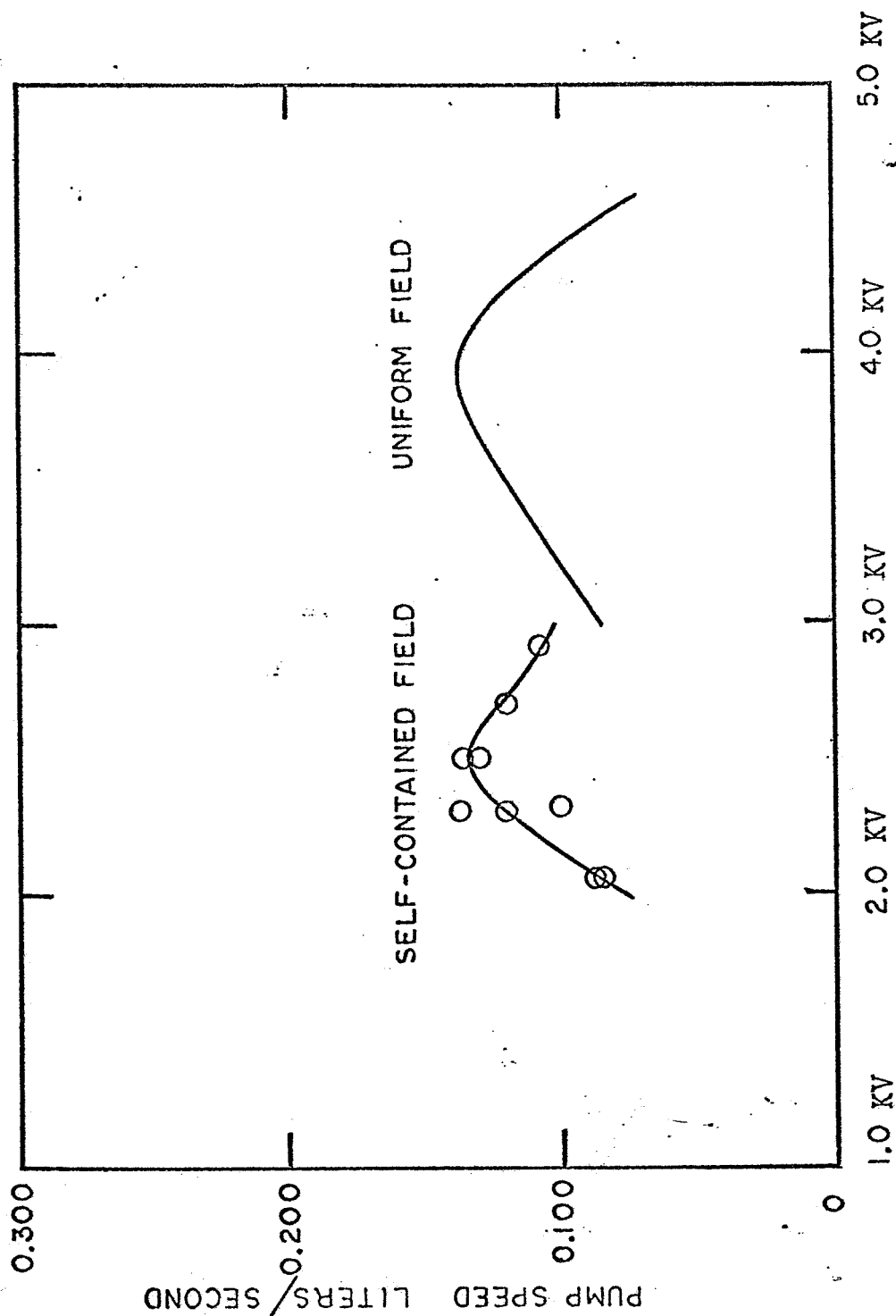
SUBLIMATOR

The sublimator of the miniature pump is located on the center post between the two cathodes of the Penning cell. It consists of one or two fine-diameter titanium wires wrapped around a supporting wire of tantalum.

The life of this filament is proportional to the mass of the titanium available for pumping and inversely proportional to the amount of gas that is being pumped. A theoretical calculation of the filament life is given in Appendix I. For a titanium mass of 1 milligram and a leak rate of 10^{-7} torr-liters per second, the maximum life of the filament is about 45 days. This calculation is based on complete utilization of the deposited titanium. In practice, the titanium is used at a much lower efficiency.

Power is supplied to the sublimator filament in current pulses of 3 to 6 seconds duration with a duty cycle of 5 to 10 percent. Current amplitudes range from 1.6 to 2.3 amps, depending on the size and age of the filament. The average power dissipated in the sublimator varies from 0.2 to 0.5 watt.

Tests of the sublimation filament show that it increases the nitrogen pumping system speed by a factor of ten or more relative to that of the Penning discharge alone. System



NITROGEN PUMP SPEED AS FUNCTION OF
ANODE POTENTIAL ($B = 0.11$ TESLA)

FIGURE 8

speeds greater than 1 liter per second have been measured. The titanium is used at lower efficiency at these higher pumping speeds. When use of the sublimator is terminated, the pumping speed drops to that of the cell alone in a manner which is roughly described by a time constant of 7 minutes.

DISCUSSION

Very significant improvements have been made recently in the design of the quadrupole mass filter. These include the use of a delayed dc ramp at the entrance to the filter,⁽³⁾ and the use of hyperbolic rod contours.⁽⁴⁾ The performance of a small quadrupole with round rods 0.150 inches in diameter, 3 inches long, is well established as these instruments have been used extensively in rocket probes of the earth's atmosphere.⁽⁵⁾ Papers describing these instruments are included in the appendix.

The requirements of the mass spectrometer for the breath analyzer usage are more demanding than those of a rocket probe. In order to obtain analytical accuracy, flat-topped peaks are required, indicating 100 percent transmission of the ions. The use of the delayed dc ramp⁽³⁾ greatly facilitates the achievement of this goal. Further, the use of rods with hyperbolic field-forming surfaces permits high performance in an instrument of small size.⁽⁴⁾ These investigations have demonstrated that a high performance quadrupole mass analyzer can be built in small size, suitable for mounting under an astronaut's chin.

Theory predicts that the performance of quadrupole mass filter is dependent upon the voltage applied to the rods, and is entirely independent of the physical size of the instrument. This conclusion assumes that the imperfections in the field-forming surfaces scale with the rod dimensions. The higher resolving power attained with mass filters of larger dimensions is attributed to the non-scaling characteristics of the imperfections in the contours of the field forming surfaces.

(3), (4), (5) See page 30.

ION SOURCE

The design of the ion source for this application is complicated by several unusual constraints, some of which tend to be mutually exclusive. First, a high sensitivity is desired to facilitate the handling of the data and to provide high response speed in the output network. Second, the composition of the gas in the ionizing region must adjust promptly to any change of the composition of the breath gas. Third, the filament must use low power and have a long life expectancy in the presence of oxygen. Further, it must have a negligible effect on the composition of the ambient gases.

High sensitivity is obtained through the use of differential pumping between the ionizing volume and the rest of the vacuum system. To provide a pressure drop of 10^{-5} torr through the apertures joining the ionizing region and the rest of the vacuum system at a gas flow rate of 10^{-7} torr-liters/second requires a conductance of 10^{-2} liters/second. Such a conductance is provided by an aperture of 0.015 inch in diameter, which is scarcely large enough to allow the ions to get out of the source. This conductance limitation certainly does not permit the use of additional apertures (or tunnels) for the admission of an ionizing electron beam from either of two filaments (one is a spare). Conversely, the use of such a small conductance from the ion source makes the time constant of the gas dynamics of the ionizing region long ($\tau = V/S$ where V is the volume of the ionizing region and S is the conductance under discussion).

The above discussion of source sensitivity and the gas dynamics indicates the great desirability of placing the filament(s) within the vacuum enclosure of the ion source. Otherwise, effective differential pumping is almost impossible to

achieve. If the filament is placed within the vacuum enclosure, it must be constructed of material that is essentially inert to oxygen when it is at electron-emitting temperature. Fortunately, rhenium is such a material.

The very small ion source designed to meet these constraints is shown in Figure 9. Its volume is only 0.3 cc. When considering the gas dynamics of the system, the volume and the conductance of the inlet tube must be taken into account. This tube is 0.091 inch in diameter and 5 inches long. Its volume is about 0.5 cc and its conductance for air at room temperature is 1.1×10^{-2} liters/second.

Ions are formed in the small ion cage, at a potential closely related to that of the cage. The enclosing opaque cylinder forms an electrical shield for the filament(s). Electrons from the filament are accelerated toward the ion cage. Some of the electrons pass completely through the cage and are reflected back to it by the opposing field in the space between the ion cage and the shield. Eventually, all of the electrons are captured by the wires of the ion cage.

Ions are urged toward the ion exit aperture by the field which results from the fact that the potential of the ion exit aperture is more negative than that of the ion cage. Sensitivities for resolved ion peaks are in excess of 10^{-6} amperes/torr.

ION SOURCE FILAMENTS

The requirements that the filament operate at low power and have a long life tend to be mutually exclusive. Therefore, obtaining data on the life expectancy of filaments of various materials and sizes in an oxygen-rich atmosphere is important. To this end, an experiment was made to observe the aging of filaments operating with electron

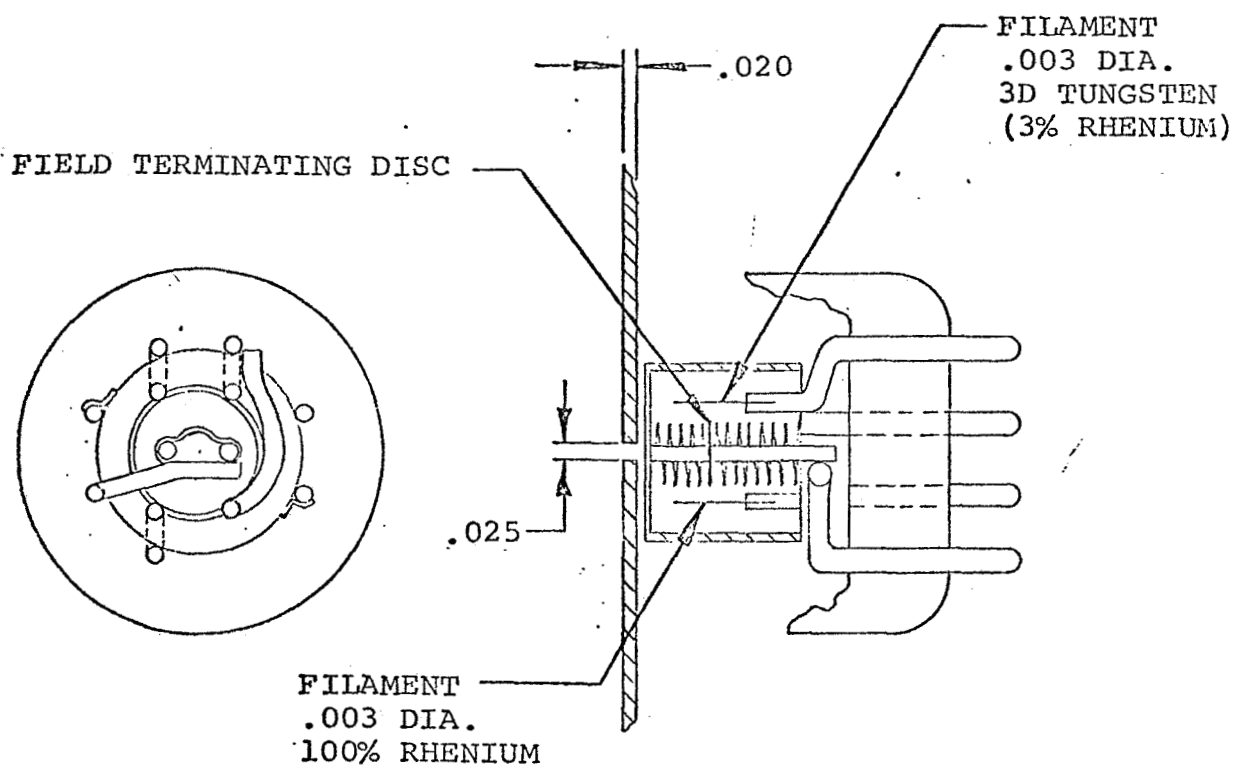


Figure 9. Miniature Ion Source

emission in an oxygen-rich atmosphere at a pressure of about 10^{-4} torr. This gives an accelerated test, so far as the oxygen interaction is concerned.

A diagram of the filament life test apparatus is shown in Figure 10. A fixture for holding seven test filaments has been provided. Each filament is surrounded on three sides by an anode structure. Stabilized power supplies provide regulated, adjustable potentials for each filament. Meters monitor the heating current and voltage and the emission (anode) current for each filament.

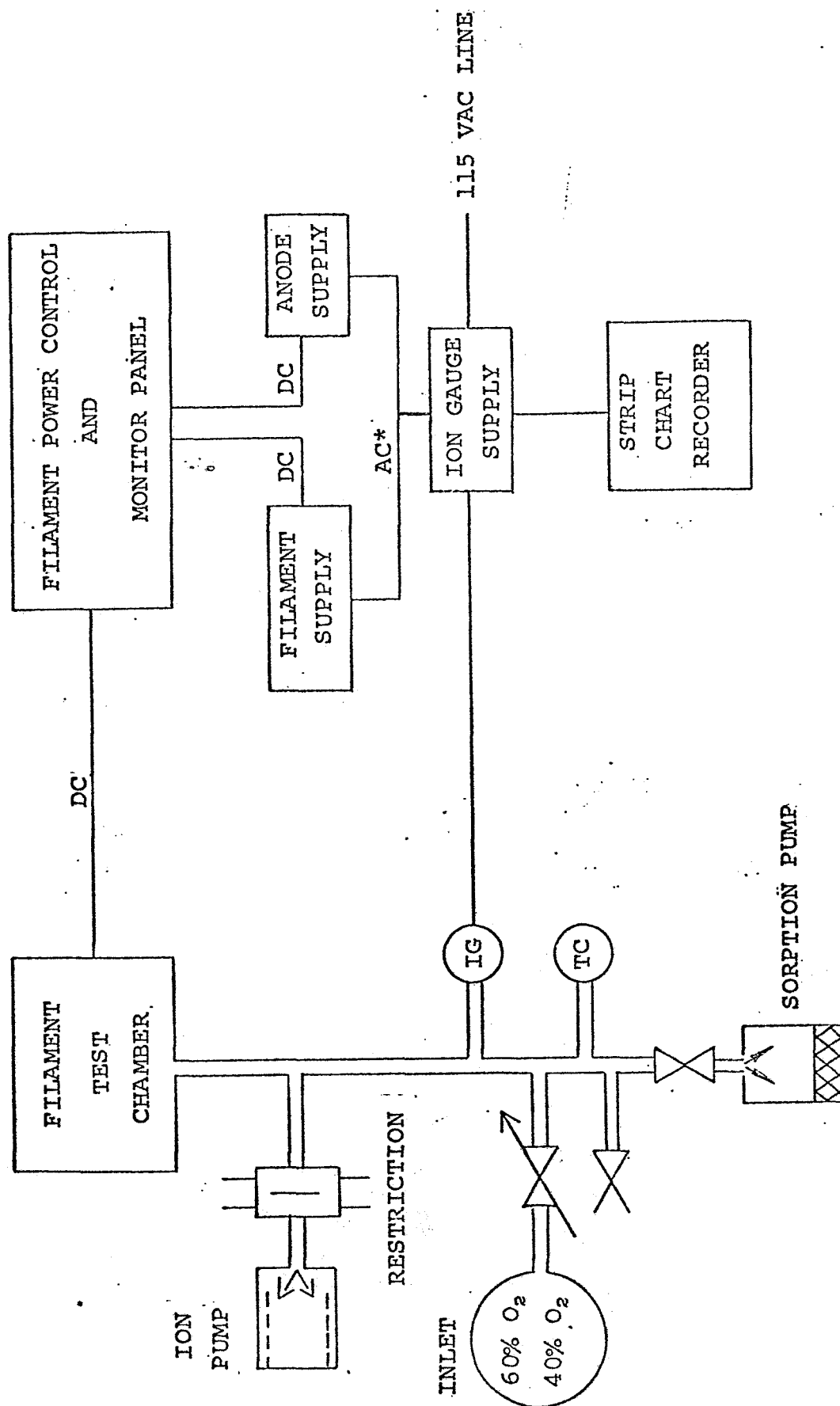
The vacuum system is pumped by a 30 liter per second DriVac ion pump. The pumping speed is conductance limited by an aperture placed in the pump line. This permits the pump to operate at low pressure while the filaments are immersed in oxygen at a much higher pressure. Precautions were taken to assure that the filaments would not fail prematurely through inadvertent operation at higher pressure. These include an overpressure shutoff by the ion gauge, as well as a shutoff in case of ion gauge filament burn-out. Additionally, a strip chart recorder, running at slow speed, gives a continuous record of the pressure during unattended periods (weekends).

FILAMENT LIFE TEST DATA

Time-to-failure data for filaments of various materials and sizes are summarized in Table 1.

QUADRUPOLE

The requirements of the mass analyzer are rather modest in terms of the capability of modern quadrupole analyzers.



*AC interrupted by ion gauge at high pressure threshold
or loss of ion gauge filament.

Figure 10. Functional Diagram Filament Life Test Apparatus

Table 1

Filament Test Data

<u>Qty</u>	<u>Filament Wire Type</u>	<u>Size (Dia)</u>	<u>Life (Hrs)</u>
2	3D Tungsten (3% Rhenium)	.001	235 avg
1	3D Tungsten (3% Rhenium)	.003	245
1	3D Tungsten (3% Rhenium)	.005	270
1	218 Tungsten (100%)	.001	235
1	218 Tungsten (100%)	.003	310
1	218 Tungsten (100%)	.005	315
3	5% Rhenium, 95% Tungsten	.003	312 avg.
3	26% Rhenium	.003	200 avg.
1	3D	.003	648
2	100% Rhenium	.003	500 avg.
1	100% Rhenium	.003	790
1	100% Rhenium	.003	>1800

However, the severe restrictions on the dimensions of the unit and the limitations on excitation power make the task more difficult.

Extrapolating from the performance of a quadrupole with rods 0.150 inch in diameter and 3 inches long⁽⁵⁾, it was concluded that a quadrupole of similar cross-sectional dimensions 2 inches long, will provide adequate performance. The smaller quadrupole would have hyperbolic rod surfaces⁽⁴⁾ and would have a set of auxiliary electrodes at the entrance to provide a delayed dc ramp mode of operation⁽³⁾. With these added improvements, it is expected that the smaller quadrupole would out-perform the larger (3 inch) one.

Although the resolving power requirements for the breath analyzer are less stringent than those of the 3-inch instrument, the demand for analytical accuracy (flat-topped peaks) requires the utmost performance of the mass analyzer. The dimensions chosen for this instrument are: $r_0 = 0.075$ inch, $L = 2$ inches.

The molybdenum rods were contoured to a hyperbolic shape by grinding with a narrow diamond wheel. The grinder was operated automatically by tape. Cast replicas of the contours were observed in a 50-power comparator. In general, the departures of the surfaces from the hyperbolic contours were found to be less than 5×10^{-4} inches. This uncertainty is larger than was desired and anticipated.

(3) W. M. Brubaker, Improved Quadrupole, Advances in Mass Spectrometry, Vol. 4, page 293.

(4) W. M. Brubaker, Comparison of Quadrupole Mass Spectrometers with Round and Hyperbolic Rods, Sixteenth Annual Conference on Mass Spectrometry and Allied Topics, 1968

(5) W. M. Brubaker, N. W. Bell, and F. B. Wiens, Quadrupole Mass Spectrometer for Atmospheric Studies in the 50- to 90-Kilometer Range, Twelfth Annual Conference on Mass Spectrometry and Allied Topics, 1964.

A novel method was used to hold the rod assembly together. The rods were cemented with sauerisen cement to a high alumina ring while they were clamped in a jig. Figure 11 shows the assembly with the jig and Figure 12 shows the assembly after the jig was removed. Wire tabs cemented to the high alumina ring provide attachments for the ion source.

Sauerisen cement was also used to form the feed-through insulators for the ion source. Before this cement was used for this purpose its outgassing characteristics were observed. A small quantity of the cement was placed in the vacuum system used to test the ion pump. The appendage in which the cement was placed was heated to over 100°C. The initial outgassing presented a gas load too large for the small pump to handle alone. However, after a few hours at the high temperature, the outgassing rate decreased to a point where the small ion pump operated satisfactorily, carrying the entire load alone. Thus, the sauerisen appeared to be a satisfactory cement for holding the rod assembly in place, even when the apparatus is operated at elevated temperatures.

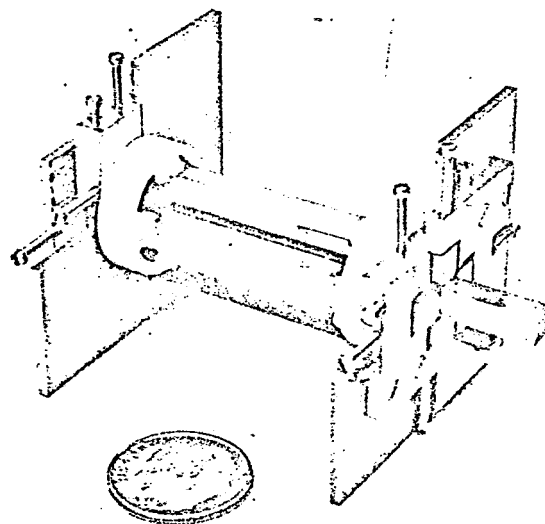


Figure 11. Miniature Quadrupole Assembly
in Alignment Jig

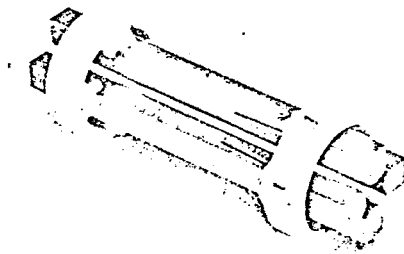


Figure 12. Miniature Quadrupole Assembly Removed
from Alignment Jig

SYSTEM PERFORMANCE

DESCRIPTION

The performance of the complete breath analysis system was effectively determined by joining all of the essential elements - pump, leak and quadrupole - into a small bench-size system. A system diagram showing the configuration of the essential parts and their associated electronics is given in Figure 13.

Although the assembled system is large relative to that of the breath analyzer, the essential features are duplicated. In particular, the gas inlet system is identical in its essentials. The gold foil-tungsten needle valve leak is connected to the ion source by a heated tube 5 inches long. The inlet to the leak is placed in a 3/4-inch metal tube about 3 inches from either end. The purpose of the tube is to conduct breath gas to the leak in a known manner so that the instrument response can be studied.

The small size inlet tube (0.091 inch diameter) terminates at the ion source. The only escape for the molecules from the source is through the ion exit aperture. The pumping speed (conductance) of this aperture was designed to be about 10 percent of the speed of the ion pump, or 1.5×10^{-2} liters/second. At a gas flow rate of 1.5×10^{-7} torr-liters/second, this gives a calculated ion source pressure of 10^{-5} torr and an analyzer-pump pressure of 10^{-6} torr.

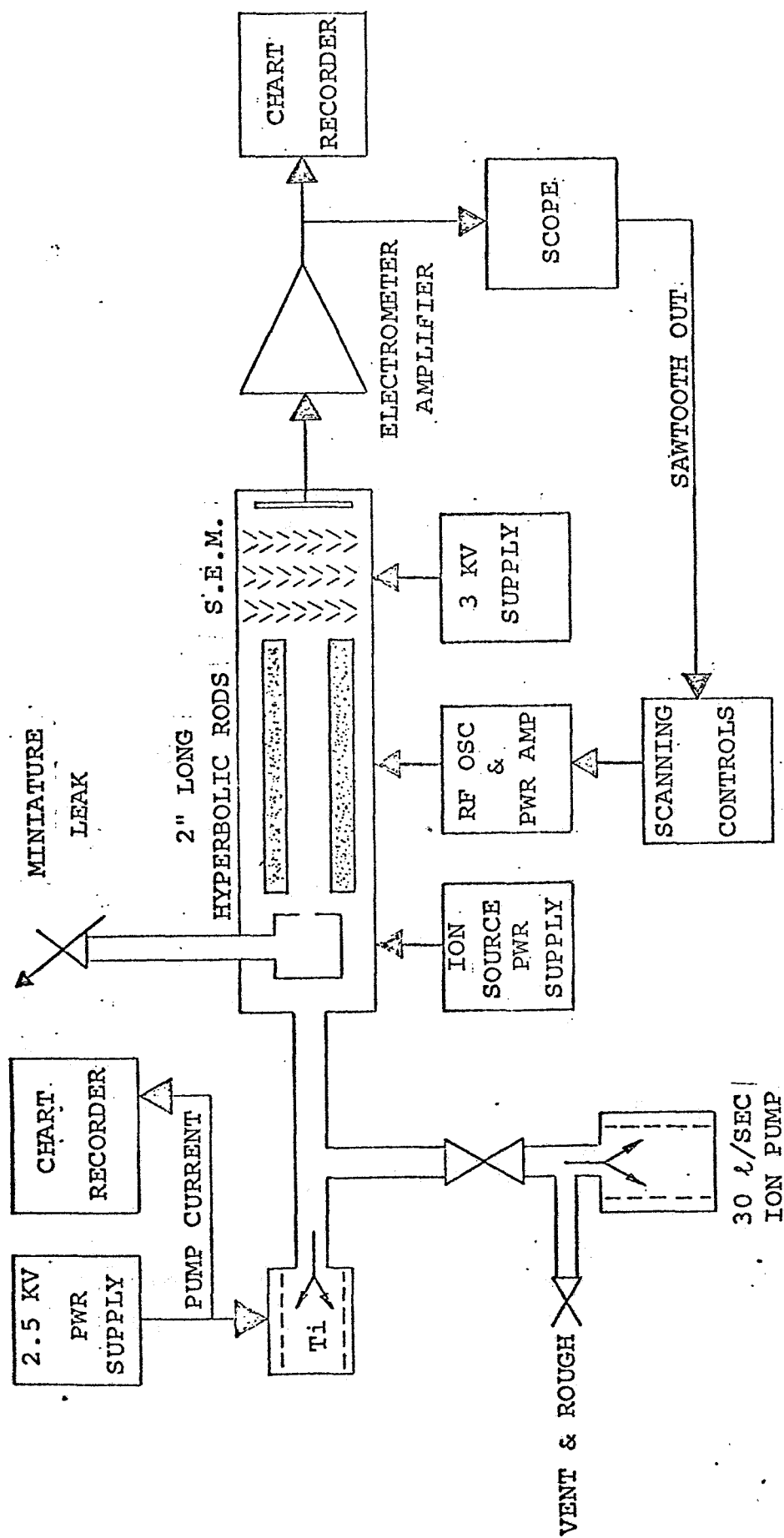


Figure 13. Breath Analyzer Test System

The totally enclosed ion source has the same geometry as that planned for the breath analyzer. Its internal volume is only 0.3 cc. The volume of the inlet tube is about 0.5 cc. The total volume of the inlet tube and the ion source is thus 0.8 cc, or 8×10^{-4} liters. If this volume were lumped into one unit and evacuated through a conductance of 1.5×10^{-2} liters/second, the time constant of the assembly would be

$$\tau = V/S = 8 \times 10^{-4} / 1.5 \times 10^{-2} = 53 \times 10^{-3} \text{ seconds.}$$

As is noted later, the observed time constant is 52 milliseconds.

The quadrupole used for these tests is identical to the one intended for the breath analyzer except that the auxiliary electrodes required to achieve a delayed dc ramp mode of operation were not in place.

The multiplier-detector is a venetian blind laboratory type. The signal from the multiplier is directed to a unity gain impedance reducer (cathode follower) and then to a high-speed, Sanborn multichannel recorder.

The large, unbaked system was pumped satisfactorily with the miniature ion pump while at room temperature. However, when the filament in the source was raised to electron emitting temperature, the outgassing of the sauerisen cement presented a gas load too large for the small pump. Additional pumping, which was conductance limited to about 2 liters/second, was provided by the DriVac ion pump. The response speed of the system is determined by the combined volume of the source and inlet tube and the conductance of the aperture from the source to the analyzer. The additional pumping, therefore, in no way alters the speed of response of the system.

EXPERIMENTS

The operation of the small quadrupole is illustrated by the background spectrum shown in Figure 14. Peaks from masses 14, 16, 17, 18, 28, 32, 40, and 44 are clearly visible. The modulations of the peak tops on masses 18 and 28 are attributed to imperfections of the contours of the field-forming rod surfaces. The spectrum clearly demonstrates the capability of the small quadrupole, with hyperbolic surfaces and without the aid of a set of auxiliary electrodes, to adequately provide the mass separation required for the breath analyzer application.

The response of the system to breath gas is shown in Figure 15, which is a recording of the CO₂ peak as the man breathed through the 3/4-inch tube mentioned above. The upper trace shows the CO₂ peaks at low amplitude and the middle trace shows them at higher amplifier gain. The bottom square trace is in response to the opening and closing of a hand switch which the subject operated upon reversing the direction of breathing.

Of prime importance is the rate of decay of the CO₂ signal on transition from exhale to inhale. The quick reversal of breathing promptly terminates the high CO₂ concentration as room air is drawn over the leak. As indicated on the chart, the decay to 10 percent of the signal occurs in about 120 milliseconds. On an exponential decay, this occurs in 2.3 time constants, which corresponds to a time constant of 52 milliseconds. This experimental value of the instrument response agrees surprisingly well with the response calculated when the volume of the inlet tube is lumped with that of the ion source. The assumptions made in the theoretical derivation (lumped volumes) would not seem to warrant such verification.

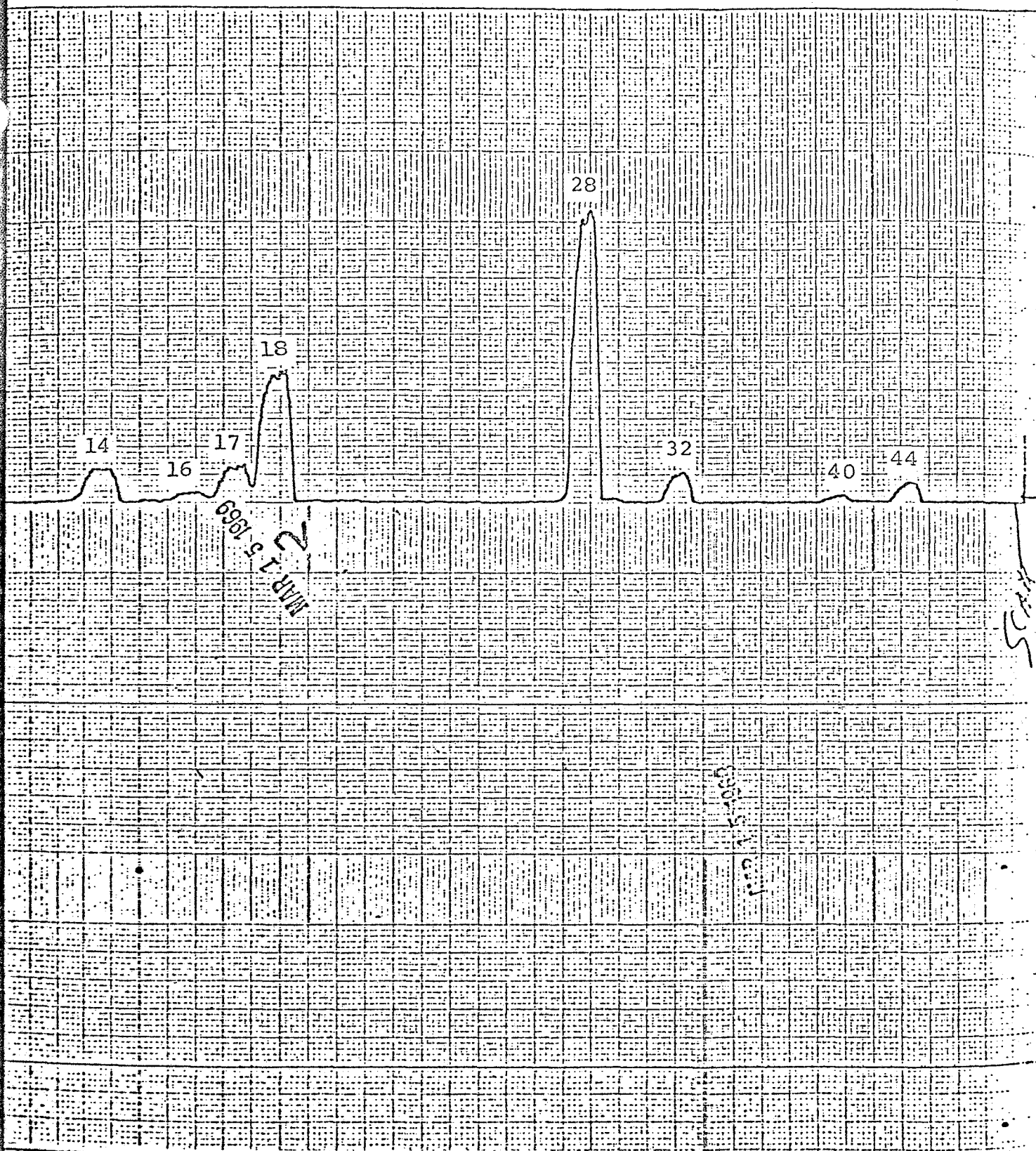


Figure 14. Mass Scan on Breath Analysis System

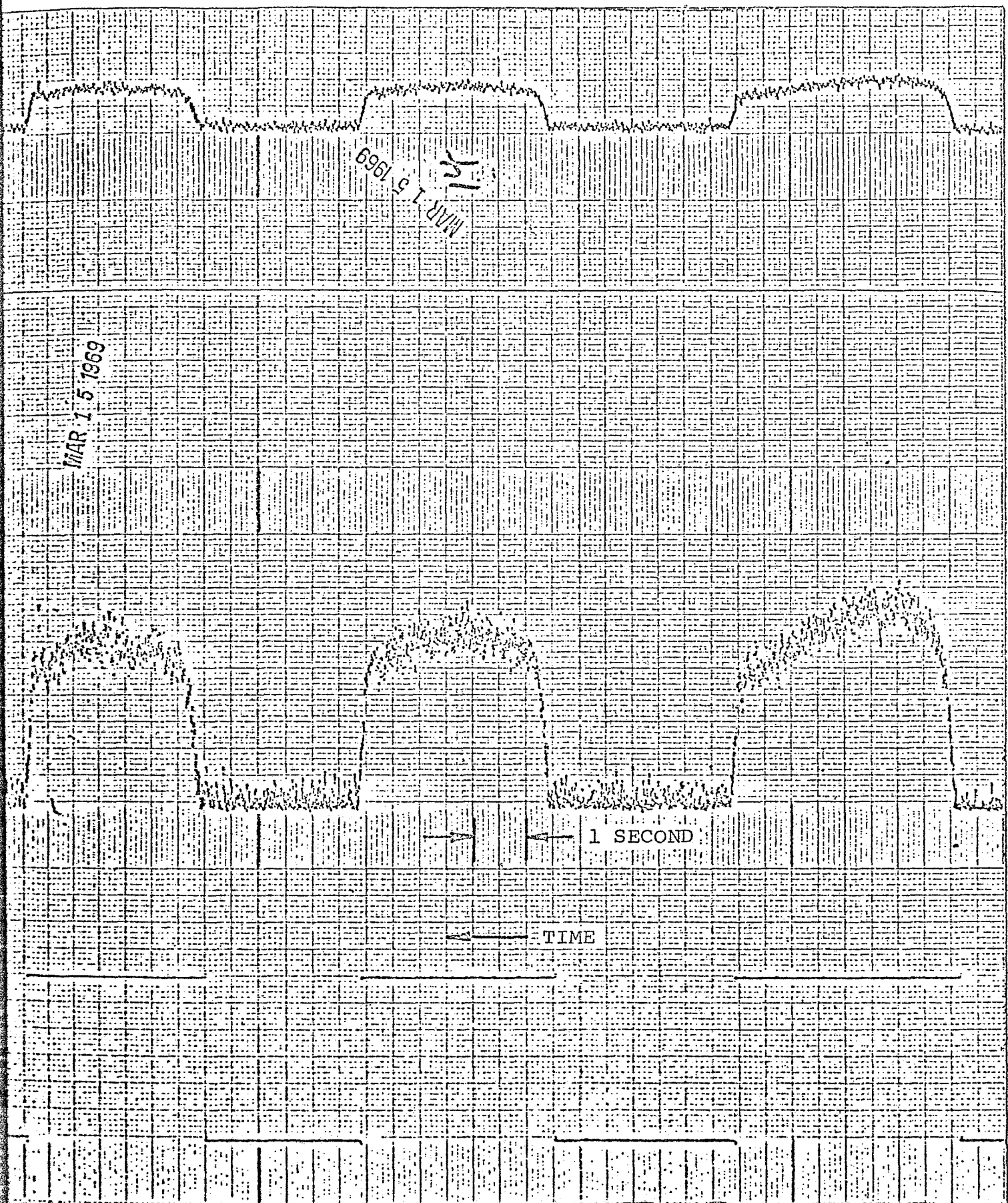


Figure 15. System Response to CO_2 during Inhale and Exhale Cycling.

SUMMARY AND RECOMMENDATIONS FOR A FOLLOW-ON PROGRAM

The feasibility of providing a mass spectrometer breath analyzer to fit under the chin of an astronaut within his helmet was demonstrated with the assembled apparatus. A set of prototype units, assembled to duplicate the critical constraints of this special application, has demonstrated a very rapid response to a step change in the composition of the breath gas. Ninety percent of the step change is made in 120 milliseconds.

The critical portions of the breath analyzer (the molecular leak and the associated ion pump) have been operated successfully in a system containing two mass spectrometers for a period of 11 weeks. There were no failures of the system to maintain vacuum during this period. The molecular flow characteristics of the leak have been demonstrated by the appearance of water vapor before CO_2 on the reversal of breathing from inhale to exhale. The small ion pump, with its well contained magnetic field, pumps nitrogen at a speed of 0.14 liter per second at a pressure of 10^{-6} torr.

The small quadrupole, with 2-inch hyperbolic rods spaced 0.150 inch, provides unit resolution with flat-topped peaks when it is excited at 6 megahertz.

Because the progress to date has unquestionably demonstrated that a miniature mass spectrometer breath analyzer system, including the difficult gas sampling devices, can be built, this program should be continued with the objective of achieving a working model. Starting with the molecular leak, the miniature actuator is to be assembled and operated. The problem areas here are concerned with the avoidance of over-closing the valve under any conditions of storage and operating

temperatures. The ion source geometry is entirely satisfactory, but the insulators used to support the electrodes must be amenable to operation at high temperatures. Further, they must effectively seal the ionizing region from the rest of the device without increasing its volume.

The quadrupole structure does not include provisions for the delayed dc ramp mode of operation. Advantage must be taken of the improved performance which results from the use of the delayed dc ramp. The technique used to fabricate the hyperbolic surfaces is unsatisfactory, and a better method (or machine shop) must be sought. The mass spectrometer and the ion pump must be integrated in a compact vacuum envelope, contoured to fit a man.

The instrument has been operated exclusively with laboratory-type electronics. Flight-type electronics, compatible with the severe limitation of the voltage and power which may be applied to any pair of leads used to interconnect the back-pack electronics and the quadrupole assembly, have yet to be designed and assembled. The appropriate data processing and read-out devices are included in the flight-type electronics.

Table 2 presents an itemized list of the tasks completed to date and also gives a summary indication of the scope of effort still required to carry this program to its conclusion.

Table 2

NAS9-8371 Statement of Work - Itemized Status Sheet

<u>TASKS</u>	<u>COMPLETION PERCENTAGE</u>	<u>COMMENTS ON ACHIEVEMENTS OR PERFORMANCES</u>
Phase A1, Leak		
Item 1. Study means of devising miniature needle valve.	100	
Item 2. Make experimental model, with no regard for refinement of actuator mechanism.	100	Excellent
Item 3. Attach to ultra-high-vacuum system with slow pumping speed and make experiments on valve operation.	100	Excellent
Item 4. Optimize valve parameters (exclusive of motion producing devices).	100	Satisfactory
Item 5. Design, devise and fabricate miniaturized valve actuator.	90	Unit was fabricated and nearly assembled. No tests run.
Item 6. Attach miniaturized valve and actuator system to vacuum apparatus and observe its performance.	0	
Item 7. Deliver five leak valves with actuators for performance tests.	40	
Phase A2, Ion Pump		
Item 1. Investigate the utility of various permanent magnetic materials for producing a magnetic field of 0.1 to 0.2 teslas in a small volume suitable for a Penning Discharge.	100	Very good
Item 2. Design self-contained magnetic circuit for producing appropriate magnetic field for the Penning Discharge.	100	Excellent

Table 2 - Continued

<u>TASKS</u>	<u>COMPLETION PERCENTAGE</u>	<u>COMMENTS ON ACHIEVEMENTS OR PERFORMANCES</u>
Item 3. Design and fabricate small Penning Discharge structures and experimentally determine their pumping capabilities as functions of pressure, magnetic field strength, cell geometry and applied potential	100	Excellent
Item 4. Choose most promising combination of geometry, magnetic field, and potential and build model.	100	Excellent
Item 5. Select titanium sublimator most suitable for use with pump model built in Item 4.	100	
Item 6. Optimize parameters of power applied to sublimator to achieve maximum deposition of titanium with minimum (average) power.	100	Very good
Item 7. Add titanium sublimator to pump model.	100	
Item 8. Perform experiments to determine operating characteristics of pump model described in Item 7.	80	Pump gave excellent performance without benefit of titanium sublimation.
Item 9. Make two ion pump assemblies and integrate with two of the leak valves to form self-contained system. These will be delivered to MSC for evaluation.	25	Only one self-contained ion pump was assembled.
Phase B, Ion Source		
Item 1. Study operating characteristics of filaments of various configurations and materials in oxygen-rich environments.	100	3 mil rhenium filament appears to be satisfactory.

Table 2 - Continued

<u>TASKS</u>	<u>COMPLETION PERCENTAGE</u>	<u>COMMENTS ON ACHIEVEMENTS OR PERFORMANCES</u>
Item 2. Based on theoretical studies made previously, design miniature ion source which incorporates:	100	Excellent, considering dimensional constraints.
a. Multiple filaments		
b. Differential pumping		
c. High-production efficiency for well collimated ion beam.		
c. Low production rate of photons which can reach detector		
Item 3. Fabricate and assemble ion source of Item 2.	100	Choice of sauerisen cement for insulating the support wires was poor. Serious outgassing and/or decomposition occurred on heating.
Item 4. Attach ion sources to system which includes small quadrupole, gas inlet and pumping system similar to that proposed for use on astronaut.	100	
Item 5. Perform experiments on assembly (Item 4) to demonstrate degree to which goals have been achieved.	100	Goals appeared to have been met. Gas load from saurisen clouded experimental data.
Item 6. Optimize parameters.	10	Only preliminary experiments were performed with breath gas on entire system.
Item 7. Prepare refined ion source for attachment to miniaturized quadrupole mass spectrometer of Phase C.	50	Geometry of field forming surfaces appeared to be quite satisfactory. Use of sauerisen for mechanical support is questionable.

Table 2 - Continued

<u>TASKS</u>	<u>COMPLETION PERCENTAGE</u>	<u>COMMENTS ON ACHIEVEMENTS OR PERFORMANCES</u>
Phase C		
Item 1. Design, fabricate and assemble a miniature quadrupole mass analyzer to match the ion source developed in Phase B.	100	Selected vendor was unable to hold dimensions to target tolerances.
Item 2. Integrate ion source, quadrupole, and a secondary emission multiplier into an assembly of minimum volume.	100	Package was placed in laboratory vacuum envelope.
Item 3. Energize assembly with laboratory electronics (supplied by Earth Sciences) and make experiments to explore the relationship between performance capabilities and the power level of the excitation of the rod system.	100	System gave every indication of meeting performance requirements except for slight abnormalities of peak shape which are attributed to rod contour imperfections.
Item 4. Review findings with Manned Spacecraft Center and mutually decide the preferred combination among the variables of Item 3.	0	
Item 5. Design, fabricate and test flight-compatible electronics, guided by choices made in Item 4.	0	
Item 6. Attach system comprising the ion source, quadrupole, and the detector to a laboratory vacuum apparatus (supplied by Earth Sciences). Using laboratory type gas inlet system, observe the performance of the mass spectrometer system to breath gas.	100	Tests were made with miniature leak, lab actuator.

Table 2 - Continued

<u>TASKS</u>	<u>COMPLETION PERCENTAGE</u>	<u>COMMENTS ON ACHIEVEMENTS OR PERFORMANCES</u>
Item 7. Replace the laboratory vacuum and inlet systems with those developed in Phases A and B and repeat the experiment described in Item 6, using electronics of Item 5.	0	
Item 8. Deliver to Manned Spacecraft Center the operating instrument used in Item 7.	?	Item 6 equipment was delivered.

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- (1) W. M. Brubaker, Space Charge and Potential Distributions in the Penning Discharge, Proceedings 20th Annual Conference Physical Electronics, MIT, 1960.
- (2) W. M. Brubaker, A Method for greatly Enhancing the Pumping Action of a Penning Discharge, Sixth National Symposium on Vacuum Technology, 1959.
- (3) W. M. Brubaker, Improved Quadrupole, Advances in Mass Spectrometry, Vol. 4, page 293.
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- (5) W. M. Brubaker, N. W. Bell, and F. B. Wiens, Quadrupole Mass Spectrometer for Atmospheric Studies in the 50- to 90-Kilometer Range, Twelfth Annual Conference on Mass Spectrometry and Allied Topics, 1964.

ACKNOWLEDGMENTS

It is a pleasure to acknowledge the benefits and stimulations which resulted from the many technical discussions held with Paul W. Schlottman, MSC, the technical monitor of this project. Fred Pickett's skill in designing and assembling the leak, pump, and mass analyzer were vital to our progress. He was ably assisted by Don Dyer, particularly in the fabrication and assembly of the miniature ion source. Don made the precision jig for holding the rods in place for the final assembly. Jim Granger designed and assembled the vacuum system used to calibrate the leak and the pump. Further, he performed the tests of these devices, and prepared the data for this report. Phil Klasky designed and assembled the control circuits for the filament life tests, and operated the apparatus to obtain the filament life expectancy data.

APPENDIX I

CALCULATION OF TITANIUM CONSUMPTION RATE DURING GETTERING

Assumptions:

1. One atom of titanium is required to getter one atom of nitrogen (or other getterable gases).
2. Availability of titanium sputtered from cathode of discharge is ignored.

Calculations:

One torr-liter of a diatomic gas (N_2 or O_2) at room temperature contains about 3.25×10^{19} molecules (or 6.5×10^{18} atoms). If these molecules react with titanium in the manner postulated, the mass of titanium involved is $(6.5 \times 10^{18} / 6 \times 10^{23}) \times 48$, or 5.2×10^{-3} grams. That is, 5.2 milligrams of titanium are used in the gettering of one torr-liter. If the gas load is admitted to the getter pump at a rate of Q torr-liters/second, the amount of titanium consumed in one day is

$$60 \times 60 \times 24 \times 5.2 \times 10^{-3} \times Q = 4.5 \times 10^2 \times Q.$$

It is possible to load one milligram of titanium on a support wire of tantalum, tungsten, or molybdenum. It is feasible to consider supplying 2 milligrams of titanium on multiple filaments which can be used in sequence. Our goal of a leak rate of 10^{-7} torr-liters/second appears to be achievable.

BASED ON THESE ASSUMPTIONS, the titanium supplied by the sublimator would last

$$2 \times 10^{-3} / 4.5 \times 10^2 \times 10^{-7} = 45 \text{ days.}$$

APPENDIX II

Abstract of a Paper Presented at Vacuum Science & Technology Symposium, Southwest Section of the American Vacuum Society, Los Angeles, May 7-9, 1969

VACUUM SYSTEM FOR ASTRONAUT BREATH ANALYZER**

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Earth Sciences, A Teledyne Company, Pasadena, California

A molecular leak and miniature ion pump have been combined to form the dynamic portions of a vacuum system for an astronaut breath analyzer. The micro-leak admits the breath gas from ambient (atmospheric) pressure directly into the vacuum system under molecular flow conditions. The less-than-golf-ball-size ion pump handles the entire gas load from the micro-leak. Gas flow rates of several times 10^{-7} torr/liters/second are pumped at a speed of about 0.1 liters/second at pressure of several times 10^{-6} torr. The entire vacuum system, which contains the quadrupole mass analyzer, fits under the astronaut's chin, inside the helmet. The electronic apparatus is carried in a back pack.

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