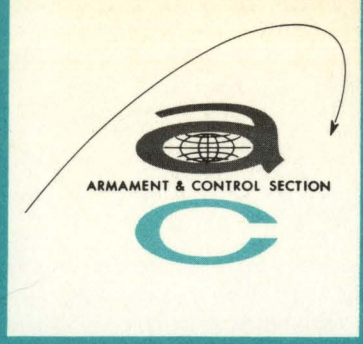

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TIS

REACTIVE METAL VACUUM PUMPING

P. H. Peters

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Title Page

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TITLE

REACTIVE METAL VACUUM PUMPING

ABSTRACT The operation of a novel pump for producing ultra-clean and high vacuum in the 10^{-8} to 10^{-7} torr region is described. Pumping processes include gas-metal diffusion and chemical gettering, with heat as the main source of input energy. Four models of different design were constructed, one of which is particularly suited for use with an electrostatic gyro. Some preliminary measurements of pumping speed are presented. Difficulties encountered

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CONCLUSIONS ABSTRACT - and their solutions are discussed. The general application of this type of pump and its use in vacuum systems utilizing no mechanical or cryo-forepumping are outlined.

CONCLUSIONS: One form of pump of all-metal construction requires a power input of about 10 watts and has an average pumping speed of 0.2 liters per second for atmospheres free of inert gas. No magnetic field or high voltage are required. Arrays of such pumps can be formed to afford greater pumping capability. The pump is small, sturdy, bakeable to high temperature, is uncritical of starting pressure, and capable of numerous exposures to air. Work is underway to incorporate means for pumping inert gas and for self-monitoring gas pressure.

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DEFENSE ELECTRONICS DIVISION — ARMAMENT AND CONTROL SECTION

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CONTENTS PAGE

Report No R62APS57

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TABLE OF CONTENTS

FIGURES

(This report covers development work on DA X-75 during the period March 1 to mid-November 1962).

1.0 INTRODUCTION

In recent years the production of ultra-clean and high vacuum has become an essential requirement for the proper conduct of work in many fields of applied science. Many new industrial devices are being developed which depend greatly on maintaining conditions free of gas and other contaminants. The business of vacuum is growing at an unprecendented rate and participants in this business can foresee a long-term and profitable future.

Ionic and cryopumping are examples of new techniques which have greatly aided in the achievement of very low pressure at high pumping speed. Selective permeation, chemical reaction, and chemisorption in metals also hold promise as means for producing high vacuum, especially in applications where a magnetic field is undesirable or cryo apparatus cannot be used.

1:1 A REACTIVE-METAL DIFFUSION PUMP

A novel vacuum pump employing the processes of permeation and chemical reaction was proposed at Advance Engineering and a feasibility demonstration performed early in 1962. An experimental program to develop such a pump was begun on DA X-75.

In particular, it was recognized that the P^2-pump, as it is called, has many features which make it attractive for use with an electrostatic gyro. Accordingly, the emphasis of the DA program was directed to the development of a pump for this special need.

1:2 APPLICATION TO THE ELECTROSTATIC GYRO

Many constraints are placed on the nature of a vacuum pump for use with an electrostatic gyro. Conventional ionic and diffusion pumping have not proven satisfactory for an operational gyro. For example, the presence of even a weak magnetic field can spoil gyro behavior. Any particulate or vapor contamination

must also be avoided. The pump should be small enough to be integral with the gyro package and resist the same vibration and shock to which the gyro is subjected. It further should generate a minimum of heat and be capable of maintaining a vacuum in the low 10^{-7} torr region over extended periods of time. A gyro pump must be capable of numerous exposures to air when the gyro is opened for inspection or repair.

1:3 SUMMARY OF REPORT

This report discusses briefly the characteristics of the typical vacuum environment and describes the pumping processes and materials involved in P^2 type vacuum pumps. It summarizes the work done on four basic pump designs, describing the operation and constructional details of laboratory models. Some measurements of pumping speed, power input, are presented. The status of the program is brought up to date and further work recommended. Attention is directed to Sections 7 and 8:5 which describe the design of pump of major interest.

2:0 THE VACUUM ENVIRONMENT

Suppose that a metal vacuum chamber, such as that used in the ESG, has been mechanically pumped to a pressure near 10^{-4} torr through a liquid nitrogen trap and has been closed off from the pump and trap. The residual gases remaining include H_2, O_2, N_2, CO, CO_2, H_2O, $C_n H_m$, and the inert gases, largely He and Ne, and a small amount of Ar. One or more of these gases may continue to evolve as outgassing of the chamber walls and parts proceeds. The extent of the outgassing will depend upon the degree of bakeout and pre-treatment of the surfaces within the vacuum, as well as upon the operating temperature of these surfaces and the presence or absence of electric and magnetic fields.

Inert gases are not permeable in materials and their presence in the vacuum chamber largely depends on their initial concentration in the chamber before any pumping begins. Argon is of course removed by the liquid nitrogen trap to a very low partial pressure. The partial pressures of Ne and He are respectively 10^{-9} and 10^{-10} torr at a seal-off pressure of 10^{-4} torr.(1) If a system pressure less than 10^{-9} torr is not

required, these gases should present no problem to a pump which does not have an inert gas pumping capability. If, however, there is even a small leak in the system to air, some constituent air gases will occur. Even though the pumping speed for these latter gases is increased to compensate for the leakage, the base pressure will continue to rise as the inert gas content builds up. In a demountable system such as the electrostatic gyro there are often several metal-gasketed seals and glass windows and there is always a finite probability that a slow permeation of air, including inert gas will occur. Over long periods of time, therefore, some means should be provided for pumping this type of gas. In its simplest form inert gas pumping is not provided in the P^2-pump, although this capability can be included in a number of ways.

One of the most abundant gases to be found in the chamber is hydrogen. It is supplied by the outgassing of metal parts. Water vapor is also found in abundance, particularly in a system like the gyro which cannot be vacuum baked to a high temperature. One way of pumping water vapor is to dissociate it thermally on a hot surface and then to remove the resulting hydrogen and oxygen separately. Hydrogen may be removed from the chamber by trapping it in a getter material chemically; or ionically, as is done in the ion pump. In the case of the ion pump, the trapping is not permanent and the hydrogen will re-evolve when the pump is subjected to heating. Physical removal of the hydrogen, therefore, has a distinct advantage over an internal trapping method since re-evolution cannot occur.

2:1 PUMPING BY GETTERING

Gases such as O_2, N_2, CO, CO_2, C_nH_m can be gettered permanently by a number of metals and alloys.[2] The gettering process involves exposing a clean metal surface to these gases and, in the case of bulk-type gettering, operating the surface at a high temperature to promote chemical reaction and chemisorption. Oxides, nitrides, and carbides of the getter material are not easily dissociated by further heating or cooling, and in this sense the pumping action is permanent.

One metal which has unique advantages as a bulk getter is titanium. When it is heated above 850°C it exhibits the

property of dissolving its own oxide in such a way as to maintain a chemically active surface not easily poisoned by ambient gases. When operated in this temperature range titanium will getter rapidly the above gases except hydrogen and the inert gases. Hydrogen gettering by titanium is thermally reversible and is greatest at low temperatures over a range from room temperature to about 400°C. Zirconium and Ti-Zr alloys have similar properties. The following tables show the chemical gettering properties of titanium operating between 700 to 1100°C as measured by Stout and Gibbons.(3)

Gettering Rate in micron liters/cm^2 - min

	Temperature °C				
Gas	700	800	900	1000	1100
Nitrogen	0.4	0.9	2.0	5.0	20
Oxygen	11	127	too rapid to measure		
Carbon Dioxide	0.15	1.7	3.9	15	46

Also, the quantities of gas that can be gettered by titanium are as follows:(3)

Gas	Amt. in micronliters per milligram
Nitrogen	160
Air	130
Oxygen	90
CO_2	50

Water vapor is dissociated by hot titanium, the oxide is gettered and the hydrogen set free within the vacuum chamber. The action on hydrocarbons is similar, in that the carbon combines and the hydrogen is again set free. If the metal can be heated above 1000°C some evaporation will take place and act to remove ambient gas including inert gas by evaporative burial on cool surrounding surfaces.

2:2 PUMPING BY PERMEATION

It is seen that a heated titanium surface is effective in permanently removing ambient gases except hydrogen and the inerts. If in addition a means were available for removing hydrogen, then all gases in the chamber except the inerts could be pumped.

A novel way to pump hydrogen from the chamber is to remove it by permeation through a heated membrane of palladium or palladium-silver alloy. Palladium is selectively permeable to hydrogen and has long been used in the purification of this gas. (A 75-25 Pd-Ag alloy has recently been developed which inhibits grain growth and increases the life of the metal as a vacuum-tight material).

The process of removing hydrogen from a vacuum chamber to air involves more than ordinary diffusion of the gas. Diffusion requires that the partial pressure of hydrogen in the chamber be always slightly greater than it is in air; namely, 3.8×10^{-4} torr. Attaining a vacuum better than this would normally not be thought possible. However, during the course of his work in 1962 Dr. J.R. Young,[4] working with a palladium hydrogen-pump, found that the pressure in a chamber filled only with hydrogen could be reduced many orders of magnitude below 10^{-4} torr. In effect, the hydrogen appeared to diffuse through the palladium from a region of low pressure to a region of high pressure. He found further than an oxidizing atmosphere on the exit side was important in producing the effect. Atomic hydrogen emanating from the metal was found to react with oxygen to form water vapor. In one experiment it was shown that no increase in the partial pressure of hydrogen occurred on the exit side as a result of hydrogen permeation from the chamber being evacuated. It was concluded that as long as oxygen is present the partial pressure for atomic hydrogen in the immediate vicinity of the exit surface will be negligible, even though the partial pressure of molecular hydrogen in the atmosphere is relatively high. This explanation was supported experimentally and hydrogen pumping to extremely low pressures near 10^{-9} torr was demonstrated. In fact the data indicates that the effective partial pressure of hydrogen on the exit side was in the order of only 10^{-12} torr. From Young's experiments it was clear how a palladium hydrogen sieve can actually pump hydrogen.

Hydrogen pumping through heated palladium is subject to some poisoning on the vacuum side particularly by hydrocarbons and films of some metals. Attention was given to a method for protecting the surface without affecting the pumping action and the idea that the palladium could be isolated from the chamber with a separate vacuum tight membrane of hot titanium was suggested.[5] This membrane would be located between the chamber and the palladium. Such an arrangement involves a dual diffusion process for hydrogen; the gas passes first through the titanium and thence through the palladium to air.

Titanium itself will act as a hydrogen pump to air at a temperature in the 600°C region. However, the pumping speed is slower than it is in palladium, according to Young, J.E. Beggs[6] has operated a 0.040-inch thick titanium disk separating a vacuum enclosure from air at 600°C for 16,000 hours and at about 700°C for more than 1000 hours without loss of vacuum. At 800°C the metal begins to oxidize rapidly in air and its life is materially shortened. It is possible that a titanium hydrogen-leak could be made to have a life and a permeation rate of practical interest. However, this possibility was not investigated experimentally during this DA program.

3:0 ELEMENTAL FORMS OF P^2-PUMP

If both the titanium and palladium pumping metals are exposed to the residual gases in the vacuum chamber they may be said to operate in a parallel manner as shown in Figure 1a. If the titanium metal entirely seals the palladium from the chamber, the metals operate in cascade as shown in Figure 1b and hydrogen must pass through both in order to be removed to air. The pressure in the inner chamber must be lower than that in the chamber being pumped in order for hydrogen to diffuse through the titanium.

The location and the configuration of the titanium element in Figure 1a with respect to the palladium is not critical. Heat may be supplied to each metal from separate sources or from a common source. Hydrogen pumping through palladium can be sustained over a wide range of temperature and therefore it can be operated at a temperature lower than that at which the titanium is operated. This condition is brought about naturally

in most configrations since the palladium is in contact with air and is naturally cooled by convection. The titanium element may take many forms, although the pumping speed increases with surface area exposed to the vacuum. The difference in temperature across the surface should be kept small in order to keep the dissociation pressure of hydrogen above the surface uniform. Thus one section of the getter will not tend to sorb more hydrogen than does any other section.

The palladium member may also take many forms. For example, instead of a flat membrane it may consist of one or more thin-walled cylinders each closed at one end and brazed to a plate of stainless steel. A wall thickness of about 0.010-inch or less has been found satisfactory for rapid permeation. In this thickness the metal is self-supporting even at operating temperatures as high as 1000°C. A greater thickness is not necessary and would involve a higher cost. Palladium has a density of 12 gm per cc and is currently priced at $0.80 per gram.

3:1 HEATING METHODS

Heat may be applied to the metals in many ways. If an infra-red window is properly located in the vacuum system it is possible to employ solar heating to affect pumping. Electron bombardment or induction heating areother possible ways to heat the elements. Electron bombardment, however, involves the use of an electron emitter which is subject to poisoning and possible rapid deterioration, especially at high starting pressures. Electric current can be passed directly through the metals to cause heating. Heating with electrical resistance heaters is also a satisfactory method. Such heaters may be located either on the air side or on the vacuum side of the palladium. It may be noted that in the cascade arrangement of Figure 1b an electrical heater may be interposed between the two metal diaphragms within the intermediate vacuum space, and so may be protected from exposure to air whether or not the pump is attached to a vacuum chamber.

There are some advantages in heating the metals from the air side. First, there is no need to contend with the outgassing of an internal heating coil. Second, the so-called water cycle

effect can occur which results in a deposition of a heater material such as tungsten on the cool walls of the vacuum system causing a rapid deterioration of the heater. The hot tungsten causes cracking of H_2O and a subsequent release of atomic hydrogen which reduces tungsten oxide arriving at the walls. This effect is particularly troublesome at high starting pressure. Embrittlement of the heater can also occur when atomic hydrogen is released by cracking. The use of an internal heater also involves the use of at least one electrically insulated feed through. This must be of the metal-ceramic type to prevent permeation of atmospheric helium. If the heater can be located outside the vacuum, no insulator is required and the heater is not subject to the water cycle effect. Then the vacuum pump may be fabricated entirely of metal. An external heater is of course easily replaced without opening the chamber to air. Longer life and reliable vacuum conditions are thereby assured.

3:2 PARALLEL PUMP--BASIC TEST

The first demonstration of a reactive-metal diffusion pump was accomplished with a parallel system shown schematically in Figure 2. This system consisted of a volume of about one-half liter and included a set of one inch diameter vacuum fittings, a commercial palladium hydrogen-leak, a RG-75 Veeco vacuum gage, a thermocouple gage, and a vacuum valve to shut the system off from a forepump. In the tests to be described the forepump was an ordinary 1.4 liter/sec rotary pump. No cryo or vapor trapping was employed and the system was filled with air at the start of pumping. The hydrogen leak was heated by a heating coil external to the vacuum environment.

A titanium ribbon 3/16 x 0.005 x 9 inches long was suspended in the main one-inch pipe and an electric current was passed directly through the metal in order to heat it to gettering temperature. The metal ribbon was not vacuum fired prior to use and so contained a considerable amount of hydrogen. Thus, as soon as heat was generated within the ribbon a large quantity of hydrogen was evolved and caused a sharp rise in system pressure. This gas was quickly removed from the system by the hydrogen leak, however.

After forepumping to about 500 microns (0.5 torr) the electric power was applied to both pumping elements, to heat the palladium and the titanium to approximately 500°C and 1000°C respectively. After an initial increase the pressure fell about three orders of magnitude in about 6 seconds to about 0.5×10^{-3} torr corresponding to a constant pumping speed of about one liter/sec. This experiment was most impressive when the relative size, weight, and power consumption, simplicity, and silent operation of the reactive metal pump were compared with similar characteristics of a rotary pump operating in the same pressure range.

Since no cryotrapping had been used, the partial pressure of argon was assumed to have been in the same proportion at 500 microns as it is at atmospheric pressure, namely one percent. When pumping began at 500 microns the pressure of argon should have been 5×10^{-3} torr. If no inert pumping occurred, this would also have been the base pressure of the reactive metal pump. During the experiment the initial rapid pumping slowed down markedly below this pressure and no doubt some argon has been pumped by evaporative burial.

The temperature of the titanium was then increased to about 1150°C and the pressure again began to decrease very slowly. At this temperature, evaporative pumping was much greater and it was reasoned that inert gas pumping was taking place. By alternately flashing and cooling the titanium it was possible to reduce the pressure into the 10^{-5} torr region. The palladium leak was in operation throughout the process.

In other tests the forepump was allowed to reduce the starting pressure to about 5 microns. A pressure of 3×10^{-7} torr was produced with the reactive metal pump. Without some capability for pumping inerts, the base pressure would only have been about 5×10^{-5} torr.

The power input to both the leak and the ribbon was estimated as about 150 watts. No thermal shielding of the ribbon was used and heat was removed by water cooling of the stainless-steel tube in which the ribbon was suspended.

The experimental set-ups were aimed chiefly at proving feasibility. In some of the early work wax seals, rubber hose, and soft solder were employed to join system components.

Seldom were the systems subjected to adequate bakeout. It is interesting that high vacuum conditions could be produced with relative ease and with minimum attention to sources of outgassing. Tests of the coaxial pump to be described were carried out more carefully, however.

4:0 PUMPING FROM STANDARD PRESSURE

At the time it seemed possible that the pump would be capable of starting from atmospheric pressure if a suitable flushing gas were used. Thus one might introduce a gas such as nitrogen which could be gettered by the titanium. The gas would have to be argon free, of course. Assuming a total capacity of 160 micron-liters per milligram it would take six grams of titanium to getter all the nitrogen in a one liter system at standard pressure. If a suitable method of achieving full saturation of the titanium and a way of handling the saturated material resulting from the reaction could be worked out, this method of pumping might find use in special applications where a rotary pump could not be accommodated. For example, the titanium used for forepumping could be located in a cannister attached to the vacuum chamber, as an appendage capable of being pinched or valved off after a pressure of a few microns had been reached. The used titanium could then be replaced for a future pump-down and the cannister again made ready for use. A reactive metal diffusion pump would then be brought into action to produce and maintain a high vacuum.

Mechanical fore pumps which utilize a pumping oil are not easily adapted for aerospace use where the pump may be required to operate in any position and under the influence of a varying gravitational force. In such situations the use of a flushing gas to achieve forepumping from the ambient pressure of a space vehicle might prove advantageous.

The principle might be extended in general to the fore-pumping needs of a thermoplastic recording apparatus. In TPR the tape transport and storage volumes are held at a pressure greater than 10 microns and the electron gun chambers are operated below 10^{-5} torr. These pressures could be maintained by continuously gettering a small quantity of make-up nitrogen admitted through a capillary leak. The power involved could

be less than is required to operate a mechanical pump and advantages in size, weight, pressure control and cost can be foreseen. The action would of course be silent and vibration free. The titanium could be located in cannisters and these could be refilled with a fresh titanium charge from time to time. If the system were exposed to air it could be flushed before gettering began. Note that this method does not require the use of cryo-pumping.

The method is also applicable to the pump-down step in preparing an electrostatic gyro for operation. The cannister for gettering the flushing gas could be pinched-off and removed when the pressure in the gyro housing was low enough for a reactive-metal diffusion pump to operate satisfactorily.

Hydrogen flushing could also be employed for affecting evacuation of a vessel from standard pressure. In this case the palladium hydrogen leak, such as that used in a reactive-metal pump, would pump the system down directly. The titanium element would not be heated until a sufficiently low pressure of hydrogen had been reached for normal high vacuum gettering to occur. The cool titanium would not be affected by the hydrogen flushing procedure since its surface would have been oxidized by prior exposure to air. Except for the potential danger of explosion that prevails in any system utilizing hydrogen, this type of flushing could be quite effective and could be generally applied. With this technique the flushing gas is removed from the system. It is not taken up by a getter and no material is consumed.

The small chamber used with the electrostatic gyro could be placed in a standard hydrogen bottle to fill it with hydrogen, and then could be sealed off in this atmosphere. After removal of the housing from the bottle, the hydrogen leak in a reactive metal pump could be heated and the hydrogen allowed to pump to the atmosphere. Thereafter the titanium could be heated to gettering temperature and the pressure further reduced to a level suitable for operating the gyro.

Some thought has been given to the use of a cannister filled with titanium hydride and attached to the gyro chamber as a source of flushing gas. When heated, the cannister would yield absolutely pure hydrogen in large quantities sufficient to thoroughly flush the chamber into an atmosphere of air, or better still, an atmosphere of nitrogen. When the flushing

was accomplished, the chamber could be closed both to the cannister and to the outside. Pumping by a reactive metal diffusion pump could then proceed as just described.

5:0 DEMOUNTABLE CASCADE PUMP

Figure 3 shows the structure of an experimental P^2-pump using a cascade arrangement of titanium and palladium pumping elements.

The titanium element is made in the form of a cylinder closed at one end and attached to a titanium flange at the other. This element is mounted within a stainless steel cylinder which forms the main pump housing. In the present art, it is not possible to directly join stainless steel to titanium by brazing or welding. The two metals form an eutectic alloy and the eutectic temperature is always lowered by the addition of a third brazing metal. Thus, a runaway action develops, melting large amounts of the parent metals. Also, the expansion coefficients of the two metals are quite different. These difficulties were avoided in the model by using a copper gasketed joint between the titanium flange and the steel housing.

The palladium element included four tubes about two inches long, 0.150-inch in diameter and having a wall thickness of 0.009-inch. Each tube was mounted on a stainless steel exit plate and closed at the end farthest from the plate. For convenience of assembly this plate was also attached to the stainless housing by a copper gasketed joint. The palladium tubes extended within the inner chamber of the housing and to within about ¼ inch of the closed end of the titanium sleeve. A vaccum tight chamber is created when both pump elements are mounted to the housing. A No. 413 Hoke valve was also brazed to the exit plate to permit the evacuation of the inner chamber in making the pump ready for use. All components were vacuum fired prior to assembly. The pump should be baked thoroughly during the evacuation of the inner chamber, although, in the experiments, this process was not always carried out.

An electrical heating element is also shown in Figure 3 surrounding the titanium sleeve. Electrical connections were made to two ceramic-metal feedthroughs in the exit plate. The heater was made of tungsten and coated by a shooping process with

alumina powder, hydrogen fired,and vacuum fired before assembly. The heater was made of 0.040-inch diameter wire wound in a bifilar manner to minimize the self-magnetic field. At 1000°C the power consumption was about 150 watts.

Heat radiation to the housing was minimized by a cylindrical radiation shield placed within the housing. This shield consisted of seven turns of perforated 0.002-inch molybdenum foil and extended the full length of the inner chamber. Radiation loss from the heated titanium is minimized by the choice of a cylindrical instead of a planar configuration for the pump. Radiation is confined to a cross section equal to the area of the hole in the titanium sleeve. This area is a small fraction of the total area of the sleeve itself. The greatest loss of heat is in the form of conduction along the titanium sleeve to the titanium flange and pump housing. Operating temperatures of the housing in ambient air were in the neighborhood of 150-200°C with full power input. Photographs of the pump and its component parts are shown in Figures 4 and 5.

In a typical test of pumping action this cascade pump was connected to a vacuum system such as is shown in Figure 6. With the equipment on hand it was difficult to produce a pressure lower than 5×10^{-5} torr in the inner chamber. The main chamber consisted merely of the vacuum plumbing and gages. Starting pressures in the main chamber ranged from 10^{-2} torr to 5×10^{-5} torr and in general were produced with a rough pump and a liquid nitrogen trap.

Conclusions about the pumping behavior must take into account the relatively poor vacuum produced within the inner chamber. When the Hoke valve was closed, the pressure in the inner chamber could not be monitored. Thus leaks and outgassing often were present and could easily inhibit hydrogen throughput. Had it been possible to achieve a permanent vacuum within the inner chamber, like that produced in a well-sealed and degassed vacuum tube, the behavior might have been considerably different. Much judgement has had to be applied in interpreting the effects observed and the types of gases which may have been in the chambers on each side of the titanium sleeve during the various stages of pumping.

5:1 PUMPING CHARACTERISTICS

Figure 7 shows the performance of a demountable cascade pump when started at a pressure of 10 microns in the outer vacuum chamber. The inner chamber had been pumped to a pressure near 5×10^{-4} torr. Fore pumping was done with a mechanical pump and liquid nitrogen trap. Initially, the tungsten heater and pump body were at room temperature. Electrical power was applied and the variations in pressure and temperature with time measured. At first the pressure in the outer chamber rose an order of magnitude, presumably due to the evolution of hydrogen from the titanium and water vapor from the walls of the system. Soon the pressure began to fall rapidly to the low 10^{-4} torr region at a time when the color of the titanium was becoming a very dull red, near 650°C. Thereafter the pressure began to rise more slowly and at about 5×10^{-4} torr tended to level out. At this point the heater power was turned off and the pressure again fell to a value near 5×10^{-5} torr. The heater power was turned on again at about one-half its previous level. The titanium was not hot enough to glow visibly and the palladium temperature was about 100°C. Under this latter condition the pressure held at about 3×10^{-5} torr.

The temperature of the palladium was measured with a thermocouple inserted in one of the palladium tubes. When the titanium temperature was near 1000°C, one could look into the Pd tubes and see a color corresponding to a tip temperature near 700°C. The Pd tubes were much cooler, about 150°C, near their base at the exit plate.

The initial upward surge, the rapid pumpdown, and the slow rise in pressure to a level about an order of magnitude below the starting pressure was a characteristic pumping pattern of the pump. When the heater was turned off, the pressure would drop another order of magnitude as the elements cooled. Evidently outgassing of the pump and system was involved, being greatest when the pump was at the highest temperature.

Note that the pump removed gas readily at least to a pressure corresponding to the initial pressure of the inner chamber between the pumping elements. Had it been possible to pump the inner chamber to a lower pressure before closing the Hoke valve, a base pressure lower than 5×10^{-5} torr might have been achieved. It was assumed that argon had been removed at least into the 10^{-5} torr range by the forepumping.

It had been recognized that the titanium flange would operate at a much lower temperature than the hot titanium sleeve. Further, there had been concern about the cooler flange acting as a sorber of hydrogen such that this gas would tend to go to the cool titanium instead of passing through the palladium leaks. However, it was felt that eventually the palladium would pump the system free of hydrogen. While this is true, the time required could be extremely long.

Tank hydrogen was passed over the palladium leaks on the air side of the pump and its passage through both the Pd and Ti elements to the vacuum chamber observed. It was easy to produce dual diffusion to the vacuum chamber, creating pressures above 1000 microns in a matter of seconds. When the tank hydrogen was removed, the system pressure would begin to drop and would approach the pressure existing before the hydrogen was applied. However, each time hydrogen was applied, and subsequently allowed to pump back out, the final pressure achieved increased. Apparently, some of the gas remained in the titanium and raised the equilibrium pressure for hydrogen above titanium. At a given temperature the equilibrium pressure for hydrogen increases as the square of the concentration of the gas in the metal.

The equilibrium concentration of hydrogen at atmospheric pressure in titanium as a function of temperature is shown in Figure 8.[7] Below about 400°C the concentration is high and temperature-insensitive. The ability of the metal to hold large amounts of hydrogen when relatively cool could markedly increase the time required for removing this gas by diffusion through a hotter region of the metal, such as the sleeve. Thus, the existence of a temperature gradient along the titanium acts to limit the base pressure of the pump. If the titanium element could have been designed to operate at a uniform high temperature the tendency to saturate would have been greatly reduced.

For example, the flange might be made of a material different than titanium, having a hydrogen solubility which is low and varies little with temperature. In the model the thickness of the sleeve was about 0.050-inch, a compromise among factors such as structural strength, life, and diffusion rate. A thinner sleeve would have permitted more rapid diffusion and no doubt a more rapid diffusion rate for hydrogen.

It is interesting to note that the permeation of a gas through a metal is dependent on the product of solubility of the gas in the metal, the diffusivity of the gas through the metal, and the pressure gradient across the metal according to the expression,[8]

$$P \sim S \cdot D \cdot \Delta P/d$$

Thus, a finite solubility is essential for permeation. Both solubility and diffusivity are exponentially temperature dependent. In titanium, if the temperature is low, the solubility is high, but the diffusivity is very low and the gas does not permeate. At high temperature the diffusivity is high and the solubility low, but permeation can occur. Evidently, there could be an optimum temperature which will achieve the greatest permeation. The tests seemed to indicate that the pumping speed was greatest when the metal was in a temperature range near 800 C, although ordinary outgassing of the system made it difficult to be certain that the effect observed was due solely to a variation in the permeation of hydrogen through titanium with temperature. The formula also points out that if the inner chamber pressure exceeds that in the vacuum chamber, permeation will reverse in direction.

A failure of the palladium leak to remove hydrogen can also stop permeation through the titanium in a cascade pump. In the demountable model there were occasions when the palladium had been poisoned by surface films of hydrocarbons, and hydrogen diffusion stopped. Activity was restored by subsequent cleaning and vacuum firing of the palladium tubes, however.

6:0 METAL-CERAMIC CASCADE PUMP

Figure 9 shows a drawing and dimensions of another model cascade pump of metal-ceramic construction built during this work. This pump was considerably smaller than the demountable pump and was suited for mounting directly on the bell-jar housing of the Phase IV electrostatic gyro. A photograph of the pump appears in Figure 10.

A main feature of this design is that the members are joined by metal-ceramic seals in a vacuum environment. The

seals are made at a temperature near 1025 C. The inner chamber is evacuated at the same time to a pressure equal to that of the vacuum sealing system. Here again the base pressure of the sealing system was limited to the low 10^{-4} torr range when the brazing oven was at sealing temperature. If it were not necessary for the pump elements to operate at elevated temperature, the pressure of the inner chamber would fall lower than the seal-off pressure, as in a standard metal-ceramic vacuum tube. However, this is not true in the case of a pump, since the pumping action depends on operating most of the internal metal parts at a high temperature. Therefore, the seal-off pressure must be as low as possible to assure the best pumping behavior. Of course it may be argued that the inner chamber should pump itself, so to speak, and except for possible poisoning effects by residual gases in the sealing vacuum, this is true. Such effects could not be excluded, however, and the character of the sealing vacuum remained a matter of concern.

The drawing shows a 0.005-inch titanium sleeve closed at its extreme end and supported at its open end on a metal disk sealed to the pump body. In the model this disk was made of titanium and, as such, constituted a hydrogen sink. However, the sleeve itself was found to operate at a nearly uniform temperature along its length and the major drop in temperature occurred radially along the supporting disk.

The palladium element is a re-entrant tube extending partially within the titanium sleeve. A tight-fitting tungsten heating coil is slipped over the tube and extends within the titanium tube to a nub on a titanium plug welded into the end of the sleeve. Electric current in the range 2 to 4 amperes is passed from the disk, up through the sleeve, down through the coil, and through the palladium tube to another disk at the air-end of the pump. The applied potential ranged from 3 to 8 volts. The palladium was heated mainly by heat conduction from the hot tungsten coil. The disk at the air-end was also made of titanium in the model. The disks were made of titanium because titanium could be reliably sealed in vacuum to an available Forsterite ceramic. Other sealing methods might have been employed, but would have required considerably more development time.

The main body of the model pump was a long ceramic tube. A molybdenum foil heat shield was rolled within this tube. The vacuum port was made of palladium in the form of a thin disk. Pumping occurred through eight small 0.100-inch holes drilled near the center of the disk. The disk was sealed to the ceramic tube with nickel and titanium 0.0005-inch shims. The titanium electrode disks were sealed with nickel shims. Some attempts were made to seal a larger flange made of Inconel directly to the ceramic tube, but while the expansion rates are similar, the stresses developed in cooling always caused a cracking of the bond. Palladium was chosen for the port instead of titanium chiefly because it will wet to soft tin solder. The pump could then be joined and removed from the Phase IV gyro housing without using excessive heat. A welded joint would have been preferable, but the pump could not have been demounted without spoiling the flange on the housing.

6:1 PUMPING CHARACTERISTICS

In operation the pump showed characteristics similar to those of the demountable pump. Starting with a pressure in the high 10^{-4} torr range, a final base pressure of 1×10^{-6} torr was produced. While heat was being applied, the pressure initially rose and then dropped to the high 10^{-6} torr range. It then rose slowly to the low 10^{-5} torr region where it acquired a steady value, presumably when the temperature of the elements had reached equilibrium. Generally an increase in heater power would cause the pressure to increase, not decrease. It seemed that the outgassing rate was increasing faster than the pumping speed in this case. When the input power was lowered to about 2 watts, the pressure dropped steadily to a base pressure of 1×10^{-6} torr. In other tests a lower starting pressure of about 5×10^{-5} torr was used and the base pressure reached the 10^{-7} torr range.

In one case the vacuum chamber consisted of a Veeco gage and a short connecting pipe and a Hoke valve. The residual gas at 10^{-5} torr was analyzed on a mass spectrometer and found to consist mainly of hydrogen with a trace of carbon monoxide. Apparently hydrogen permeation was not occurring as expected. It was concluded that the hydrogen pressure in

the inner chamber was not being maintained sufficiently low by the palladium to permit diffusion through the titanium. In the particular model a piece of palladium had been inserted in the inner chamber as an optical shield. This piece was not in direct contact with a hot surface and its temperature was not under control. It may have influenced the permeation by increasing the equilibrium pressure of hydrogen in the inner chamber. It is believed that the seals closing the inner chamber were tight and its vacuum integrity remained unchanged, judging from the nature of the I-V characteristic of the heater coil. Hydrogen diffusion from the air side of the pump to the vacuum chamber was demonstrated and a saturation effect again noted. It was concluded that the titanium support disks and palladium port were acting to sorb hydrogen and prevent rapid removal of hydrogen.

The pump was mounted on the gyro housing and the housing was kept near room temperature. A test was made in which the pump body was heated in an oven to about 350°C. Electrical heat was also employed to raise the titanium to gettering temperature. When the heat was applied the chamber pressure actually decreased about half an order of magnitude in the 10^{-5} torr range. In fact, an increase in electrical power produced a small decrease in pressure. This was further evidence that non-uniform heating of the titanium parts of the pump was responsible for the high base pressure and slow pumping speed.

There was an intermediate value of heater current near three amperes, corresponding to about 15 watts input, that would cause the fastest pumping at the lowest pressure level. This indicated that there was an optimum temperature for maximum hydrogen permeation.

At this point in the program attention was turned to the design of a pump in which the titanium parts would operate at a more uniform temperature.

7:0 COAXIAL PARALLEL PUMP

Experience with the two cascade pumps pointed to the need for preventing a localized concentration of hydrogen somewhere in the getter material. This can be done only by operating the

entire titanium getter at a high and uniform temperature. Figure 11 shows a coaxial pump configuration which accomplishes this type of operation. A photograph of such a pump is shown in Figure 12.

7:1 STRUCTURE

As may be seen the palladium element is in the form of a tube positioned axially within a cylinder. The cylinder is closed at one end except for a small central hole. One end of the palladium tube is brazed in this hole and the other end is closed. Thus, the tube is made re-entrant within the cylinder. One surface of the palladium tube is exposed to the vacuum and the other surface is exposed to the outside atmosphere. The titanium bulk getter element is in the form of a sleeve pressed over the closed end of the palladium tube. Although working models have thus far had housings made of stainless steel they might well have been made with housings of ceramic.

A heating coil is inserted within the palladium tube on the air-side of the pump. The coil used in the tests was fabricated as a bifilar winding of 0.010-inch platinum wire near one end of a 0.040-inch alumina tube. The alumina tube contained two paraxial holes and the platinum leads from the coil were passed through these holes to the other end of the tube where electrical power could be applied. The coil was made rigid and insulated with a coating of high temperature Sauereisen No. P-78 cement. When in position, this heater concentrates heat at the closed end of the palladium tube. Other heater materials might be used such as Kanthal or another nickel-chromium alloy. The only requirement is that the heater be operable in air for long periods near 1000°C.

A measurement of the magnetic field strength due to heater current was made using a simple heater in the form of a hair-pin inserted in the palladium tube. At a distance of about 3/4-inch from the palladium tip, corresponding to the port glange position, the field strength was only 0.006-oersted.

It is evident that when the coil is heated, a temperature gradient will be established along the palladium tube and the temperature of the closed end will rise above that of the pump housing. Normally the closed end is operated at a temperature near 850-950°C. Since the dissociation pressure of oxygen in palladium in this temperature range is greater than the partial pressure of oxygen in air, oxygen will not react with the palladium and the metal will operate for long periods of time without harm. A molybdenum-foil heat shield is used within the pump housing to minimize radiation cooling of the palladium and titanium elements and to establish the greatest possible temperature difference along the palladium tube for a given tube length and power input. The bulk getter is located near the closed end of the tube and is therefore heated by conduction to a nearly uniform temperature, typically in the 800 to 1000°C range.

7:2 THERMAL GRADIENT

In any configuration of pump it is necessary to maintain a temperature difference between the bulk getter element and the pump housing. In this configuration the necessary temperature changes takes place along the hydrogen pumping element itself rather than along an additional type of support for the getter element. This approach is simple and advantageous. The palladium is not only a mechanical support for the titanium but serves as a pump for hydrogen as well. Hydrogen permeation to air through palladium will take place from nearly room temperature upward. While the speed of permeation increases with temperature, there is very little tendency for the process to stop, even at relatively low temperature, as there is in the case of titanium. In this design the entire inside surface of the palladium is separated from air by only the thickness of the palladium wall, nominally 0.010-inch.

7:3 THERMAL TRANSPIRATION

The port of the pump may be left fully open or may be partially closed by a thin stainless steel grid. If left open, the pumping speed is greatest, but heat will radiate to the vacuum environment. If partially closed, the radiation loss

is reduced, but with an accompanying loss in pumping speed. A back pressure due to dissimilar temperatures of the gas on either side of the screen can develop due to the effect of thermal transpiration.[9] This effect slows pumping speed and is most pronounced when a screen is used.

A thin steel disk with several small holes drilled through it has been found to provide some heat shielding without greatly reducing the pumping speed. Basically the transpiration effect is least when the temperature difference across the orifice is small.

In the case of a perforated steel disk, heat radiating from the pump element can be rapidly removed in a radial direction by conduction and dissipated externally. Thus, the disk will not acquire a high temperature and the gas temperatures on each side will be nearly the same. Therefore the passage of gas through the small holes will not be greatly inhibited by the effect of thermal transpiration. This feature is important in an application such as the electrostatic gyro where it is necessary to minimize heat radiation to gyro components within the vacuum housing without losing pumping capability. Of course the normal conductance for molecular flow must be taken into account in choosing the size of the holes in the plate.

It should be noted that the palladium not only removes hydrogen directly from the vacuum environment, but also tends to pump hydrogen which is within or has diffused through the titanium getter sleeve. Thus, both single and dual diffusion of hydrogen can take place.

7:4 SHAPE OF TITANIUM ELEMENT

Lastly, the shape of the titanium part can be made so as to present the greatest surface area to the incoming gas to increase pumping speed. For example, it may be grooved in a number of ways. Its outer diameter and length may also be selected to provide the maximum gettering action. As its surface area is increased, a greater amount of heat is radiated to the walls, and since the shielding is not perfect, a greater power input is needed to heat the pumping elements to operating temperature. In the model pumps the outer surface of the titanium sleeve was smooth.

7:5 PUMPING PERFORMANCE

A system was assembled for measuring the pumping speed of the coaxial P^2-pump as shown in Figure 13. It consisted of various one-inch diameter Varian type fittings, a Veeco RG 75 gage, a 5 liter per second VacIon pump, and the P^2-Pump. A one-inch Varian valve with a Teflon seat was used to close the VacIon pump from the rest of the system. When this valve was closed, the volume seen by the P^2-pump was about 2 liters. An identical valve also permitted closing the P^2-pump from the system. A Vacsorb pump was used to forepump the system from air to the 10^{-4} torr range.

The system was pumped at room temperature by the VacIon pump for about 12 hours with the I-gage on. At the end of this time the pressure had reached 3×10^{-6} torr. The valve to the VacIon pump was closed and the pressure began to rise, indicating a rather high amount of outgassing was taking place. The effect was not due to a leak to air, however. The system had been helium leak-checked and was argon leak-checked just prior to closing the valve.

While the constituent gases in the system were not known in detail, it was assumed that CO, H_2O, H_2 and some CO_2, O_2, N_2 and inert gases were present. The residual gas environment was typical of that existing in a chamber which had been pumped by an ion pump and it is unlikely that only hydrogen, for example, was present.

Figure 14 shows plots of pressure versus time for three cases when the pump was turned on and was pumping; and three cases when the pump was turned off and outgassing was occurring. The plots represent data taken in time sequence and in cyclic order; that is, first with the pump on, then off, etc. The crosses show the first pumpdown and outgassing values of pressure and time. The zeros show the second pumpdown and outgassing values and the dots the third set of such values. The heater voltage was set at 5 volts and the current at 2.5 amperes during each pumpdown.

Of course the response of the P^2-pump is not instantaneous and about 10 seconds is required for pumping to begin. A greater delay is involved when the pump power is removed, since the pumping elements cool slowly and the pumping action continues. Thus the measured rate of outgassing with time includes some residual pumping and is therefore lower than it would be if the

pump could be cooled instantaneously.

The P^2-pump was first turned on when the pressure was about 6.4×10^{-4} torr. The pressure dropped exponentially to the mid 10^{-5} torr range in about 20 seconds. A base pressure, including the outgassing, of 3.5×10^{-5} torr was reached in about one minute.

The following formula may be used to calculate an effective pumping speed, S, in the presence of outgassing.(10)

$$S = \frac{V}{t_{12}} \ln \frac{P_1 - P_o}{P_2 - P_o}$$

where V is the system volume, t_{12} is the time interval between two pressures, P_1 and P_2, along the pressure-time curve, and P_o is the base pressure reached after a long period of the pump.

Using the values,

$P_1 = 1 \times 10^{-3}$ torr

$P_2 = 6 \times 10^{-5}$ torr

$P_o = 3.5 \times 10^{-5}$ torr

$t_{12} = 20$ seconds

$V = 2$ liters

the pumping speed is about 0.37 liters per second.

In the pump tested the titanium sleeve was 0.250-inch O.D., and 0.500-inch long. The palladium tube had a 0.010-inch wall and was 0.146-inch O.D. and 1.625-inch long. The pump body was 0.750-inch O.D. and 2.0-inch long. The port opening was a 3/4-inch hole.

At the time of the test, this pump had been operated for 370 hours at temperature. It had been used on several system

setups in accumulating this life period and therefore had been subject to several exposures to air. It was run about 30 hours during the test just described.

Following this test, the valve to the Ion pump was again opened and the P^2 pump power removed. Ion pumping continued for about five days with the I-gage on. The system pressure reached 5×10^{-6} torr at the end of this period. When the ion pump valve was closed, residual outgassing began and the pressure increased in about three minutes to 3.6×10^{-5} torr when the P^2-pump was again turned on. Data representing a sequence of off-on runs taken at this time is shown in Figure 15. The curves show performance at a pressure about 10^{-5} torr, one order of magnitude lower than those of Figure 14.

The pressure did not rise rapidly when the pump was turned on, indicating that the titanium elements had not become highly saturated with hydrogen while cool. In fact, no change in pressure would occur for a few seconds as the element heated, and then the pressure would commence to decrease steadily toward the base pressure. Repeatedly, the effect of increased heater power was to lower the pressure.

An approximate pumping speed for the second test can be calculated using the values:

$P_1 = 4.5 \times 10^{-5}$ torr $\qquad P_o = 6 \times 10^{-6}$ torr

$P_2 = 1.6 \times {}^{-5}$ torr $\qquad t_{12} = 30$ seconds

$V = 2$ liters

and is equal to 0.09-liters/second, about one-fourth the speed calculated for the pump in the previous test, where the pressure was in the 10^{-4} torr range.

It might be implied from the results in Figure 15 that the pumping speed was pressure dependent. For example, the sticking parameter for gas atoms impinging on the hot titanium could have been lower at lower pressures. However, the reduction in

pumping speed may also have been due to a number of other factors. Thermal transpiration, getter poisoning during the long period between tests, or a change in the type of gas may have been involved.

Clearly, a test of the pumping speed for specific gases should be carried out to appraise the true pumping capability of the reactive metal pump. A speed in the 0.1 to 0.5 liter sec per second range is satisfactory for the degree of outgassing that is encountered in an electrostatic gyro chambers which has been cleanly assembled. In particular, it was not known to what extent inert gases were present in the vacuum chamber used in the laboratory tests. Therefore, their effect on both pumping speed and base pressure was not known.

8:0 APPLICATION OF COAXIAL PUMP TO THE ELECTROSTATIC GYRO

A number of coaxial pumps have been built and soldered to a standard Phase IV gyro chamber. Cryo forepumping and ion pumping were employed to produce pressures in the low 10^{-5} torr region in the chamber. A Type 5966 ion gage operating at 0.6-ma. emission was employed to indicate pressure. It was connected about five inches from the chamber with one bend through a ¼-inch I.D. stainless pipe. The conductance of this connection was only 0.23-liters per second. The pump was located directly atop the gyro chamber and therefore the pressure readings at the gage tended to be slightly high, due to outgassing resulting from the effect of the heated gage filament. Such small piping is used for pumping the chamber of all gyro chambers and of course makes measurement of the pressure within the chamber difficult with a gage connected in this manner. The coastdown time constant of the gyro rotor perhaps serves as the best indicator of pressure within the chamber.

8:1 ULTIMATE PRESSURES

When the ion pump was valved off from the system, the P^2-pump reduced the pressure at the gage as low as 4×10^{-8} torr. The chamber was empty in this case. At another time a Mycalex electrode housing was placed in the chamber and an ultimate pressure of 5×10^{-7} torr was achieved. Some heat radiation from the pump

element shone on the Mycalex through the small holes in the pump port and caused a slow release of gas, probably water vapor. This of course would account for the increase in ultimate pressure over that obtained with an empty chamber.

These pressures were attained after pumping for a few days. With the Mycalex as a pump load, the pressure was held in the 10^{-7} torr range for several days until the pump was removed. The input power to the heater used in the tests was 12-18 watts. Later the heater was redesigned to operate the pumping element properly with only 8-10 watts input. The pressures attained and held are adequate for satisfactory gyro performance.

8:2 GYRO HEATING

For some time there was concern about the effect of heat radiating to the rotor housing. Such heat will cause non-uniform expansion of the housing and consequently introduce errors in gyro readout. Efforts were made to minimize this heating by the use of a reflective heat shield. It was possible to reduce the local temperature on the housing directly under the pump port to about 45 C, with full pump power applied and the pump elements operating near 900 C. However, because this heat was localized and the temperature of the rest of the housing was nearer 30 C, it was felt that the resulting temperature gradient on the housing was still too large for satisfactory operation.

As a result of these tests, it was decided to locate the pump outside of and parallel to the chamber. This location will of course prevent heat from falling directly on the housing. Any heating effect will be more uniformly distributed around the chamber by conduction through the chamber wall. For many other applications of the pump, however, the total heat dissipated by the pump is relatively small and could be tolerated.

8:3 RECENT PERFORMANCE ON OPERATING GYROS

More recently the pump has been designed with a standard gyro flange having a 3/8-inch port for connecting the pump to the pipe bend extending beneath the gyro chamber. With such a connection the pumping speed is somewhat reduced, but radiated heat does not enter the chamber. Three pumps were furnished and were mounted on an electrostatic gyro chamber in this way. In one gyro test a pressure of 7×10^{-7} torr was determined by measuring the coastdown time-constant of the gyro rotor.

8:4 OPERATING LIFE OF SAMPLE PUMPS

During the course of the work it was observed that many of the sample coaxial pumps would develop very small leaks to air. Always these leaks appeared at the tip, the closed end, of the palladium tube. The region of the tip where the leak was found was not covered by or in contact with titanium.

In some pumps the trouble developed after only 50 hours of operation. In others the life was 160 to 250 hours, although in one pump, which was operated for 1000 hours, no leak developed at all.

A number of factors may have promoted this type of failure. First, there was concern about whether palladium can be operated in the 900-1000°C range for long periods of time. A number of individuals familiar with the behavior of palladium hydrogen leaks were consulted on this point and there is no known reason why leakage of this sort should occur due to some inherent property of palladium.

Secondly, the method used to close the palladium tube has been studied. The tip is spun over and the remaining tiny hole heliarc welded shut with a small piece of palladium rod as a filler. Examination of the tip surface suggests that a grain growth may have occurred with time. In any event, the closure process involves a thinning and working of the metal, which is not done to the rest of the tube surface, and this metal forming could easily lead to grain growth and leakage along grain boundries in the metal at the tip. The fact that the leaks occur only at the tips, leads one to suspect that if operation at high temperature is

possible, the chief cause of failure may be due to the way the metal tip is formed. The end of the tube could be closed by other methods which avoid the need for spinning the metal such as by heliarc welding a small palladium plug in the end of the tube.

Thirdly, it is possible that the effect of the phase change in palladium at about 180 C could lead to tip failure.[11] When the temperature of the palladium tube is cycled through this value of temperature over and again, grain growth can occur and cause cracking. The 75-25 palladium-silver alloy inhibits such grain growth and has been found to solve the problem satisfactorily in commercial hydrogen purifiers. It is interesting to note, however, that leaks developed in P⁻-pumps having tubes made of this alloy as well as pure palladium.

Chemical reaction of the palladium with the heater cement or with the alumina insulator at high temperature has been considered but no conclusions have been drawn about such reactions.

8:5 DOUBLY-SUPPORTED COAXIAL PUMP

Another approach is to eliminate the tip altogether by extending the palladium tube axially from one end to the other of a pump housing. Such a design is shown in Figure 16. The tube is supported at both ends in regions where the operating temperature is low. A bellows at one end is employed to allow for expansion of the palladium tube.

This method of support requires no metal forming or welding of the palladium and eliminates the need for closing over an end which must operate at high temperature. The external heater is inserted within the tube as before and made so as to supply the most heat in the middle of the tube. The titanium sleeve element is supported on the palladium tube along the central region of the tube. Small tungsten wires extending radially from the titanium sleeve to the heat shield may be used to give rigidity to the pump element against radially directed shock and vibration. The vacuum port extends from the side of the pump near one end and need not view the titanium element directly. Heat radiation through the port is thereby kept at a minimum.

Models of this improved design have been built and are undergoing life test. Starting at a pressure of 10^{-4} torr such a pump has maintained the pressure at 2×10^{-8} torr over a period of 260 hours. However, this design requires about twice the output power required by the pump of Figure 11 and, for this reason, is felt to be less desirable for use in an electrostatic gyro package.

9:0 GENERAL DISCUSSION AND SUMMARY

The basic principles of reactive metal and diffusion pumping have been applied in a number of ways during this program. A vacuum pump of novel design has evolved which is of general use as well as applicable specifically to the electrostatic gyro.

This pump provides an ultra-clean vacuum in the 10^{-7}-10^{-8} torr range starting at pressures in the range of several microns, in environments from which inert gases have been removed by other means. The pump will operate on a continuous basis. Pumping speed of the small model pumps is a few tenths of a liter per second. Only very small surges in pressure occur when pumping power is restored after having been removed. The pump actually removes rather than sorbs the most abundant gas, hydrogen, and prevents re-evolution of this gas in the system. Other gases are gettered irreversibly. The design is mechanically sturdy, light in weight, small, and inexpensive. No magnetic field or high voltage are employed. The design is thermally efficient and only a few watts of power are required to maintain the pumping elements at operating temperature.

The pump is uncritical of starting pressure and capable of numerous exposures to air. It is self-cleaning at operating temperature, and the elements do not have to be flushed. The entire structure is bakeable to a high temperature in air and is insensitive during operating to changes in ambient temperature. It is both radiation tolerant and silent.

Arrays of such pumps may be assembled to increase pumping speed. For example, they may be distributed along the walls

of a vacuum chamber to minimize pressure variations throughout the chamber. Any one pump may be made with as large a pumping element as desired.

Supporting the palladium tube at both ends eliminates the problem of failure at the tip. However, it is expected that the problem of closing the palladium tube at its inner end, which has been experienced in some experimental models, will be solved shortly and that the singly supported form of pump can also be made to have the long life and reliability required in the gyro application. To date a singly-supported pump model undergoing on-off temperature cycling periods has operated over 1000 hours.

As the development work proceeds, a facility for pumping inert gases will be included within the pump itself. Several methods are under consideration. However, the choice in the case of the electrostatic gyro must be among those which do not require a strong static or time-varying magnetic field. Also, a more careful measurement of the pumping speed for specific gases must be carried out, and it is also hoped that a means for obtaining an electrical indication of pressure can be provided.

10:0 ACKNOWLEDGEMENTS

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APPENDIX I

MEASURING PUMPING SPEED BY THE TWO-GAGE METHOD

The two-gage method is an alternative method for measuring the speed of a vacuum pump, and has recently become the preferred procedure in vacuum technology. A two-gage apparatus was not available during the early work on P^2 pumps. However, such an apparatus is being constructed and will be used in subsequent work. It will permit a measurement of pumping speed over a range of pressure levels for specific gases. The following development briefly describes the method and the governing equations.

Consider Figure 17 which shows two vacuum gages displaced along a pipe of molecular-flow conductance C_{12} liters per second. This conductance can be calculated from the formula for circular pipe as

$$C = \pi/3\sqrt{\frac{KT}{2\pi m}}\ D^3/L \text{ liters/sec.} \quad \text{(c.g.s. units)} \qquad (1)$$

where T is the gas temperature, m is the molecular mass of the test gas, k is Boltmann's constant, L is the pipe length and D is the pipe diameter. The gages read the pressure P_1 and P_2 at each end of the pipe. The test gas is admitted near gage #1 through a capillary leak from a supply of test gas. Between the second gage and the pump to be tested there is a connecting pipe of conductance C_{2p}.

The pumping speed at the pump port is

$$S_p = \frac{Q}{P_p} \qquad (2)$$

where Q is the mass flow caused by pumping action in micron-liters per second. The effective pumping speed at gage #2 is

$$S_2 = \frac{Q}{P_2} \qquad (3)$$

The conductance C_{2p} is related to the drop in pressure for mass flow Q as,

$$\frac{1}{C_{2p}} = \frac{P_2 - P_p}{Q} \tag{4}$$

From Equations 2, 3, 4 the pressures can be eliminated such that,

$$\frac{1}{S_p} = \frac{1}{S_2} - \frac{1}{C_{2p}} \quad \text{or}$$

$$S_p = \frac{S_2}{1 - S2/C_{2p}} \tag{5}.$$

The effective pumping speed at gage #2 can be expressed in terms of the drop in pressure between the gages and the conductance C_{12}. Now

$$C_{12} = \frac{Q}{P_1 - P_2} \tag{6}$$

and from 3) $Q = S_2 P_2$ so that

$$S_2 = C_{12} (P_1/P_2 - 1) \tag{7}$$

Substituting 7) in 5), the true pump speed is related to the pressure readings and the calculated conductance s as

$$S_p = \frac{C_{12} (P_1/P_2 - 1)}{1 - \frac{C_{12}}{C_{2p}} (P_1/P_2 - 1)} \tag{8}$$

A two-gage test system must be designed with some knowledge of the approximate speed of the pump so that a sufficiently large pressure difference is established to permit calculating the term $(P_1/P_2 - 1)$ to a reasonable accuracy. Thus a long pipe might be used to measure a slow pumping speed.

12:0 REFERENCES

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(2) Kohl, W.H. Material and Techniques for Electron Tubes Reinhold, N.Y. 1960, pp. 578-581

(3) Stout, V.L., Gibbons, M.D. "Gettering of Gas by Titanium", General Electric Research Laboratory Rpt. No. 55-RL-1336, July 1955

(4) Young, J.R. "A Palladium Diaphragm Hydrogen Pump", General Electric Research Laboratory Rpt. No. 62-RL-3041E, June 1962

(5) Peters, P.H., Patent Docket 35-19D-884S, Reactive Metal Diffusion Pump

(6) Beggs, J.E., General Electric Research Laboratory, Conversation

(7) Dushman, S., Lafferty, J.M., Scientific Foundations of Vacuum Technique, Sec. Ed., John Wiley, N.Y., 1962, p. 538

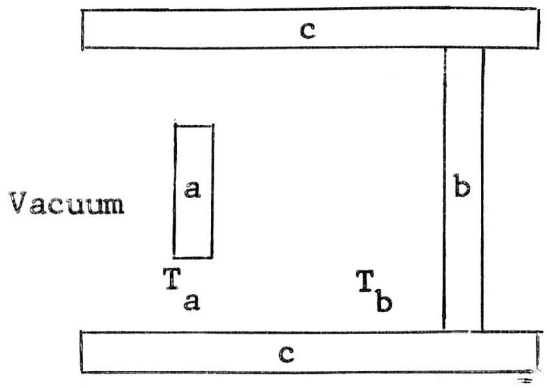
(8) ibid 7, pp. 500, 587

(9) Guthrie, A., Wakerling, R.K. Vacuum Equipment and Techniques McGraw-Hill, N.Y., 1949, pp. 43-45

(10) ibid 9, pp. 49-51

(11) ibid 8, pp. 539-544

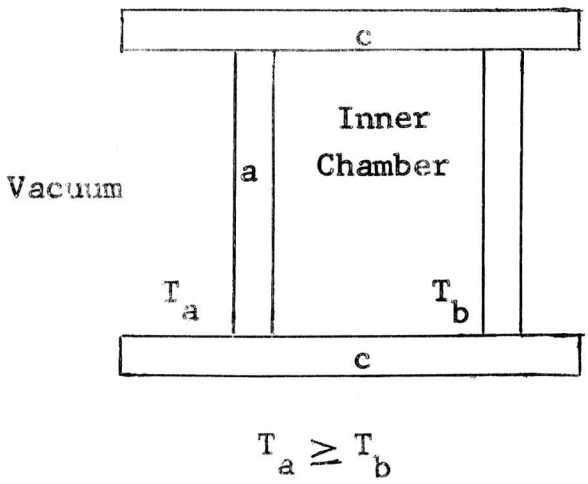
13:0 FIGURES PARALLEL FORM



a - Titanium
b - Palladium
c - Embodiment
T - Temperature

Figure 1a

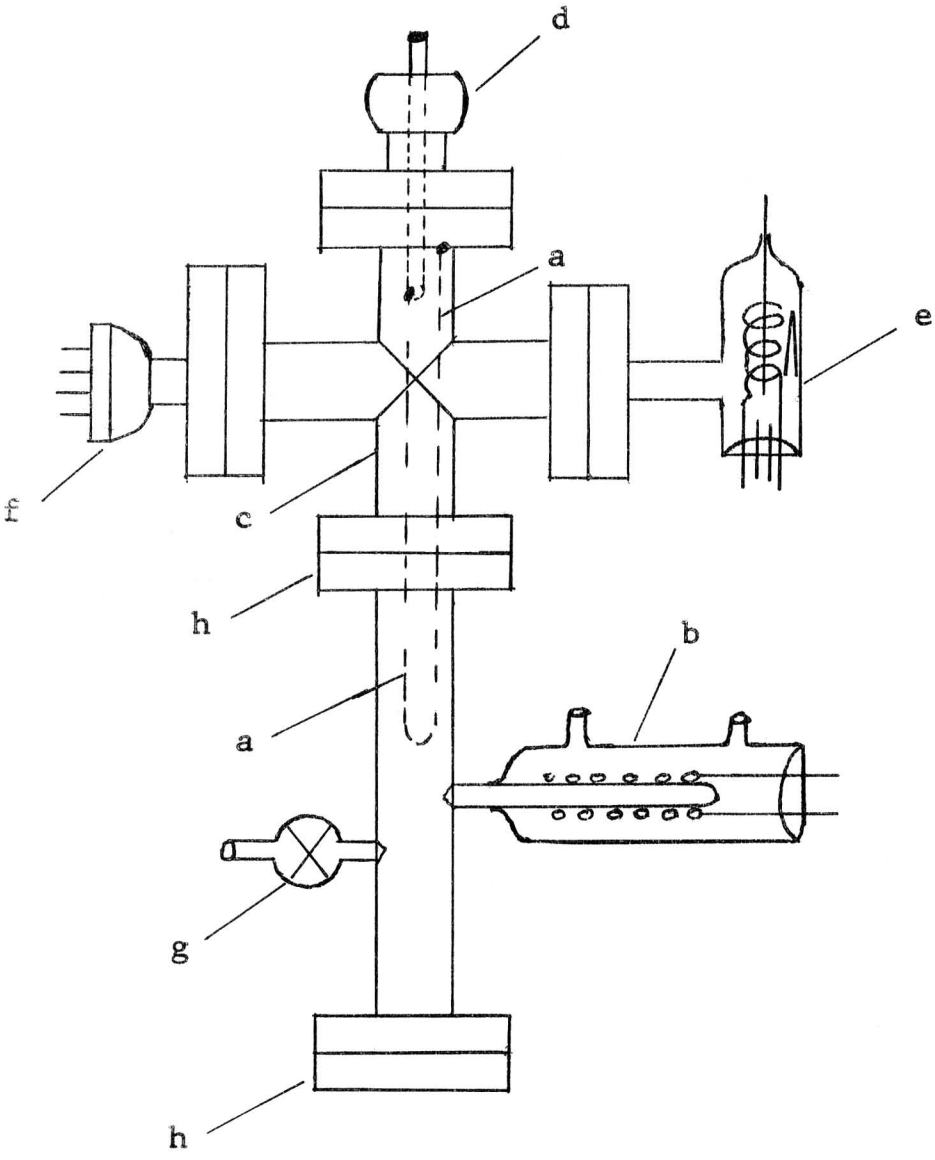
CASCADE FORM



a - Titanium
b - Palladium
c - Embodiment
T - Temperature

Figure 1b

BASIC PUMP TEST



a - Titanium Ribbon

b - Palladium Leak

c - 1" Dia. Cross Fitting

d - Feedthru

e - Ion Gage

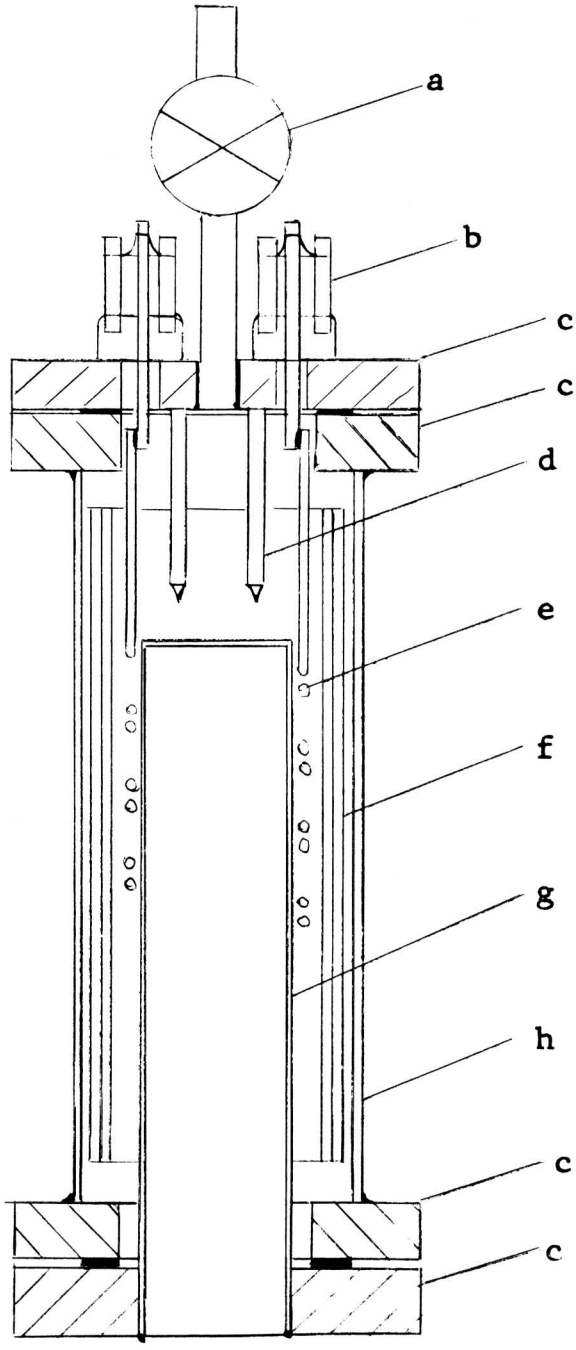
f - Thermocouple Gage

g - Hoke Valve - 413

h - 1" Dia. SS Flanges

Figure 2

DEMOUNTABLE CASCADE PUMP



a - Hoke Valve - 413

b - Electrical Feedthrus - 2 Req.

c - Flanges

d - Palladium Tubes - 4 Req.

e - Heater Coil - Bifilar Wound

f - Heat Shields

g - Titanium Tube

h - Outer Cylinder

Figure 3

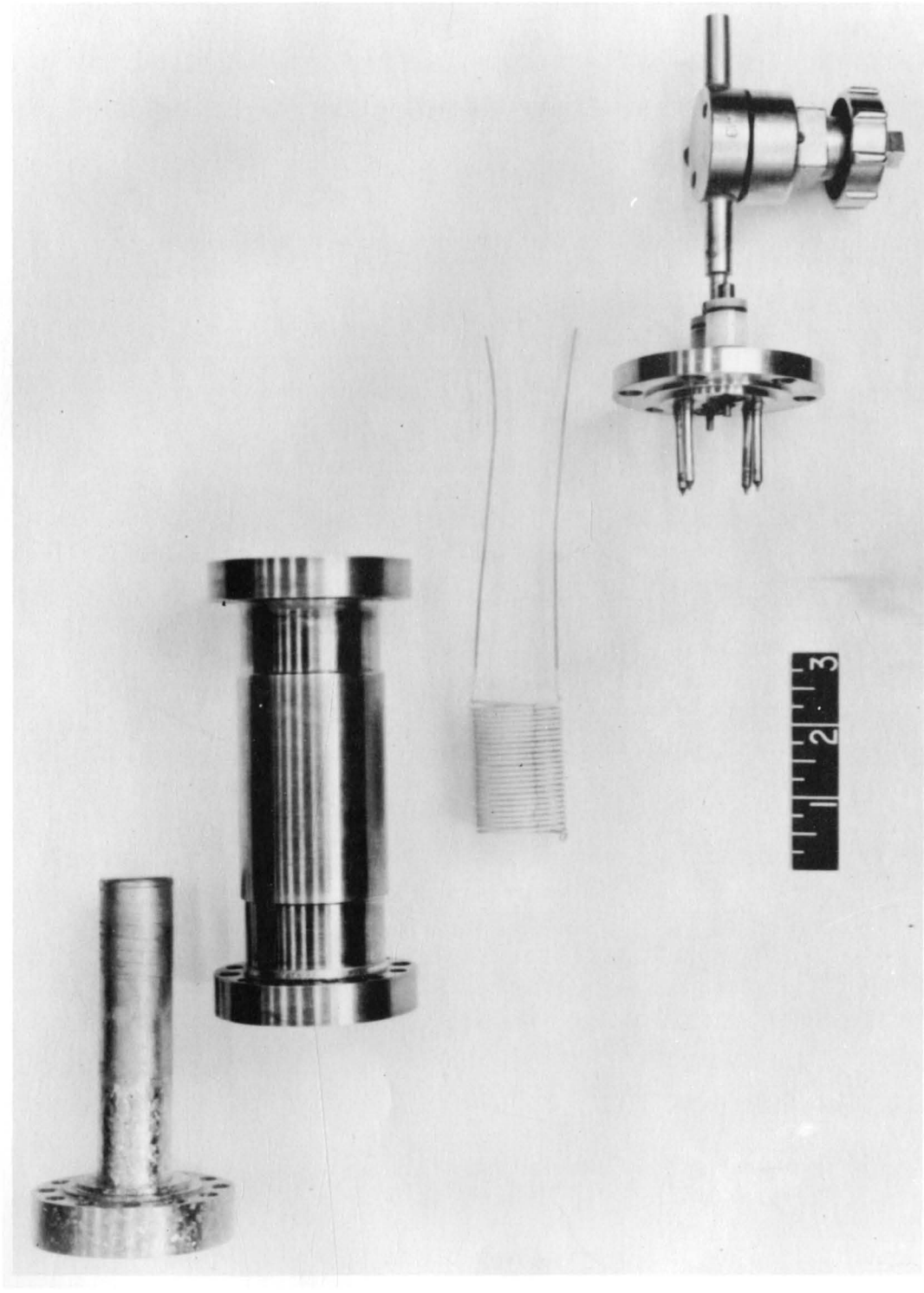


Figure 4

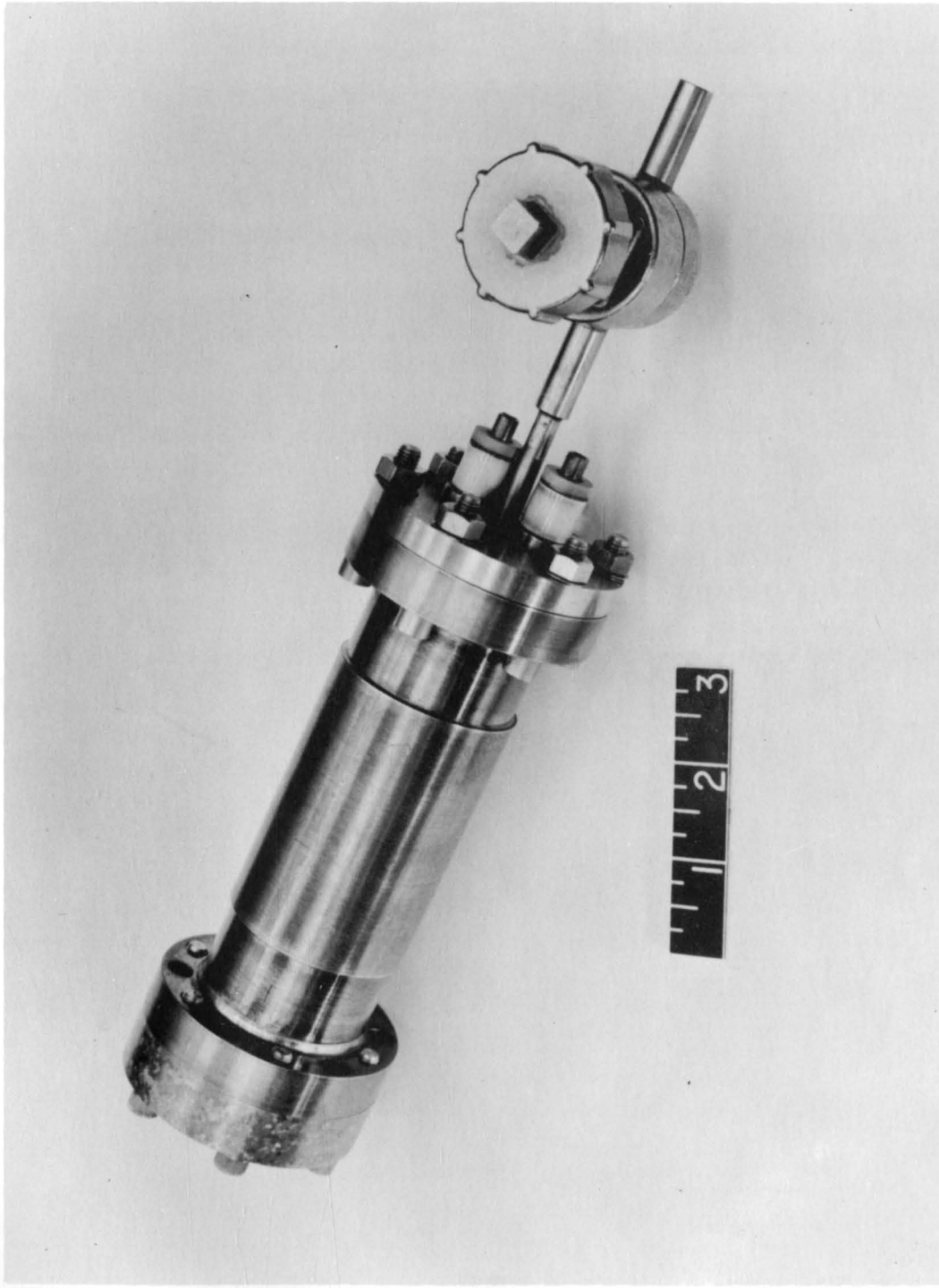
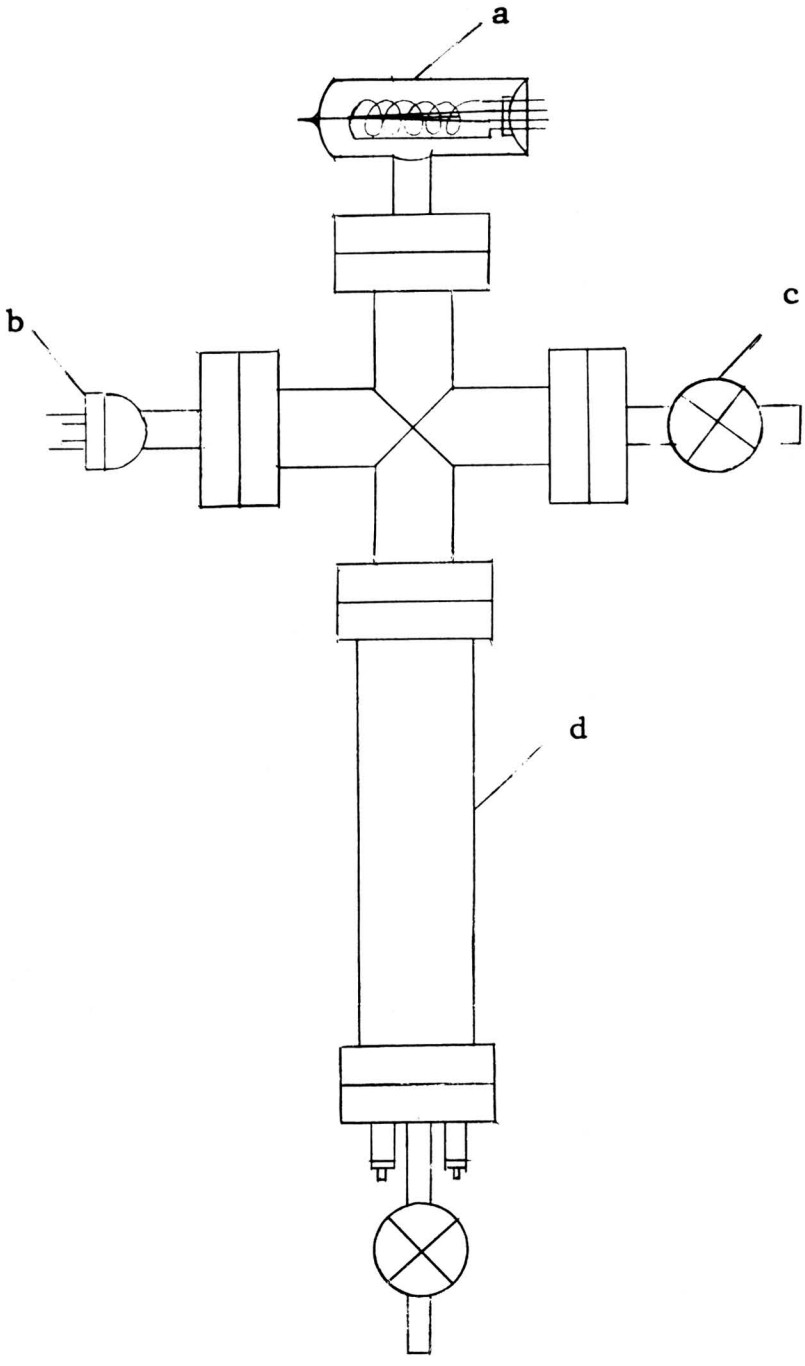


Figure 5



a - Ion Gage

b - Thermocouple Gage

c - Hoke Valve #413

d - P^2 Pump Assembly

Figure 6

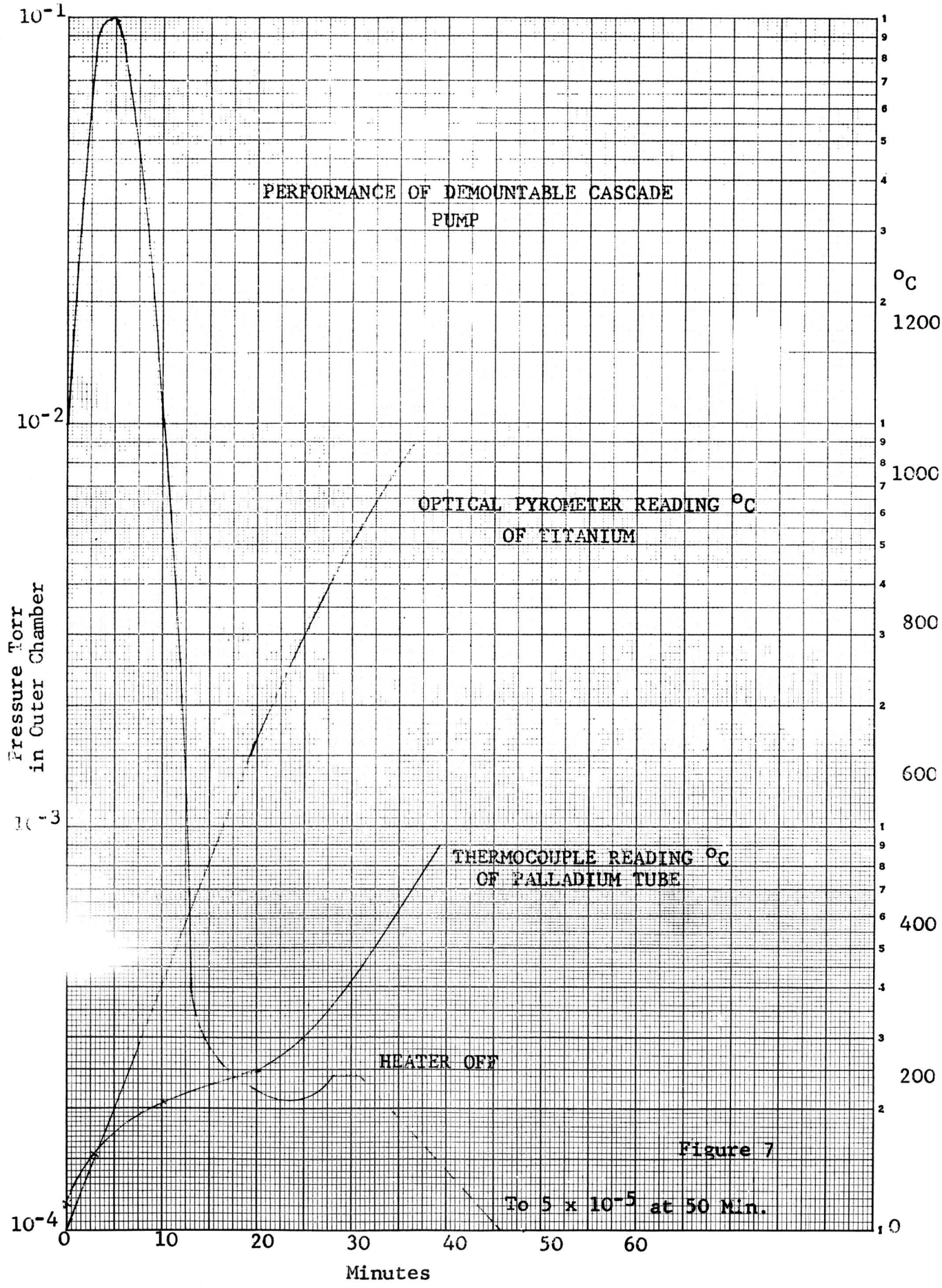
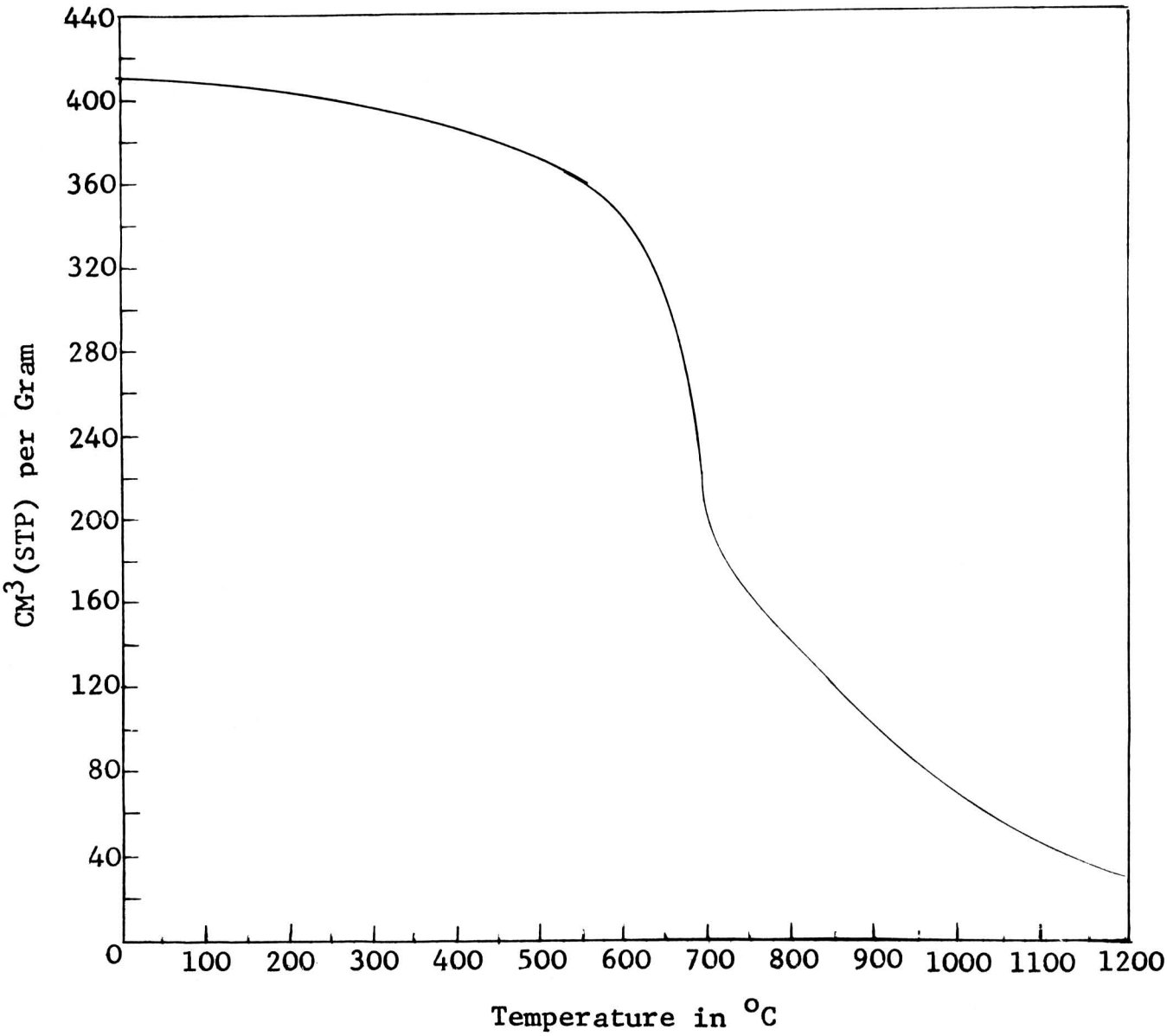

PERFORMANCE OF DEMOUNTABLE CASCADE PUMP
OPTICAL PYROMETER READING °C OF TITANIUM
THERMOCOUPLE READING °C OF PALLADIUM TUBE
HEATER OFF
To 5 x 10-5 at 50 Min.
Pressure Torr in Outer Chamber
10-1
10-2
10-3
10-4
°C
1200
1000
800
600
400
200
0
10
20
30
40
50
60
Minutes

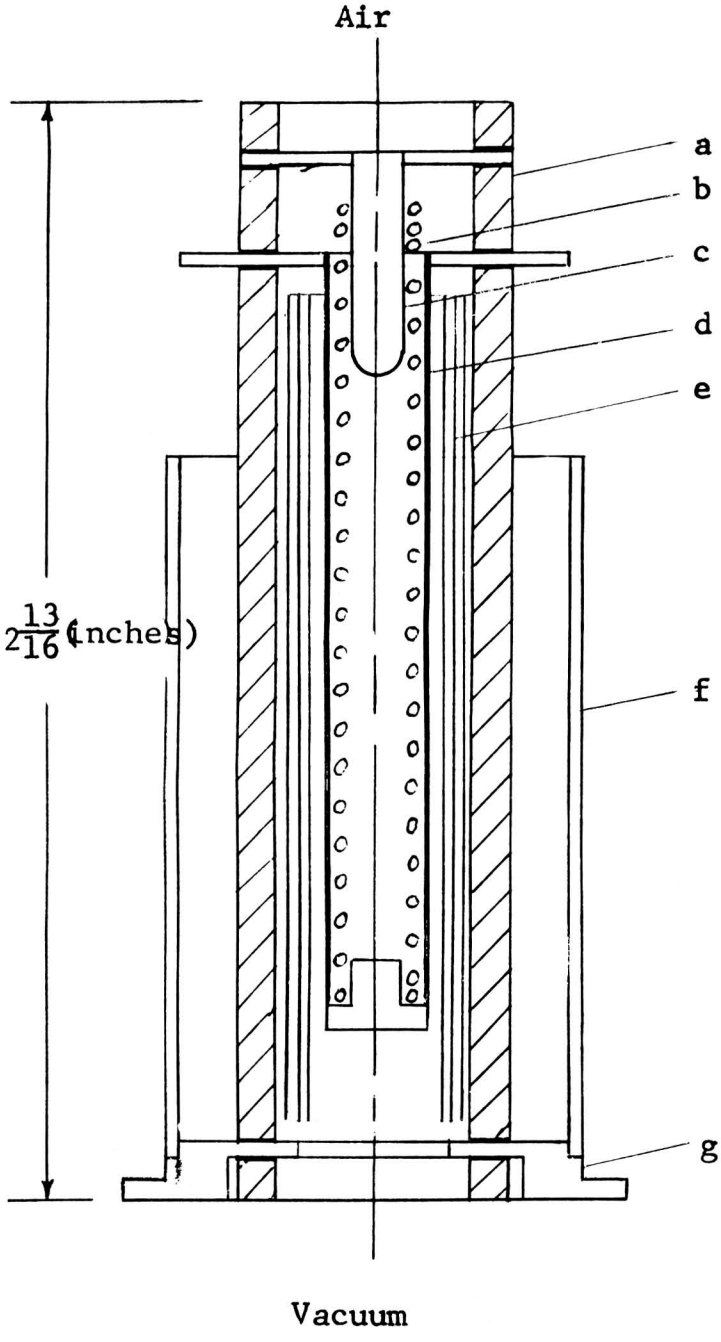
Figure 7



SOLUBILITY OF HYDROGEN AT 1 ATM (CM^3, STP, PER GRAM)
OF TITANIUM AT VARIOUS TEMPERATURES

Figure 8

METAL CERAMIC CASCADE PUMP



a - Ceramic Body

b - Heater Coil

c - Palladium

d - Titanium

e - Heat Shield - Inner

f - Heat Shield - Outer

g - Connection Flange

Figure 9

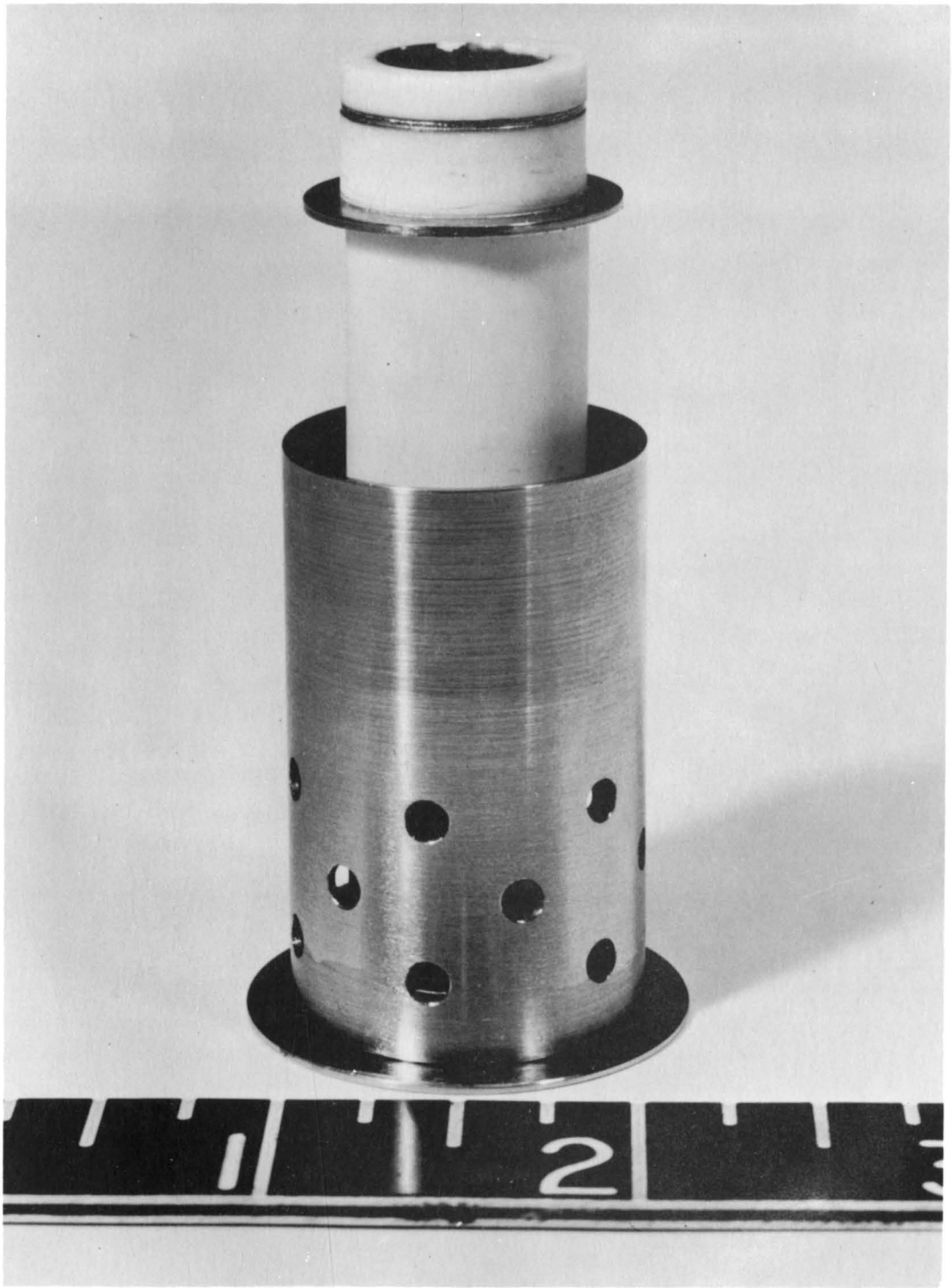
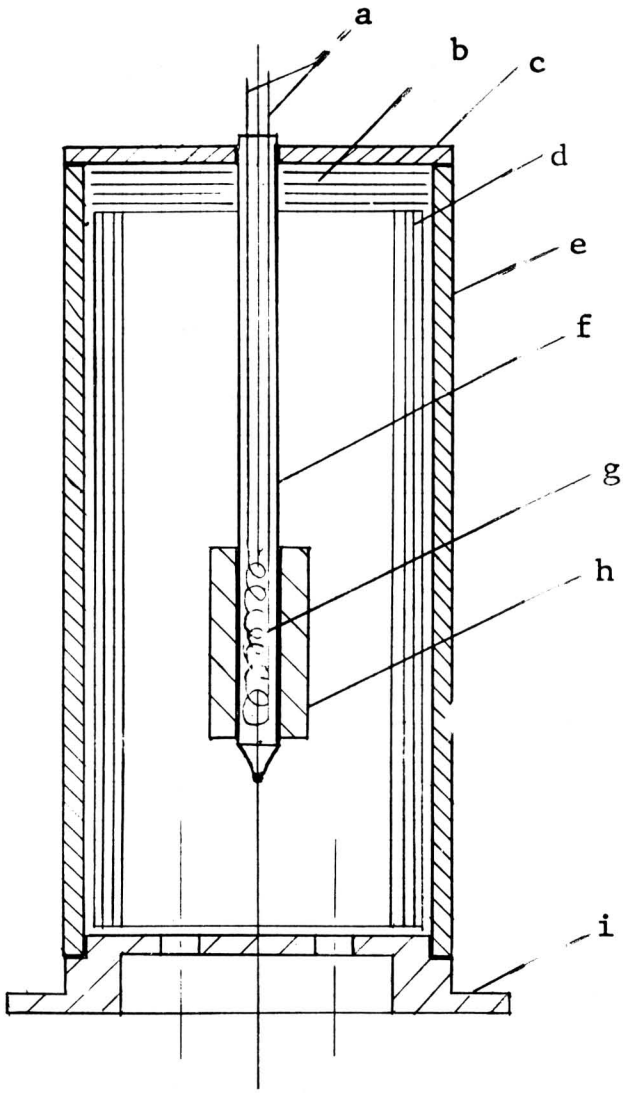


Figure 10

COAXIAL PARALLEL PUMP



a - Heater Leads

b - Heat Shield

c - End Cap

d - Heat Shield

e - Housing

f - Palladium Tube

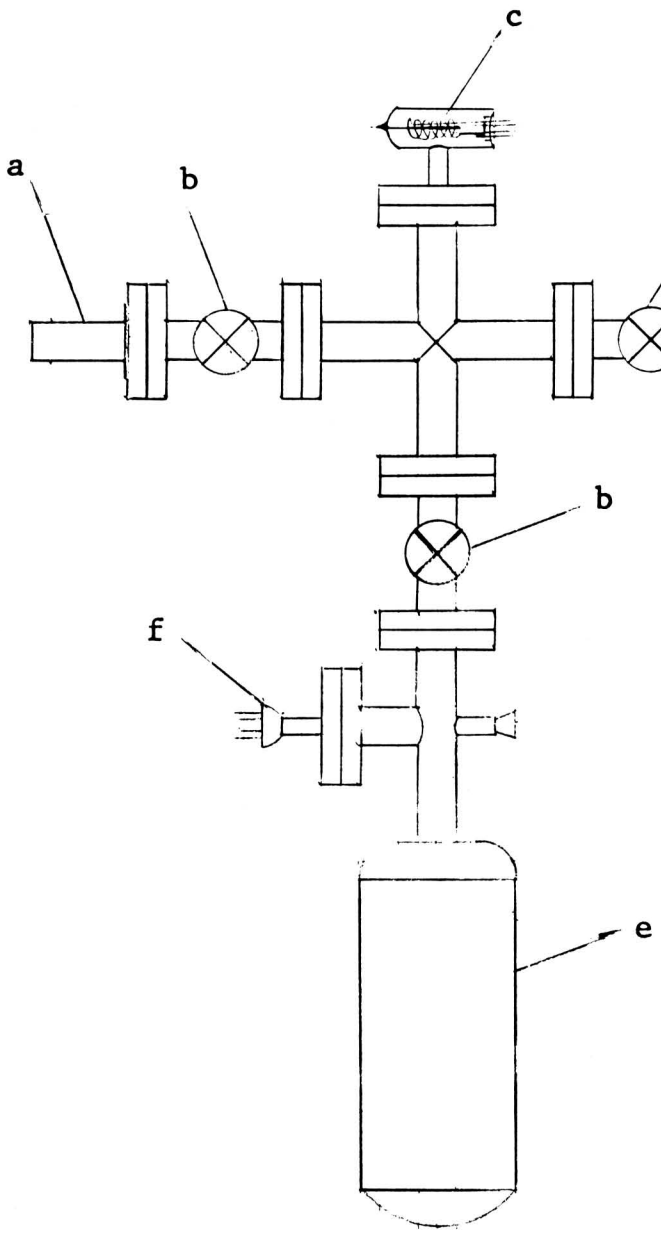
g - Heater

h - Titanium Slug

i - Flange

Figure 11

Figure 12



a - Coaxial P^2 Pump

b - Valve

c - Ion Gage

d - VacIon Pump

e - VacSorbe Pump

f - Thermocouple

Figure 13

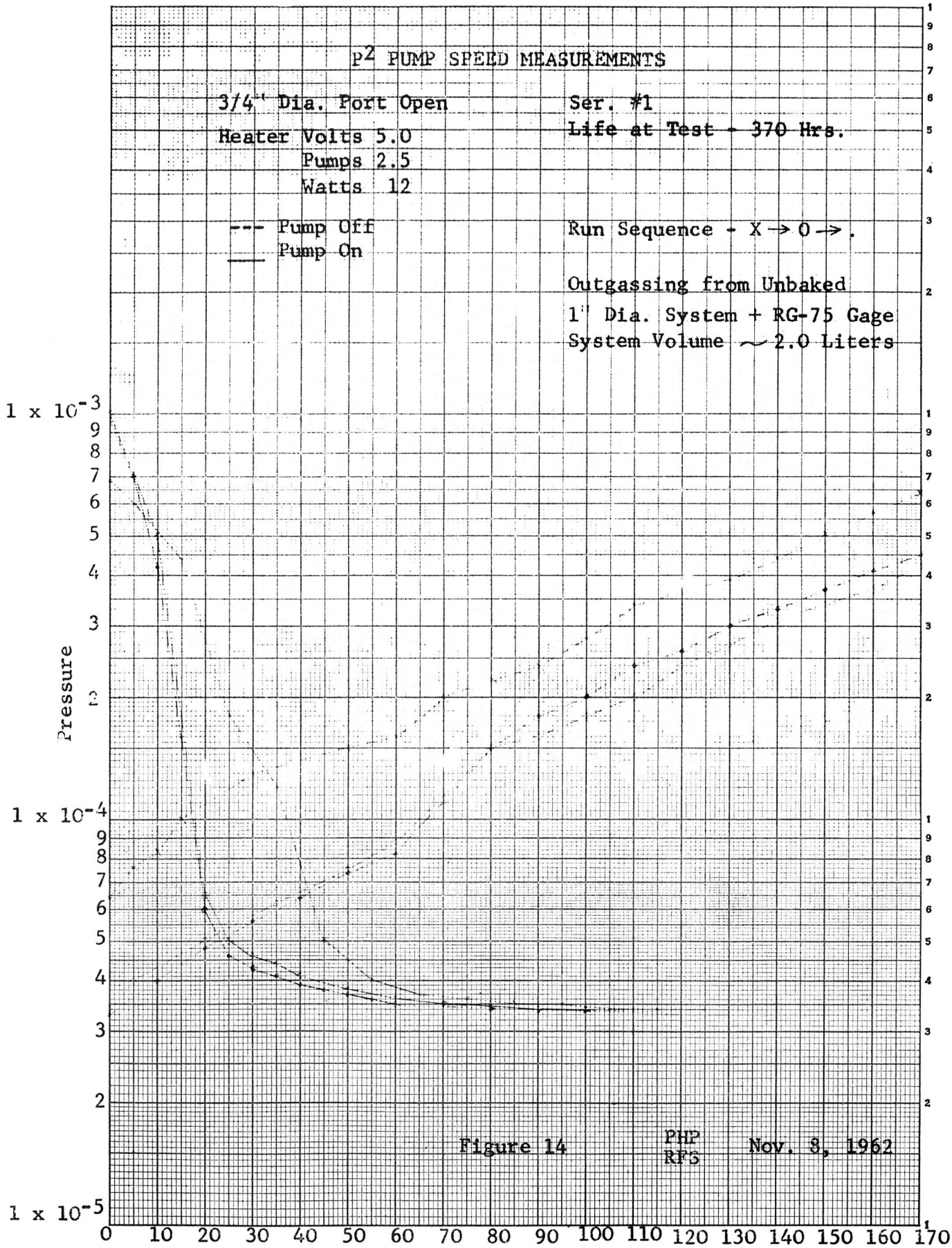


Figure 14 PHP RFS Nov. 8, 1962

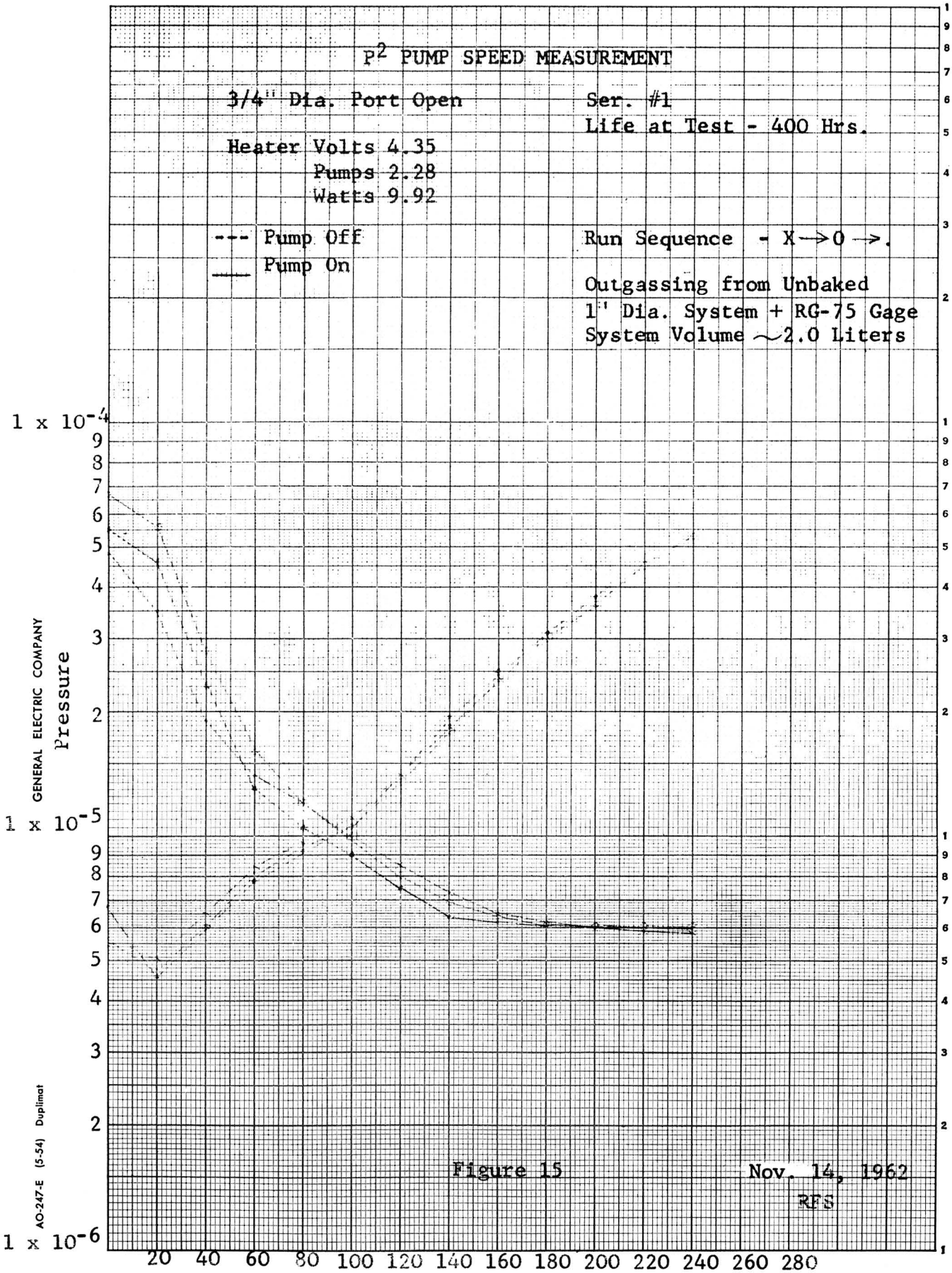
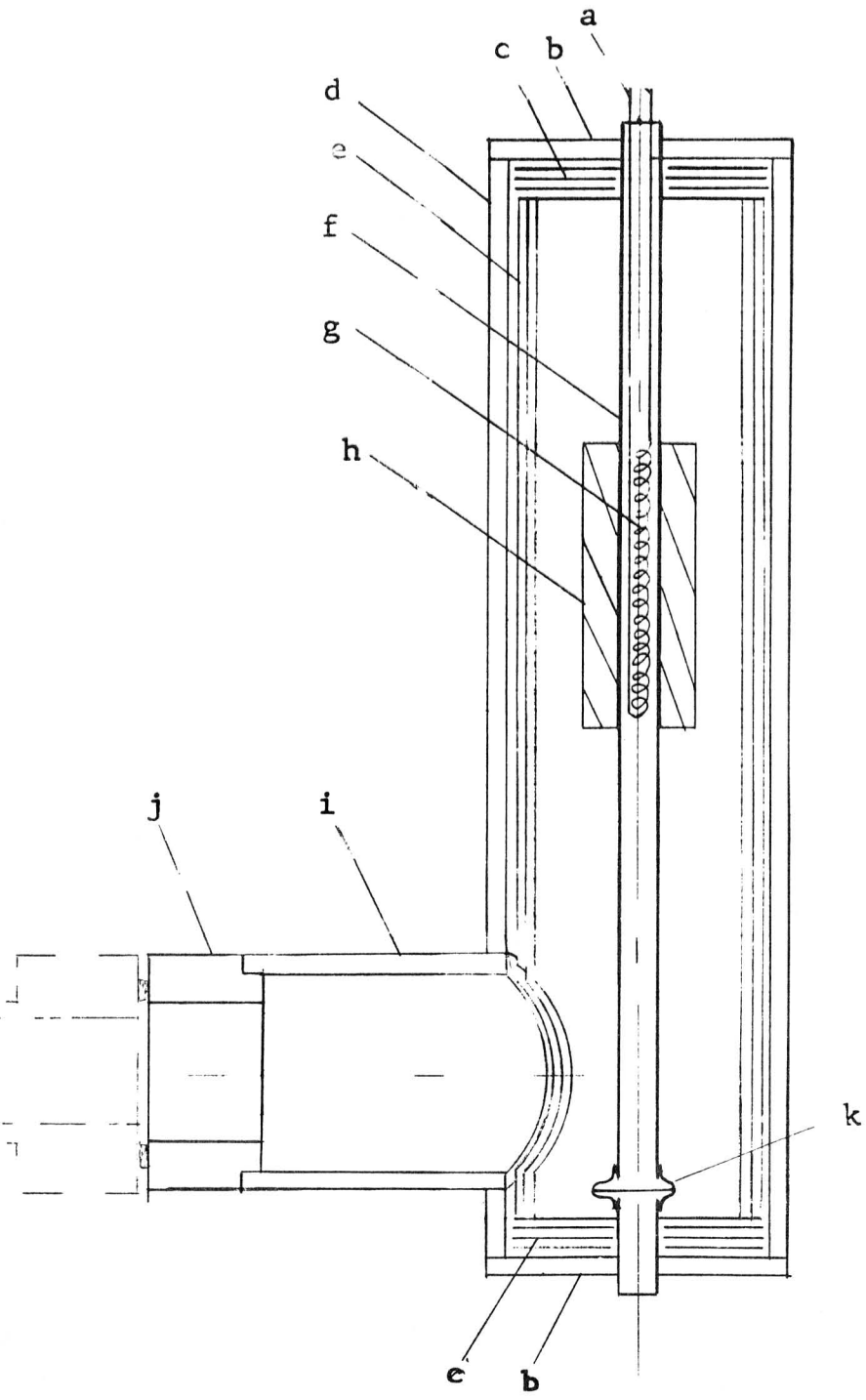


Figure 15

Nov. 14, 1962
RFS



a - Heater Leads

b - End Cap

c - Heat Shield

d - Housing

e - Heat Shield

f - Palladium Tube

g - Heater

h - Titanium

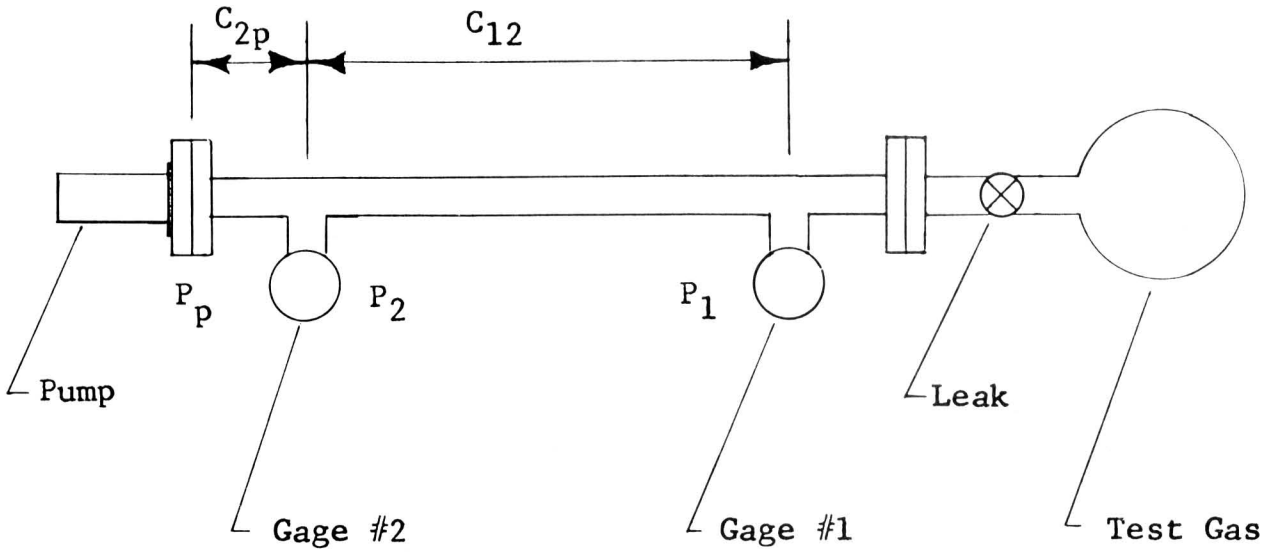
i - Transition Piece

j - Flange

k - Expansion Bellows

Figure 16

TWO GAGE SYSTEM



P_p - Pressure at Pump

P_1 - Pressure at Gage #1

P_2 - Pressure at Gage #2

C_{12} - Conductance of Tubulation Between Points Indicated

C_{2p} - Conductance of Tubulation Between Points Indicated

Figure 17

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GENERAL ELECTRIC