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**DEVELOPMENT AND EVALUATION
OF A CRYODEPOSIT SORPTION PUMP
CAPABLE OF PUMPING HELIUM**

Ronald Dawbarn and J. D. Haygood

ARO, Inc.

September 1968

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FOREWORD

The research presented herein was sponsored by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 6540223F, Project 8951, Task 895104.

The results of research reported herein were obtained by ARO, Inc. (a subsidiary of Sverdrup & Parcel and Associates, Inc.), contract operator of AEDC, AFSC, under Contract F40600-69-C-0001. The research was conducted in the Aerospace Environmental Facility (AEF) under ARO Project No. SW2705, from April to June 1967, and the manuscript was submitted for publication on March 26, 1968.

This technical report has been reviewed and is approved.

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ABSTRACT

A small cryogenically cooled pump has been developed which is capable of pumping all gases including helium and hydrogen. Its low base pressure makes it suitable for use in ultrahigh vacuum systems, and its high pumping speed for all gases is maintained up to pressures in the 10^{-6} torr range. The pump operates by sorbing the normally noncondensable gases in a layer of argon cryofrost which is predeposited on its liquid-helium-cooled surface. As with any sorption pump, the sorbent eventually becomes saturated, and the sorbate partial pressure starts to rise. However, unlike most sorbent pumps, this one can be reactivated simply by recoating the surface with a fresh layer of sorbent in situ. Sorption isotherms have been obtained for He on 4.2°K cryodeposits of N₂, Ar, O₂, and CO₂ over a pressure range from 10^{-8} to 10^{-3} torr.

CONTENTS

	<u>Page</u>
ABSTRACT	iii
I. INTRODUCTION	1
II. APPARATUS	
2.1 Vacuum Chamber	1
2.2 Test Pump	2
2.3 Gas Addition Systems	2
2.4 Instrumentation	3
III. CALIBRATION	
3.1 Leaks	3
3.2 Gage Calibration	3
IV. EXPERIMENTAL PROCEDURES	4
V. RESULTS AND DISCUSSION	
5.1 LN ₂ Radiation Shroud	5
5.2 Activating the Pump	6
5.3 Sorption Isotherms	6
5.4 Pumping Speed Measurements	7
5.5 Stability of Sorbed He	7
5.6 Rejuvenation of the Pump	8
5.7 Effects of Contamination	10
5.8 Use of CO ₂ as a Substrate	11
5.9 Thermal Loads on Pump	12
VI. CONCLUSIONS	13
REFERENCES	13

APPENDIXES

I. ILLUSTRATIONS

Figure

1. 2- by 3-ft Research Ultrahigh Vacuum Chamber	17
2. Cryosorption Pump	18
3. LHe Transfer Line	19
4. Disconnect in LHe Transfer Line	20
5. Vacuum Chamber and LN ₂ System	21
6. Sorption Isotherms He/A	22
7. Pumping Speed versus Addition Rate	23

<u>Figure</u>	<u>Page</u>
8. Sorption Isotherms He/A at 4.2°K, 2.3°K, and 2.18°K	24
9. Sorption Isotherms for Multilayer Deposits of He/A . . .	25
10. Sorption Isotherms for Multilayer Deposits of He/A . . .	26
11. Sorption Isotherms for He on CO ₂ , A, O ₂ , and N ₂ . . .	27
12. Measured Heat Loads on Pump as a Function of Chamber Pressure (He)	28

II. TABLE

I. Pumping Speeds of Various Gases	29
--	----

III. DETAILS OF COMPONENTS IN LN ₂ SYSTEM	30
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SECTION I INTRODUCTION

Oil diffusion pumps have long been the work horses in producing and maintaining vacuum environments. However, the ever increasing requirements for ultraclean vacuum systems have prompted a search for alternative pumping methods. Ion pumps, sublimation pumps, and sorption pumps each have some advantages as well as some limitations. In many cases a limitation is a low or negligible pumping speed for helium (He). The one exception is the sorption pump using molecular sieve material cooled by liquid helium (LHe). Present models, however, are expensive, cumbersome and require elaborate bakeout procedures to activate the molecular sieve. Since the sieve material is sensitive to contamination by other gases, extensive baffling by 20 and 77°K louvers is required to condense all gases except hydrogen (H₂) and He. These baffles present a limiting conductance which restricts the pumping speed of this type of pump. Also, there is a practical limitation on the quantity of sieve material that can be coated on the pump and then cooled to LHe temperatures.

The work reported herein resulted from an attempt to use the advantages of a sorption pump and at the same time overcome some of the limitations of available models.

Work at AEDC had previously indicated that the capture coefficient of He on argon (A) cryodeposits at 4.2°K was sufficiently high to be developed into a practical pumping system (Ref. 1). Moody (Ref. 2), in measuring capture coefficients of carbon dioxide (CO₂) on 77°K surfaces, had indicated the advantages of a concave hemispherical pumping surface. This information suggested a suitable configuration from which a prototype pump was built and evaluated.

SECTION II APPARATUS

2.1 VACUUM CHAMBER

This work was performed in the 2- by 3-ft Research Ultrahigh Vacuum Chamber described in detail in Ref. 1 and shown in Fig. 1, Appendix I. After deducting the space taken by the test pump and the liquid nitrogen (LN₂) shrouds, it has a free volume of 300 liters. The LN₂ shrouds are vented to allow free passage of gas molecules, yet

they are optically tight between the outer chamber walls and the test pump. The chamber is pumped by a 6-in. diffusion pump with an LN₂ cold trap which may be valved off with a 6-in. gate valve.

2.2 TEST PUMP

The test pump (Fig. 2) is an 11-in. -diam by 11-in. -high stainless steel cylinder. The pumping end consists of a 10-in. -diam concave hemisphere. The pump is mounted to the low heat leak LHe fill tube and is surrounded by a polished aluminum radiation shield. This shield also serves to limit pumping to the hemispherical surface. The pump will hold 13 liters of LHe.

The LHe used in these experiments was produced by a small cryostat. Since the cryostat could only produce batches of 15 liters which then had to be transferred to the experimental pump, transfer line losses had to be reduced to a minimum. Also, since the cryostat was used to support other experimental programs in the laboratory, a transfer line with a simple disconnect was desirable.

To reduce transfer losses it was decided that a superinsulated quad-concentric line could be built. The line is shown in Fig. 3. A foot valve in the cryostat was used to control the flow of LHe into the center line. This line is wrapped in aluminized Mylar[®] and surrounded by a vacuum jacket. The third tube returns the venting boiloff gas and thus acts as a radiation shield. An outer tube is used to vacuum jacket the boiloff gas and thus reduce the heat leak from room-temperature surroundings.

The tubulation entering the pump consists of a central tube which supplies the LHe. This tube is surrounded by a boiloff tube which is in turn vacuum jacketed. The transition from quad- to tri-concentric tubing is made in the shear seal disconnect. The disconnect is shown in detail in Fig. 4. The disconnect was used repeatedly during the period of these experiments with no evidence of leaks.

2.3 GAS ADDITION SYSTEMS

Associated with the chamber are two gas addition systems. Different test gases may be metered into the chamber either separately or simultaneously, at various rates. These systems use a set of sintered stainless steel leaks which have been calibrated in situ. Details of these systems may be found in Ref. 3.

One of the gas addition systems is connected to a 0.5-in. -diam tube which penetrates the shroud and ends facing the center of the pump. Its opening is 5 in. below the plane of the hemisphere. The other gas addition system enters the chamber and ends outside the shroud. Gas added from this system thus has to diffuse through the open shroud before reaching the pump surface.

2.4 INSTRUMENTATION

Pressures in the vacuum chamber were monitored by a mass spectrometer connected to a tube which penetrated the shroud and thus sampled a gas flux similar to that "seen" by the pump surface. An ion gage detected pressures on the outside of the shroud.

Before the mass spectrometer was mounted, an ion gage was installed in its place. Several tests were made with the two ion gages on the chamber. The sensitivities of this pair of ion gages were adjusted until the gages indicated identical pressure readings as steady chamber pressures were maintained over the 10^{-4} to 10^{-8} torr range. Several rates of pressure rise were then recorded simultaneously by both gages. Regardless of which gas addition system was used, both gages traced similar pressure histories. The results of these tests supported the conviction that the conductance through the open shroud was sufficiently large not to interfere with the distribution of gases during calibration and pumping runs.

SECTION III CALIBRATION

3.1 LEAKS

Each leak in the gas addition system was calibrated for the gases used from the rate of pressure drop in a known volume as the gas flowed from this volume into the vacuum chamber. Full details of this procedure and analysis of the method are included in Ref. 3.

3.2 GAGE CALIBRATION

Gages were calibrated before each experimental run. Since all the shrouds were at operating temperatures, no thermal transpiration correction terms were required. The method used was to compare the

measured rate of rise of pressure in the vacuum chamber with a known rate of gas being admitted via the calibrated leak system and no pumps operating. This method, outlined in detail in Ref. 3, is known to be quite reliable for this chamber which has a low base pressure (10^{-9} torr) and a low outgassing rate (5×10^{-11} torr-liters/sec).

SECTION IV EXPERIMENTAL PROCEDURES

Before a series of pumping tests was performed, the vacuum system was baked at 300°C for 24 hr. During the bakeout a small flow (~ 0.1 torr-liters/sec) of A was bled into the chamber and pumped via the diffusion pump. It was found that the sweeping action of the inert gas helped to reduce the bakeout time required. After the heaters were shut off, the A flow was stopped and LN_2 supplied to the shrouds. The chamber pressure dropped to the low 10^{-9} torr range each time. Before LHe was transferred to the pump, the gages were calibrated.

After calibration the chamber was pumped to its base pressure and LHe was transferred to the sorption pump. Then the diffusion pump system was valved off and the pressure in the chamber monitored as it dropped to mid 10^{-10} torr. The boiloff gas from the LHe was routed through a wet test meter. Periodically during the test sequence, the rate of LHe usage was recorded. An aneroid-type pressure gage was used to monitor the vapor pressure of the LHe in the pump. The pumping speed measurements were made by introducing the test gas at a known rate and recording the steady-state pressure in the chamber during the addition. Several gas addition rates were used in order to evaluate the pump's capability at various pressure levels.

Isotherms were obtained by first depositing a known amount of the gas to be used as the sorbent on the pump surface. The helium was then added at known rates for measured intervals. During each addition the helium pumping speed was measured and, after each addition, the equilibrium partial pressure was recorded.

To conduct tests at temperatures below 4.2°K the pump was first filled with LHe. Then the helium compressors, normally used to supply high pressure He for the cryostat, were used in reverse to reduce the pressure over the LHe. By valving the gaseous He boiloff flow stable temperatures, in the 2.1 to 2.8°K range, were obtained.

SECTION V RESULTS AND DISCUSSION

5.1 LN₂ RADIATION SHROUD

Liquid-nitrogen-cooled shrouds are an integral part of this chamber, not only to reduce the radiation load on the LHe-cooled pump but to assist in cryopumping gases condensable at 77°K, namely CO₂ and water (H₂O). Also, an LN₂-cooled baffle is installed between the vacuum chamber and pumping system to prevent oil vapors from backstreaming into the chamber. Besides trapping oil molecules from the pumps, the cold trap also supplements the pumping system, by cryopumping condensable gases from the chamber. Regardless of their location or prime purpose, LN₂-cooled panels soon accumulate many layers of condensed gas or vapor molecules.

These condensed layers offer a secondary pumping capacity by the process of sorption. Gas molecules not normally cryopumped by 77°K, now impinging upon the coated cryopanel, may become trapped at sorption sites either on the surface or within the existing cryodeposit matrix. Fite et al. (Ref. 4), reported pumping O₂ and N₂ by H₂O frosts cooled to 77°K.

In most cases this secondary pumping by cryodeposits is so small as to be insignificant compared to the main pumping system, but one factor is extremely important in these tests. Whereas the condensed gases such as the CO₂ and H₂O are relatively insensitive to slight fluctuations of the cryosurface temperature ($\pm 1.0^\circ\text{K}$), sorbed gases are extremely sensitive to temperature fluctuations ($\pm 0.1^\circ\text{K}$). Therefore, in a system operating below the 10^{-8} torr range, slight fluctuations in the temperature of the cryosurfaces lead to relatively large pressure spikes caused by sorbed gases being released from the cryodeposits.

Thus, it becomes very important to maintain a stable temperature control of all cryogenically cooled surfaces. Initially the cryogenic liquid level in this system was monitored by a level sensor which provided a signal that was electronically amplified to operate a solenoid valve in the cryogenic supply line. Even though the supply lines were vacuum jacketed, there was enough heat leak to form a gas pocket in the system when the solenoid valve was closed. When the valve opened this gas preceded the cryogenic liquid and produced a slight warming of the cryosurfaces, causing objectional pressure fluctuations each time the level controller cycled.

Such gas pressure spikes from the 77°K shroud prompted the development of a proportional LN₂ level control. This simple design has provided trouble-free operation with stable temperatures. An overall view of the system is shown in Fig. 5.

During initial cooldown of cold traps and shrouds it is found that LN₂ is entrained in the returning boiloff gas. This percolation of liquid nitrogen had been noticed previously and had resulted in erratic behavior of a commercially available electronic level sensor located in the vent line. The present system recovers this liquid, as the boiloff gas vents back through the reservoir, and thus any liquid is returned to the supply.

The boiloff rate of the LN₂ reservoir without any feed systems open is 0.25 liters per hour. Cooldown time of the cold trap and shroud is 20-percent longer than with the previous forced feed system. Once cool, however, there are no noticeable temperature fluctuations. Details of the LN₂ system are described in the Appendix.

5.2 ACTIVATING THE PUMP

The first deposits were added to the pump surface by introducing the A through the gas addition tube which ended facing the pump. The addition rate (10^{-2} torr-liters/sec) was kept low in an attempt to avoid jet streaming, since an even layer of cryofrost over as large an area of the pump surface as possible was desired. Lower pumping speeds than expected for He by these deposits, however, indicated that the gas flow was evidently so directional that the pump surface was only partially coated. Subsequent tests were made by adding all gases through the addition system which ended outside the radiation shroud. This assured a random incidence of gas molecules at the plane of the pump orifice and thus gave more even cryodeposits. This method of adding the substrate cryodeposit to the pump surface was preferred for these tests since a partial coating implied an uneven thickness of the coating, leading to possible ambiguities in interpreting test results.

5.3 SORPTION ISOTHERMS

Previous exploratory tests (Ref. 1) had indicated that the total cryodeposit was active in the sorption process. Therefore, a series of runs was made with different thicknesses of A deposited on the pump. The isotherm data for different thicknesses are shown in Fig. 6. The shift of the data for the thickest layer could be caused by a temperature

gradient existing in the cryodeposit. Although no reliable data on the density and thermal conductivity of this cryodeposit are available, rough calculations indicate that a surface temperature of 4.4°K is not unreasonable. This high temperature would have a drastic effect on the capacity of the cryodeposit. The thinner layers do indicate that the capacity of the cryodeposit is proportional to the amount.

5.4 PUMPING SPEED MEASUREMENTS

The pumping speed for the common atmospheric gases was measured and is shown in Table I, Appendix II. These values are close to those predicted from free-molecule rate of strike calculations assuming unity capture coefficient.

Pumping speed measurements for He were made with three thicknesses of A cryodeposit. It was noted that the initial pumping speed was independent of the thickness. In all cases the effective pumping speed started to deteriorate as the sorbent became saturated; this was expected. Tests with the thickest deposit also showed that the pumping speed was pressure dependent. That is, an increase in He addition rate resulted in a decrease in the pumping speed. Conversely, when the He addition rate was lowered the pumping speed recovered to its original value. A plot of pumping speed versus He addition rate is shown in Fig. 7. This type of dependence of the pumping speed had been noted previously with H₂ sorption by 20°K CO₂ (Ref. 3). Subcooling the CO₂ cryodeposit to 12°K had significantly influenced this dependence and led to the postulation that the pumping speed was in fact limited by the rate of diffusion of the sorbed molecules into the sorbent. To check the similarity, a series of tests was made with the A cryodeposit subcooled. Two effects were noted: (1) The capacity of the cryodeposit, as reflected in the isotherm, was significantly improved at the lower temperature (Fig. 8), and (2) the pumping speed was maintained with much higher He addition rates (Fig. 7).

5.5 STABILITY OF SORBED He

At this point in the test series it was determined that for the thin layers, the complete cryodeposit was active as a sorbent, and the diffusion rate into the cryodeposit matrix from a region of high concentration at the surface was quite rapid. Thus, it was reasoned that if an A cryodeposit were saturated with He and then a comparable layer of A were overcoated, the He should also diffuse out through this overcoating.

To check this possibility a thin layer (6.6 torr-liters)* of A was precoated on the pump and He was added (0.2 torr-liters) until the sorbent was saturated and the He partial pressure was 1×10^{-5} torr. Then a second layer (6.6 torr-liters) of A was added to the pump. From the previous isotherm data the final equilibrium He partial pressure was expected to be 1.5×10^{-7} torr. The He partial pressure was monitored during the A addition by the mass spectrometer. By the end of the addition the He partial pressure had dropped below 1×10^{-9} torr. The system was monitored for 1 hr with no indication of any rise in the He partial pressure. This second coating of A was then saturated with another addition of He and again overcoated with A with similar results.

Since these results suggested that the He, once sorbed, was held tenaciously in the A matrix, a test was made by depositing a thin layer of A (6.6 torr-liters) as before and saturating it with He to a partial pressure of 1×10^{-5} torr. Then the gate valve to the diffusion pump was opened. The He partial pressure as indicated by the mass spectrometer did not fall as rapidly as expected, and when the gate valve was closed the He partial pressure rose to an intermediate value. This indicates that the He is mobile enough to escape from the cryodeposit.

To date there is no satisfying explanation for this anomaly. The results of the experiments reported in the next section may hold the answer. Unfortunately, this answer raises other questions. Nonetheless, the phenomenon noted is favorable in the operation of the pump since a saturated layer can be sealed by a subsequent cryodeposit.

5.6 REJUVENATION OF THE PUMP

Several runs were made where a measured quantity (6 torr-liters) of A was precoated on the pump and then this deposit used to pump He. When the cryodeposit had become saturated and the He partial pressure had risen to the 10^{-5} torr range, a second layer of A was added, reducing the cell pressure to the 10^{-10} torr range. The pump thus rejuvenated was used to pump a second set of He additions again up to a saturation pressure of 10^{-5} torr. Three rejuvenations were made with no significant change in the pumping speed.

These data are reported two ways. In Fig. 9 the sorption isotherm for each deposit is plotted as though it was a complete and separate deposit. The isotherm for the first layer agrees well with data taken

*(Approximately 10^{-5} cm thick)

previously (Fig. 6). The change in slope of the isotherms for subsequent layers indicate that they cannot be considered as independent deposits.

Figure 10 displays the same data but as each layer is added it is assumed that the complete cryodeposit is the effective sorbent. The fact that these curves are parallel indicates that this is a more logical assumption. A possible explanation of the shift of each curve may be that the sorption has both a surface and a volume contribution.

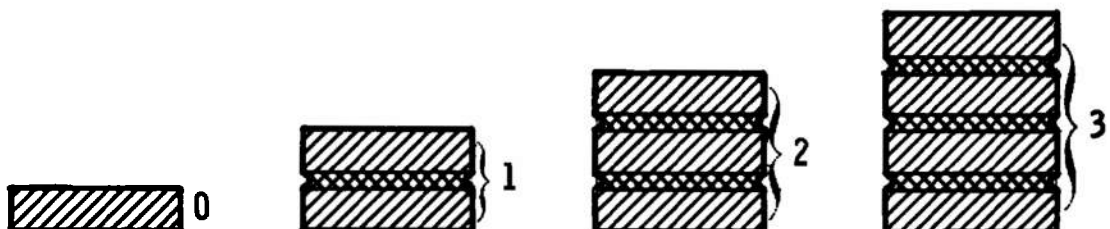
To examine the shift of these curves a little more closely, the total quantity of He sorbed by the cryodeposits for an equilibrium pressure of 1×10^{-6} torr is shown below.

<u>A, torr-liters</u>	<u>He/A</u>	<u>He, torr-liters</u>	<u>He Normalized to the value of 0.118</u>
6	0.0196	0.118	1
12	0.0345	0.414	3.5
18	0.0395	0.710	6
24	0.0425	1.020	~8.5

The data reported in Section 5.3 indicate that for these deposits the "capacity" should be directly proportional to the volume. Therefore, if we divide the helium sorbed into volume and surface components then the normalized values can be chosen as

<u>x (volume)</u>		<u>y (interface)</u>	<u>Number of Interfaces</u>
1	+	0	0
2	+	1.5	1
3	+	3	2
4	+	4.5	3

Thus, in considering the sequence of events in building up the cryodeposit, the sequence of saturated layers is shown by the following sketch.



In looking at these deposits and trying to correlate the observed sorption phenomena with some consistent model, if we assume that the quantity sorbed tabulated as (x) is a volume sorption, then the quantity

tabulated as (y) could be the quantity of He trapped at the interface between layers of cryodeposit. This model explains the observed facts that the capacity of a single deposit is proportional to the volume, and accounts for the additional capacity of the multilayers (up to the limit as noted in Section 5.2). The additional capacity of the interface could also be the explanation of why the pressure did not rise as expected when the deposit was overcoated as described in Section 5.4. However, there is no ready explanation why this interface should be so active in sorbing the He.

The proposed model is speculative at this point since it is based on a single set of experimental data. A definitive experiment is planned for the future where the procedure of overcoating will be repeated with a different quantity of A used for the layers. If this model is correct then the interface capacity should stay approximately the same, whereas the volume capacity should vary with the thickness of the layers.

5.7 EFFECTS OF CONTAMINATION

This prototype pump was designed primarily to pump He. Since in most applications of the pump the gas load from the vacuum system will be a mixture of gases, it was necessary to study the effects of pumping some of the common atmospheric gases. A preliminary test run, where the pump was precoated with A and then contaminated by small quantities of O₂ and N₂, indicated that there was no significant change in the He pumping speed. To define more clearly the effects of contamination, a series of runs was made where the pump was precoated with a known amount of O₂ and an He sorption isotherm made. A second series with the pump precoated with N₂ was used to obtain an isotherm for the N₂/He system. The isotherms obtained for He on A, N₂, O₂, and CO₂ are shown in Fig. 11. These isotherms are in each case for cryodeposits of about 50 torr-liters precoated on the pump. These data indicate that it is not necessary to shield the pumping surface from contamination by these gases.

The final contamination study was made using H₂ as the contaminant. A thick layer of A (2420 torr-liters) was precoated as before, and several additions of He were made to determine its pumping speed on this cryodeposit. Then H₂ was added at different rates and its pumping speed noted. After each H₂ addition the chamber pressure dropped to its base level (4×10^{-9} torr). Since the vapor pressure of H₂ at 4.2°K is on the order of 3×10^{-7} torr, these results would indicate that the H₂ is also being absorbed by the A cryodeposit. A total of 28.3 torr-liters of H₂ were added to the deposit. Then the gas addition system was

purged and He was again added to the chamber. Three effects were noted: (1) the pumping speed of the He was noticeably reduced (from 7200 to 270 liters/sec), (2) the time for the system to reach its equilibrium value after each He addition was increased (10 min as compared to 2 min previously), and (3) the capacity for He was reduced (the equilibrium partial pressure after adding only 0.8 torr-liters of He was as high as when 14 torr-liters had been added to a similar A deposit previously). Since only one data point is available it may be just coincidence that sorption of 28 torr-liters of H₂ was equivalent to 14 torr-liters of He.

A qualitative appraisal of the observed effects suggests that not only does the H₂ occupy adsorption sites within the cryodeposit matrix which would normally be available for the He molecules, but also acts as a barrier slowing the diffusion of the He into the cryodeposit.

A separate test was made to measure the pumping speed of H₂ on the bare stainless steel pump surface cooled to 4.2°K. The measured pumping speed of 10,000 liters/sec agreed with the values obtained for the H₂ pumped by the A-coated surface. This value is close to the pump's theoretical maximum pumping speed, based on the molecular strike rate calculated from the kinetic theory. The equilibrium H₂ vapor pressure measured during these runs was 9×10^{-7} torr.

An attempt was made to overcoat this H₂ deposit with a layer of A cryodeposit to see if it would effectively seal the H₂ surface. After adding the A it was noticed that small vibrations of the chamber caused large pressure bursts (up to 10^{-3} torr). Using the mass spectrometer it was determined that these pressure bursts were caused by the A. Although the chamber has no viewing ports it is surmised that the A had been deposited in small crystals upon the H₂ substrate, and that small vibrations were shaking them loose. Upon dropping to the 77°K shroud they evaporated, causing a rapid pressure rise, and then the A was pumped again on the 4.2°K pump surface.

Since adhesion of the cryodeposit to the pump surface could be a major problem, several combinations of gas additions were tried. The only problems occurred when H₂ was used as the prime coating. Combinations of A-H₂-A-H₂ showed no tendencies to shedding.

5.8 USE OF CO₂ AS A SUBSTRATE

The radiation shroud in the test chamber was normally cooled to 77°K for all pumping tests. A separate test was made where the

radiation shroud was left at room temperature. The pump was cooled with LHe, and CO₂ was precoated on its surface. During this time the pumping speed of the CO₂ was measured (Table I). Then He was added at several rates and its pumping speed recorded. The LHe usage rate was high because of the high radiation heat load on the pump, and there was not enough time to make isotherm measurements for this CO₂/He system. The examination of CO₂ indicates that it is superior to any of the other gases tested as a sorbent for He. However, there are several problems attendant to its use as a substrate. Several modifications were required to obtain an isotherm of He on CO₂ with the shroud cooled to 77°K. Since the gas addition tube normally is cooled by radiation to LN₂ temperatures, a heater wire had to be installed to prevent the CO₂ from cryopumping inside the tube. Also, the lip of the radiation shield around the pump is cooled to temperatures between the 4.2°K pump and the 77°K shroud in the chamber. During the coating of the pump, 50 percent of the CO₂ is cryopumped on this intermediate temperature surface and the LN₂ shroud. The vapor pressure of CO₂ on this surface resulted in a limiting base pressure for the system (10⁻⁸ torr). This base pressure slowly dropped over a 24-hr period indicating a gradual transfer of CO₂ from 77°K to 4.2°K surfaces. The problems encountered in running these tests, although not insurmountable, were considered serious enough to warrant recommending A as the basic substrate for general use of the pump.

5.9 THERMAL LOADS ON PUMP

Prior to building the pumping system, a rough calculation was made of the heat loads from radiation and conduction. The radiation load was estimated at 0.15 w. Conduction load from the main support tube was expected to be 0.04 w, and heat conduction through a tube installed to monitor the He vapor pressure was estimated at 0.32 w. This gave a total heat load of 0.51 w.

The actual heat load was measured by recording the boiloff rate of the LHe. The He gas was vented via the transfer line, through a warm-up coil, and then through a wet test meter. During the pumping runs the gas boiloff rate was so low that it had reached room temperature by the time it passed through the wet test meter. The average boiloff gas rate was measured at 12.5 liters/min (NTP). Using only the heat of vaporization of LHe, this represents a heat load at the pump of 0.49 w.

Assuming that the calculated values are correct, then elimination of the vapor pressure monitoring tube should reduce the heat load by a factor of 2.7 and thus increase the operating time by a similar amount.

The heat load on the pumping system was measured as a function of the He partial pressure in the chamber and is shown in Fig. 12. These data were taken while He pumping runs were being made. Although the pump still operated at these higher heat loads, the penalty paid in high LHe usage rates would suggest a practical operating limit in the 10^{-5} torr range.

Liquid helium consumption rates in the 2.3°K experiments indicated that approximately one-third of the LHe supply was used during the subcooling period and thereafter the usage rate was not significantly different from that at 4.2°K.

SECTION VI CONCLUSIONS

A prototype cryosorption pump using a cryodeposit as the working medium has been constructed and shown to be practical. It can be easily built and is relatively inexpensive when compared to the cost of pumping systems that can match its performance. It is flexible enough that it can be adapted to existing chambers either as an appendage pump or as part of an integral shroud and pumping system. It combines the advantage of providing an ultrapure, ultrahigh vacuum with an exceptionally high pumping speed (Table I). No bakeout is necessary to activate the pump.

The contamination tests indicate that no selective pumping baffles are required. Although H₂ and He are competitive for the available sorption sites, this presents no problem since the pump is capable of being rejuvenated in situ. If the pump is pumping gas mixtures which include the common atmospheric gases (O₂, N₂, and A) then it is self-rejuvenating. If the sole gas load is H₂ or He then when the pump becomes saturated it may be rejuvenated by addition of a further A cryodeposit.

REFERENCES

1. Haygood, J. D. and Dawbarn, R. "Helium Pumping by 4.2°K Cryodeposits." AEDC-TR-66-204 (AD645511), January 1967.
2. Moody, T. L. "Capture Coefficient of 300°K CO₂ on a 77°K Surface as Measured by the Rotating Gage Technique." AEDC-TR-66-231 (AD645510), January 1967.

3. Dawbarn, R. "Cryosorption of Hydrogen by 12-20°K Carbon Dioxide Cryodeposits." AEDC-TR-67-125 (AD655067), July 1967.
4. Brackmann, R. T. and Fite, W. L. "Condensation of Atomic and Molecular Hydrogen at Low Temperatures." J. Chem. Phys. Vol. 34, No. 5, May 1961, p. 1572.
5. Nichols, J. E. "Trapping and Sorption of Noncondensable Gases in the Cold Diffusion Pump." AEDC-TDR-62-124 (AD282904), August 1962.
6. Haygood, J. D. and Wang, E. S. J. "The Trapping Effect in Cryopumping." AEDC-TDR-62-133 (AD282891), August 1962.

APPENDIXES

- I. ILLUSTRATIONS**
- II. TABLES**
- III. DETAILS OF COMPONENTS IN LN₂ SYSTEM**

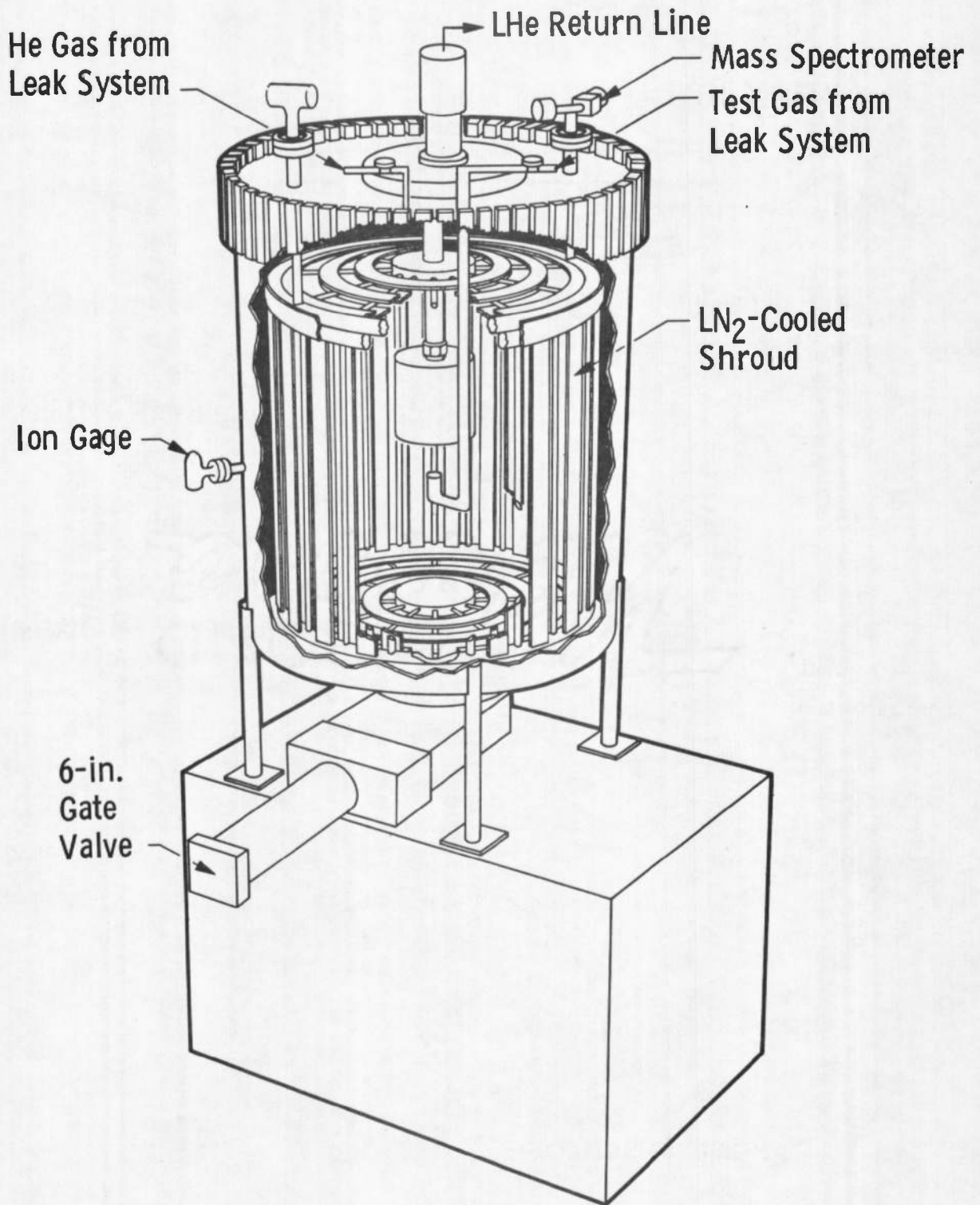


Fig. 1 2- by 3-ft Research Ultrahigh Vacuum Chamber

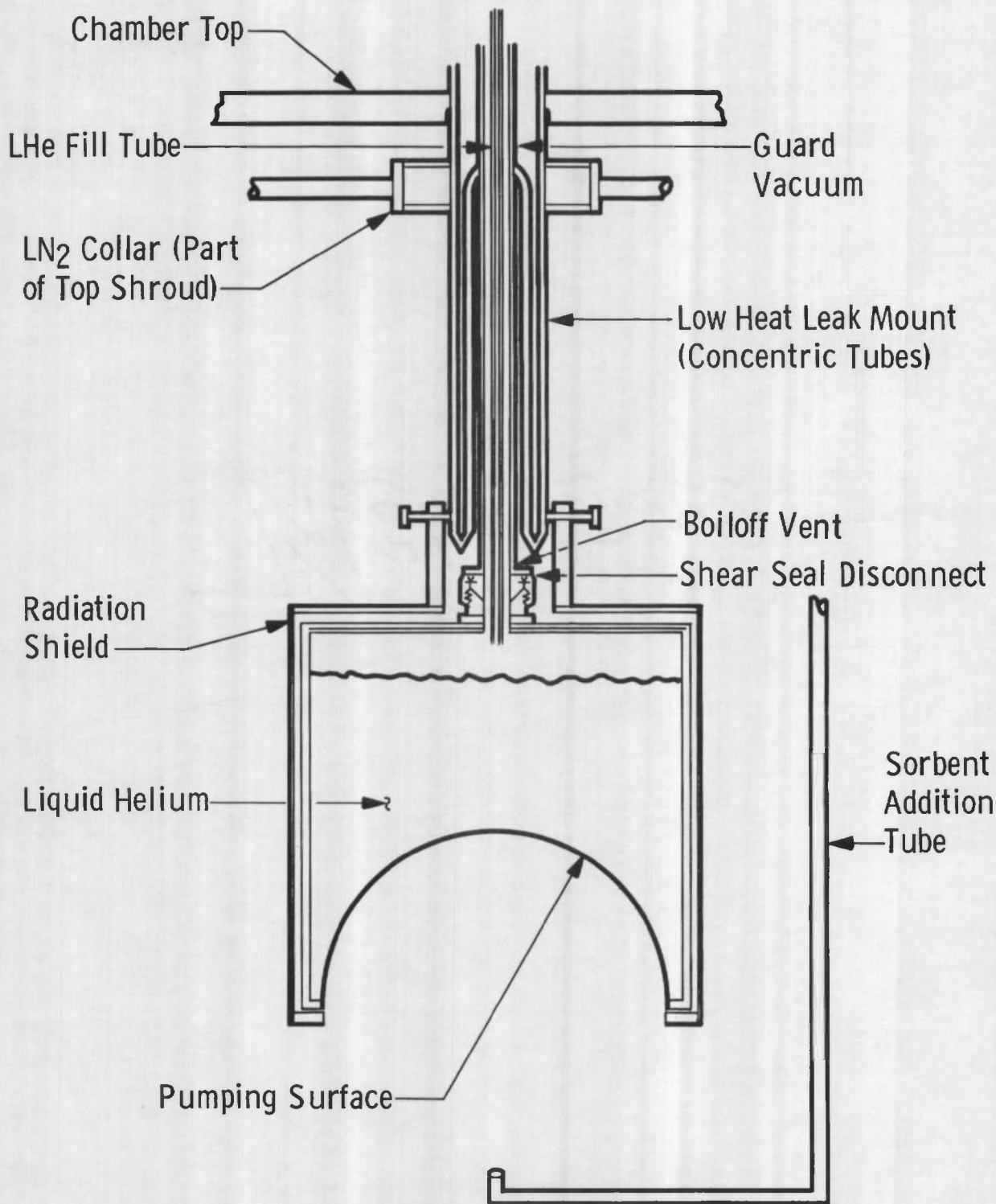


Fig. 2 Cryosorption Pump

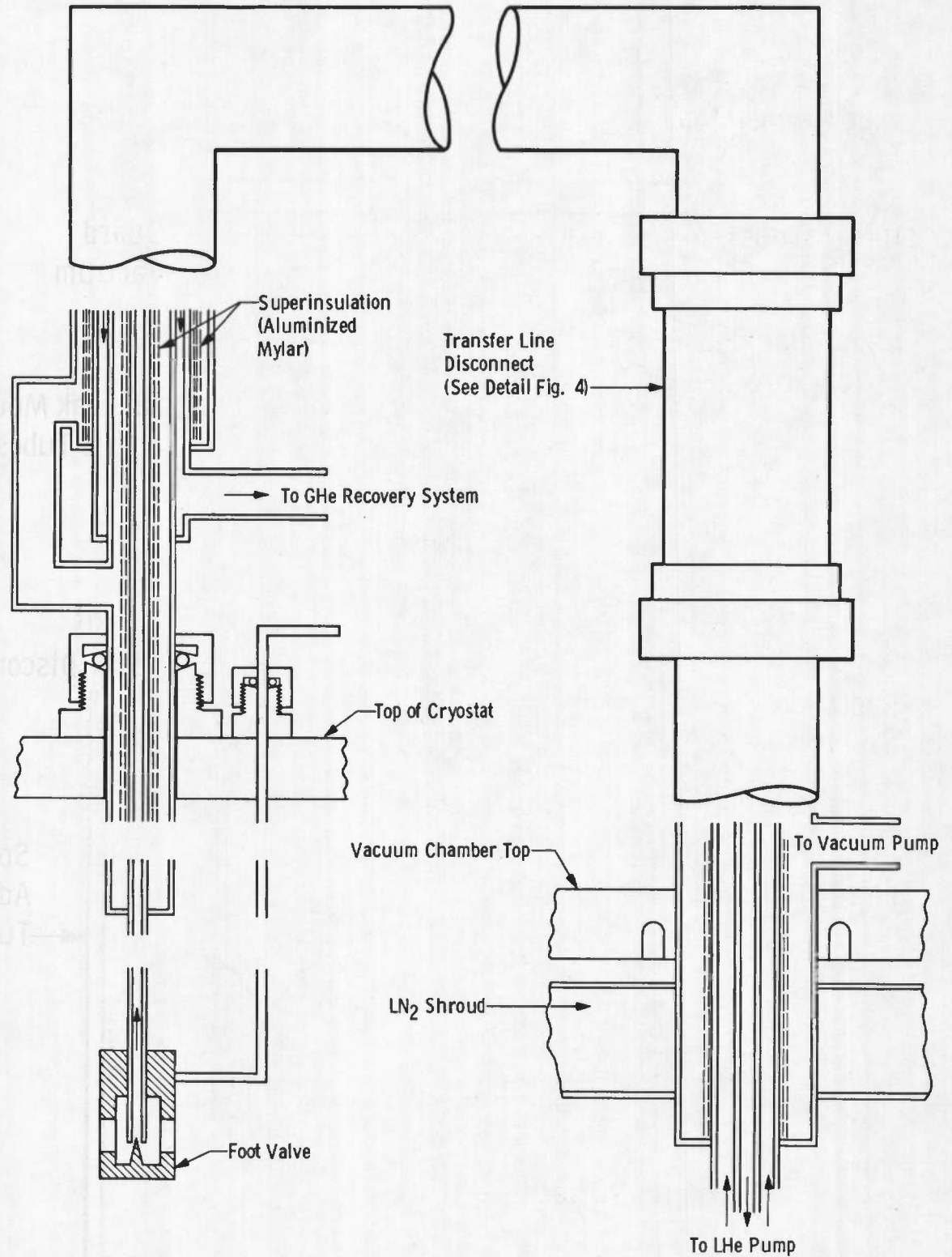


Fig. 3 LHe Transfer Line

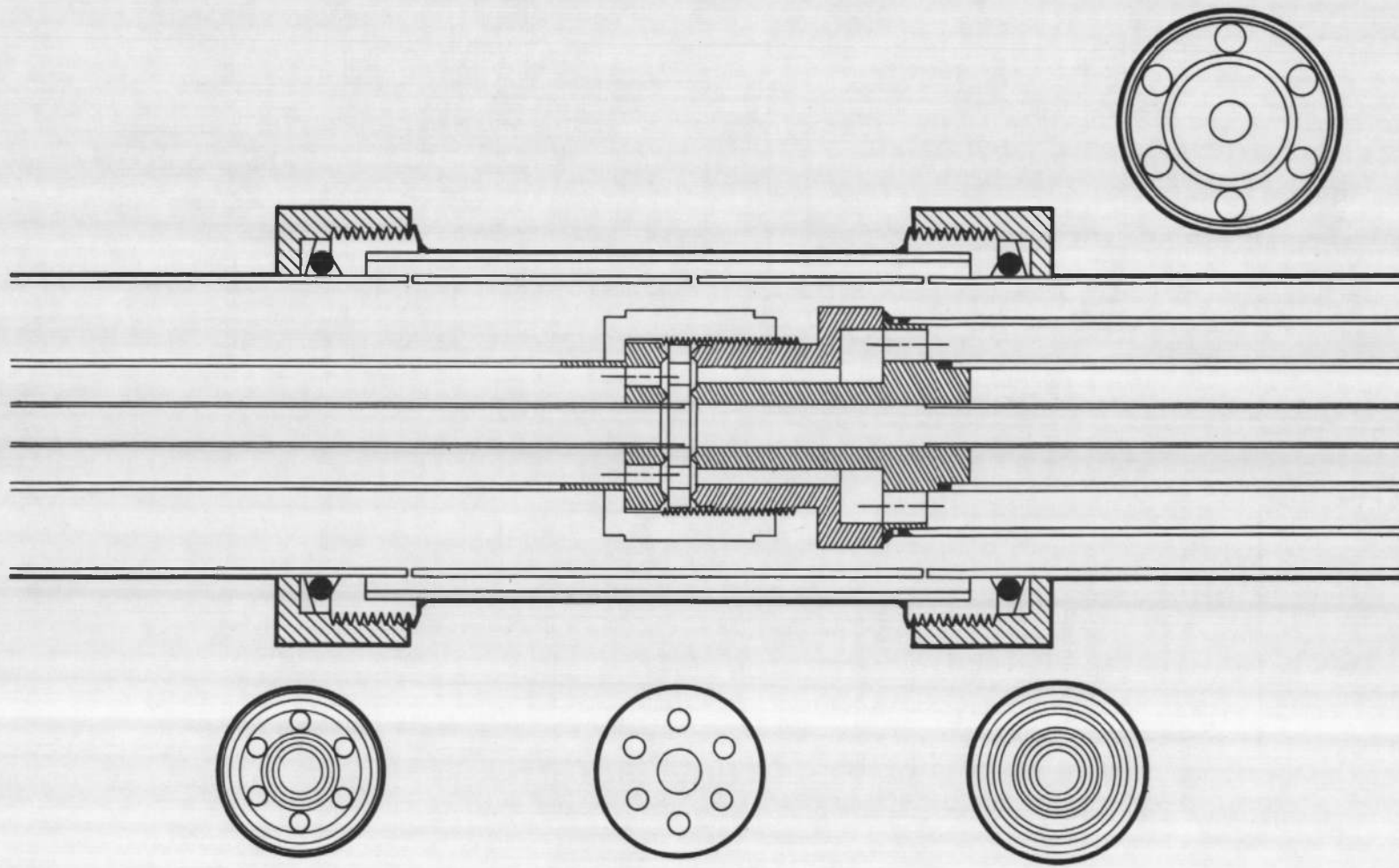


Fig. 4 Disconnect in LHe Transfer Line

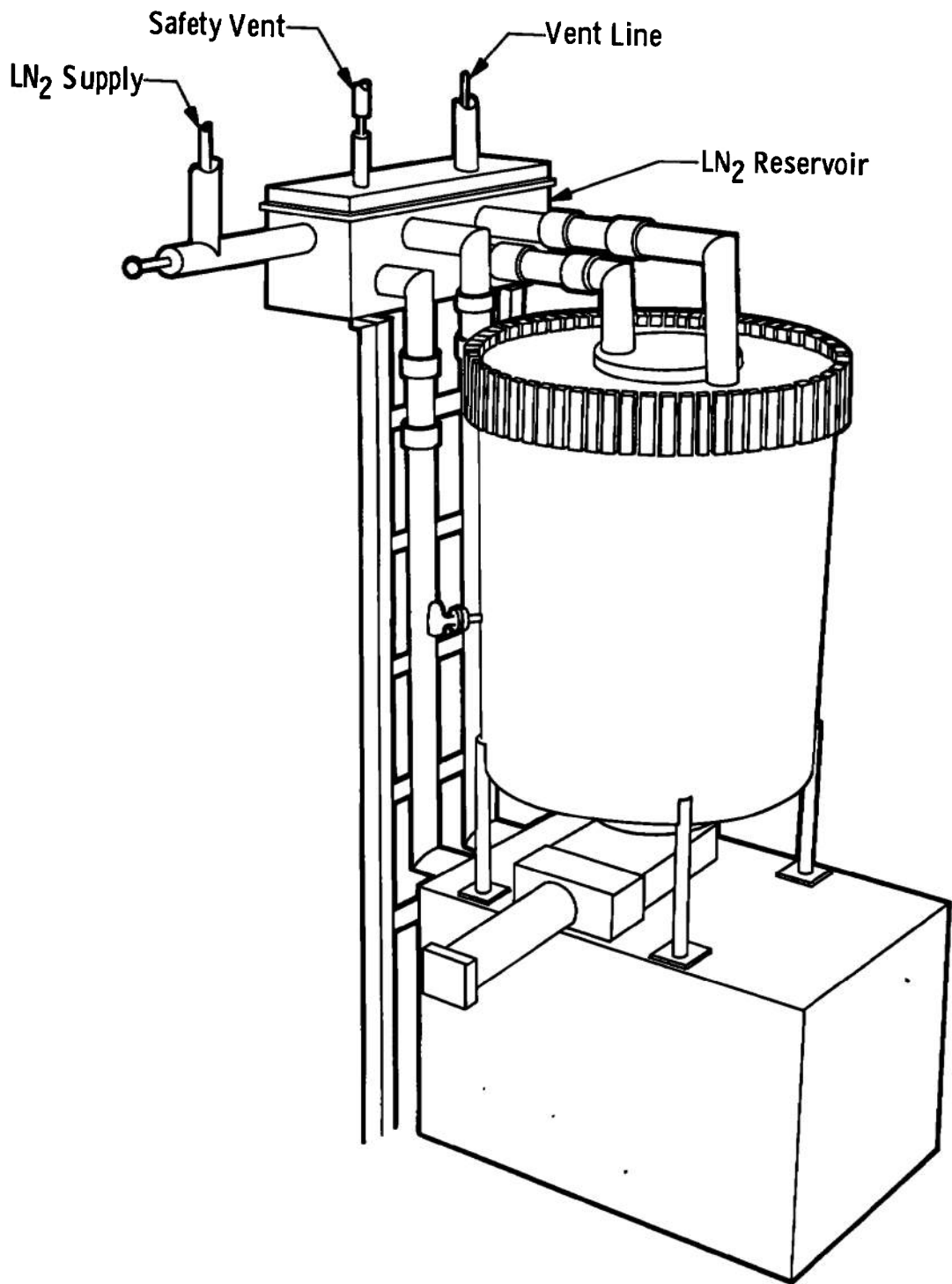


Fig. 5 Vacuum Chamber and LN₂ System

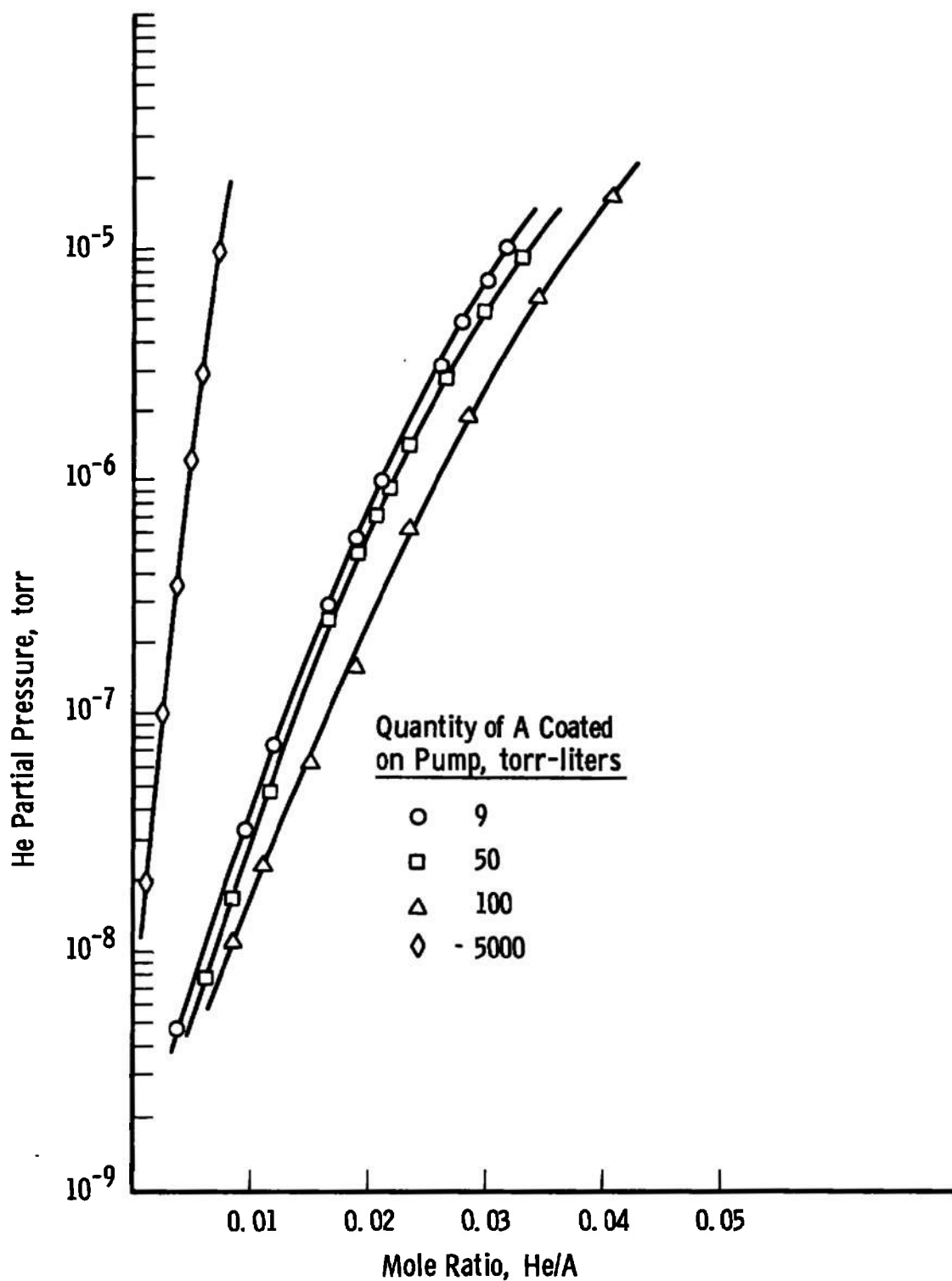


Fig. 6 Sorption Isotherms He/A

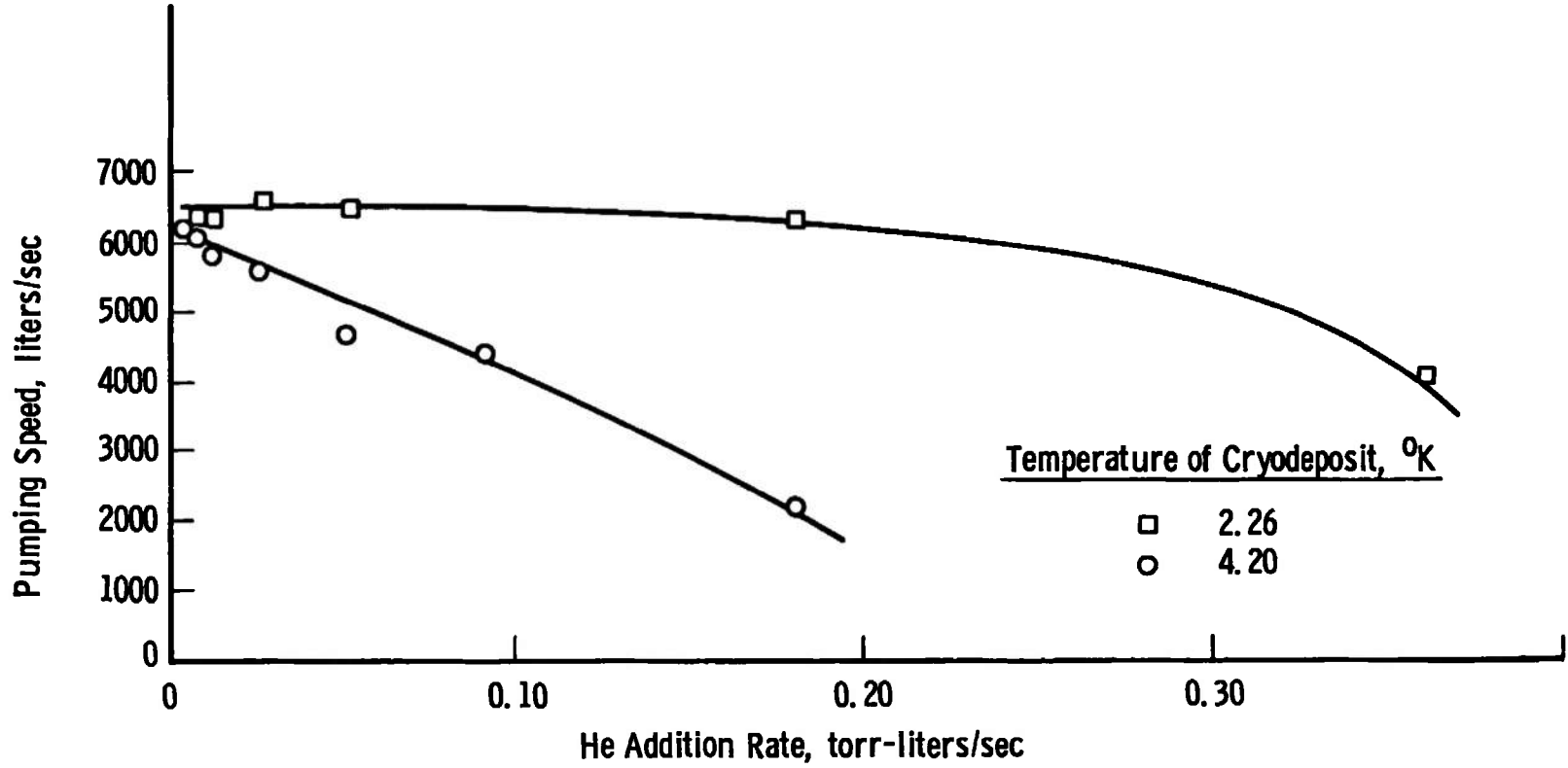


Fig. 7 Pumping Speed versus Addition Rate

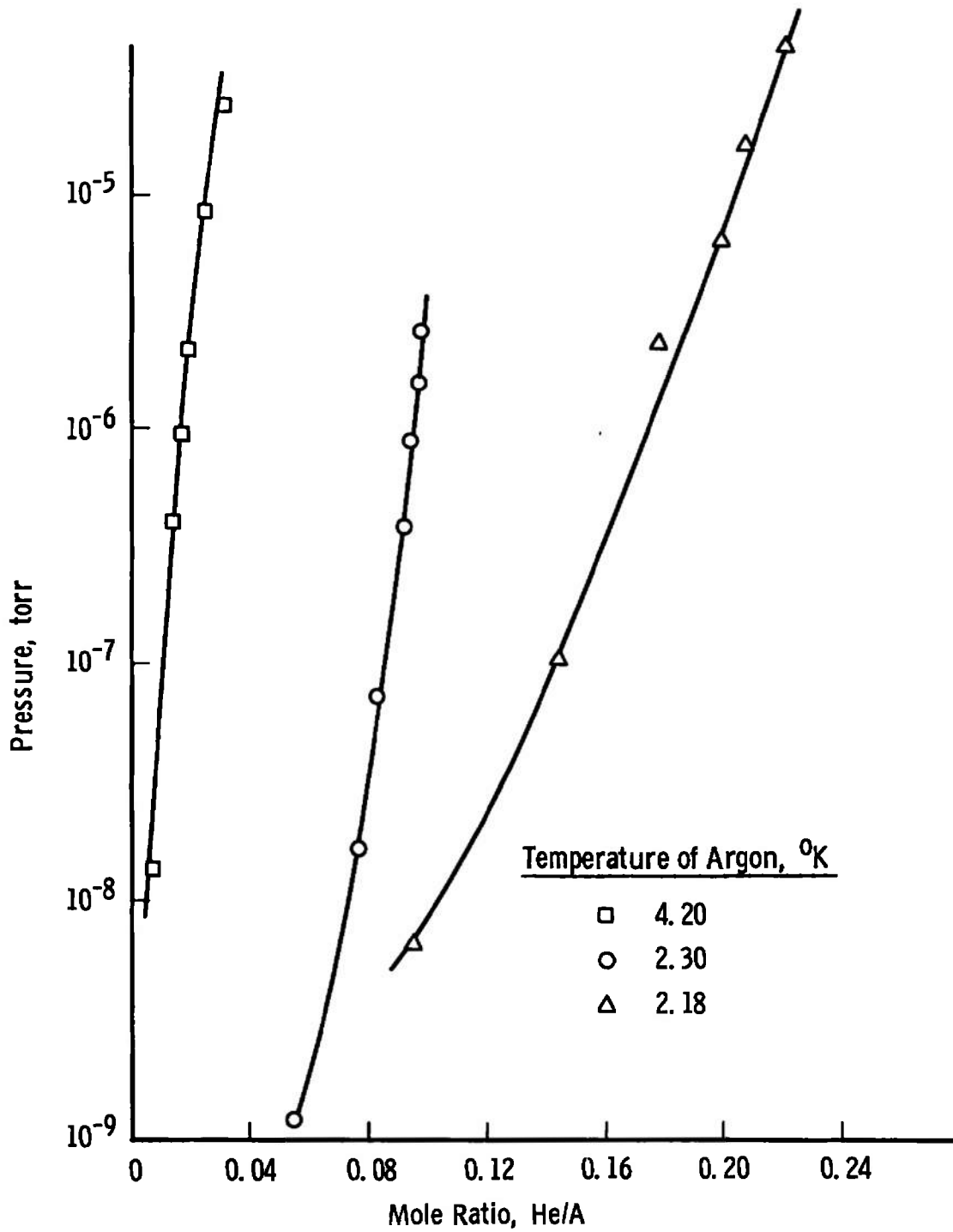


Fig. 8 Sorption Isotherms He/A at 4.2°K, 2.3°K, and 2.18°K

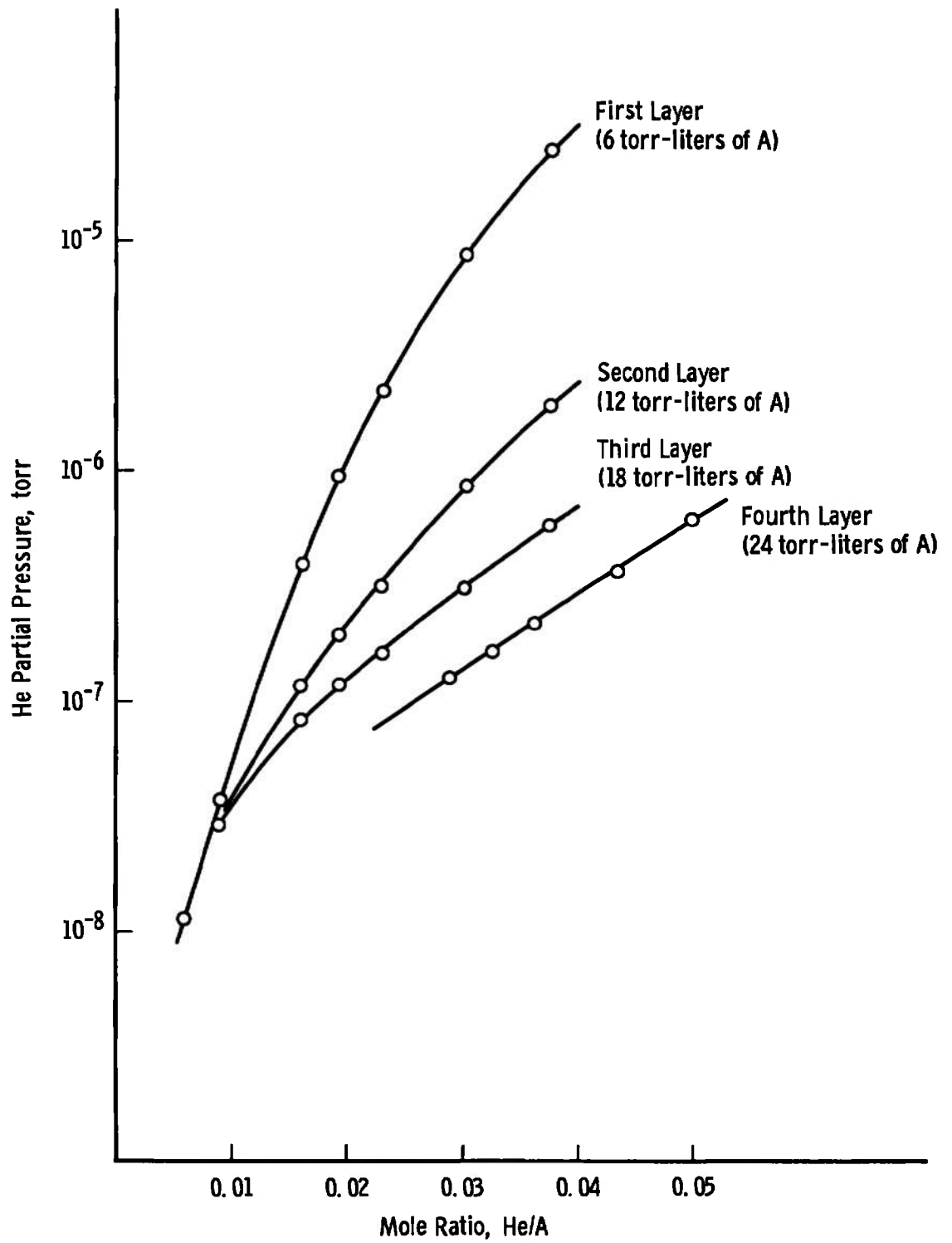


Fig. 9 Sorption Isotherms for Multilayer Deposits of He/A

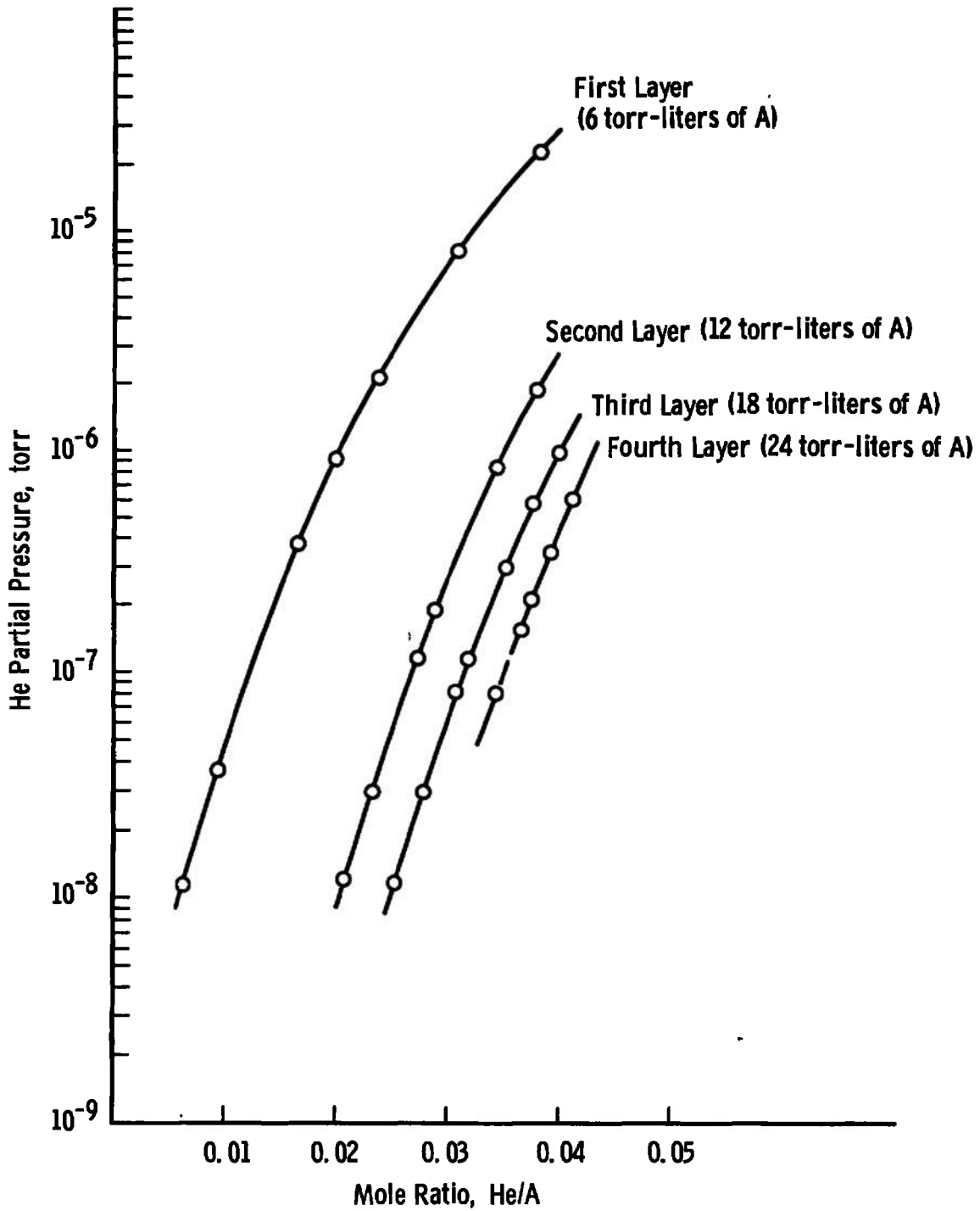


Fig. 10 Sorption Isotherms for Multilayer Deposits of He/A

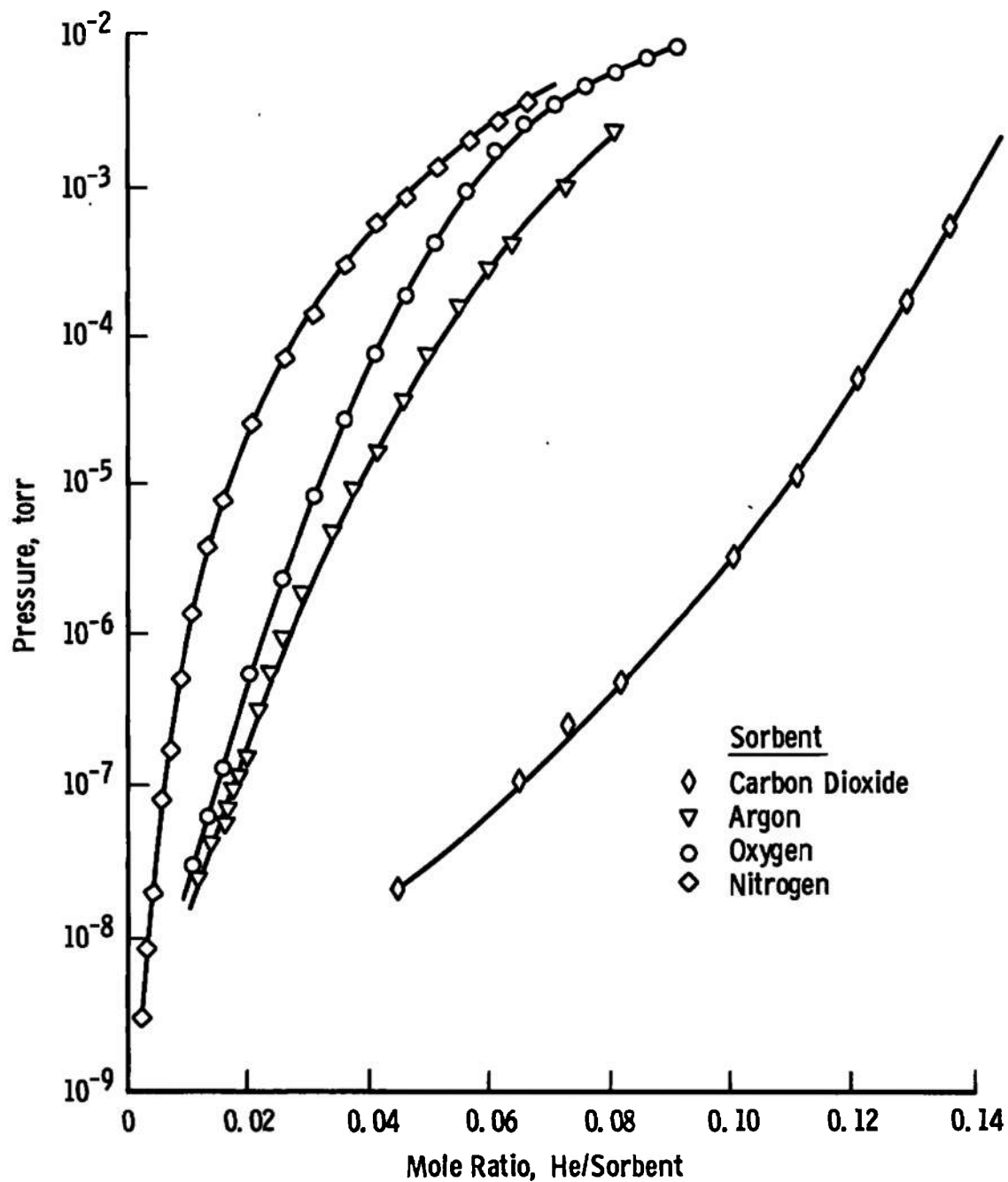


Fig. 11 Sorption Isotherms for He on CO₂, A, O₂, and N₂

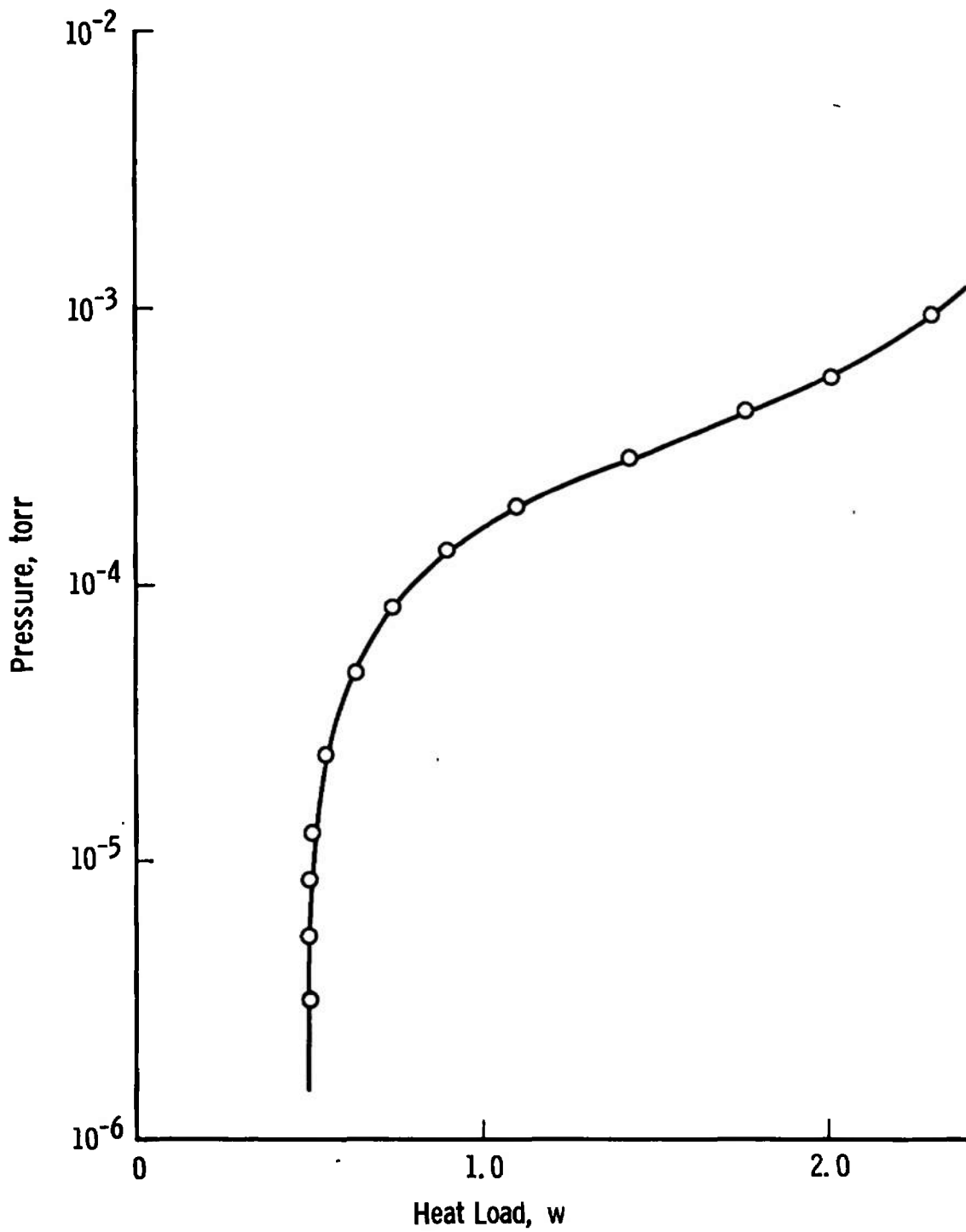


Fig. 12 Measured Heat Loads on Pump as a Function of Chamber Pressure (He)

TABLE I
PUMPING SPEEDS OF VARIOUS GASES

Gas	Substrate	Gas Temperature, °K	Pumping Speed (at gas temperature noted), liters/sec	Pump Temperature, °K
A	A	77	2,580	4.2
N ₂	N ₂	77	3,170	4.2
H ₂	H ₂	77	10,900	4.2
CO ₂	CO ₂	300	4,750	4.2
He	A	77	6,250*	4.2
He	A	77	6,900	2.8
He	N ₂	77	6,300	4.2
He	O ₂	77	6,600	4.2
He	CO ₂	77	6,200	4.2
He	CO ₂	300	8,300	4.2
H ₂	A	77	9,400	4.2

*This value is an average value from four different series of tests. Total scatter of pumping speed data from different cryodeposits was less than 5 percent.

APPENDIX III DETAILS OF COMPONENTS IN LN₂ SYSTEM

RESERVOIR

The reservoir consists of a vacuum-jacketed steel box scaled as shown in Fig. III-1. The vacuum jacket, besides providing heat insulation for the LN₂ in the reservoir, also serves as the vacuum manifold to pump out the vacuum jackets of the feeder lines after assembly. The top to the reservoir is bolted on, and is sealed by a leather gasket. Although this material is not gas tight, it remains flexible at 77°K and provides sufficient sealing to prevent H₂O vapor and O₂ from condensing in the liquid nitrogen.

VALVE AND FLOAT

The valve and float assemblies are shown in detail in Fig. III-2. The valve assembly consists of a steel housing built from modified AN flare fittings with a machined plunger and copper seat. In operation it provides a steady venting of cold gas from the central supply line and, when necessary, opens sufficiently to maintain a constant liquid level in the reservoir. The float assembly consists of a Styrofoam[®] block. The length of the lever arms and dimensions of the float were calculated to be sufficient to close the valve against a maximum LN₂ supply pressure of 50 psi.

CONTROL VALVES

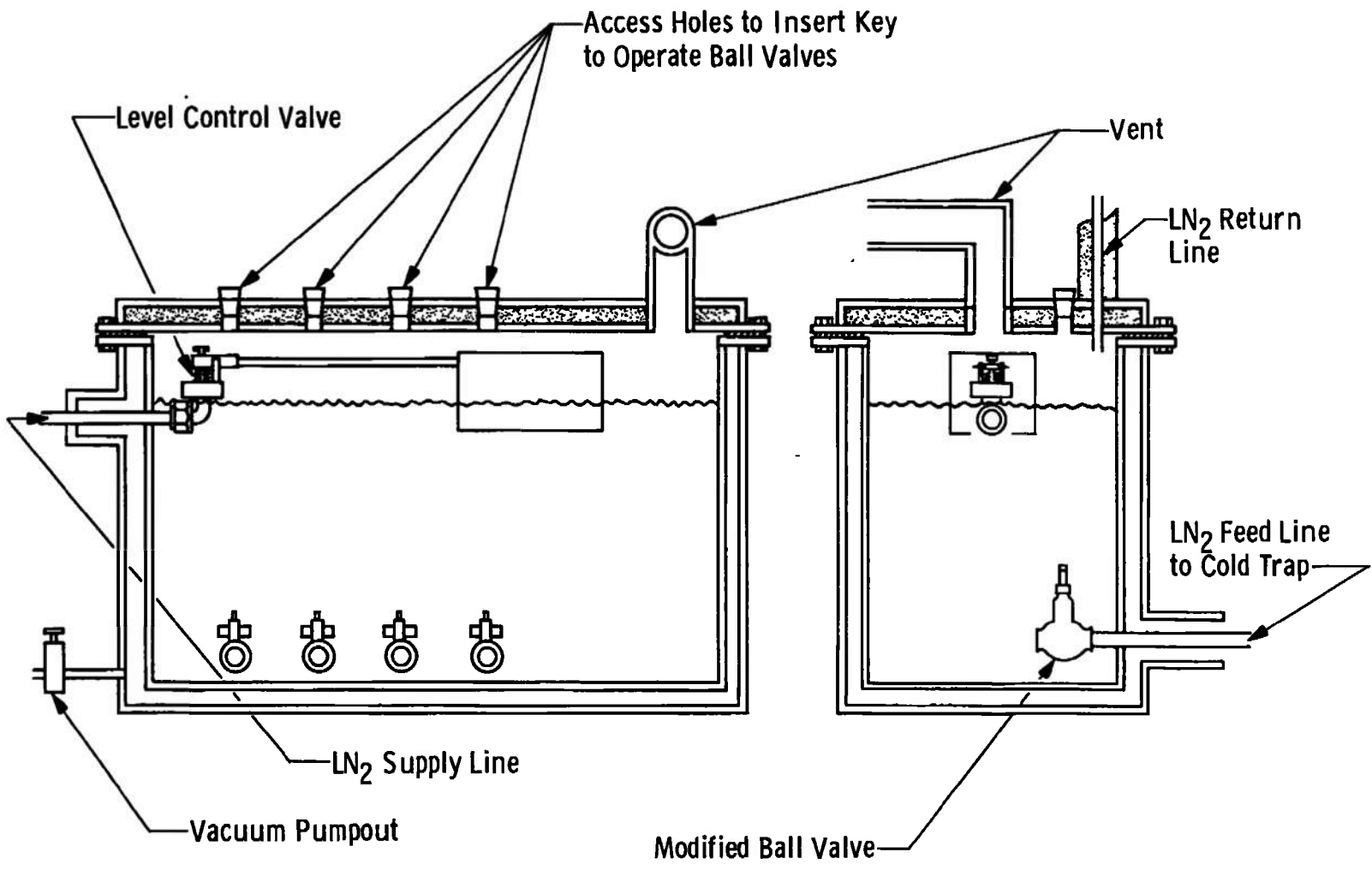
To provide a variety of modes of operation of the cryogenic system in the vacuum chamber, a shutoff valve was installed for each feedline. These valves are standard ball valves modified by removing valve stem packings and handles. The valves are operated by removing the appropriate cap and using an extension key. Since the drive handle only makes contact with the valve when opening or closing, no heat load is introduced to the system except during operation of the valve.

VACUUM-JACKETED DISCONNECTS (PATENT PENDING)

In order to allow for disassembly of the vacuum chamber and to provide flexibility for changes that might be required for specific tests in the future, all LN₂ lines were attached with simple vacuum-jacketed disconnects

(Fig. III-3a). Two knife edges cutting into a copper gasket provide a seal for the cryogenic line, and rubber O-rings are used on the vacuum jacket. The knife-edge disconnect is made by modifying an AN flare cap and plug as indicated in Fig. III-3b. Copper gaskets are punched from 3/32-in. copper sheet.

These disconnects have been made using fittings up to 1.5 in. in diameter. The included angle at the knife edge does not seem to be critical. Fittings with knife-edge angles ranging from 50 to 90 deg have been constructed and found to be leak tight. These disconnects have been used in supply lines carrying 20°K GHe and 4.2°K LHe with no detectable He leak (equipment capable of detecting 10^{-10} std cc/sec leak rates), and have retained their vacuum integrity when heat shocked from bakeout temperatures to LN₂ temperatures.



32

Fig. III-1 Liquid Nitrogen Reservoir

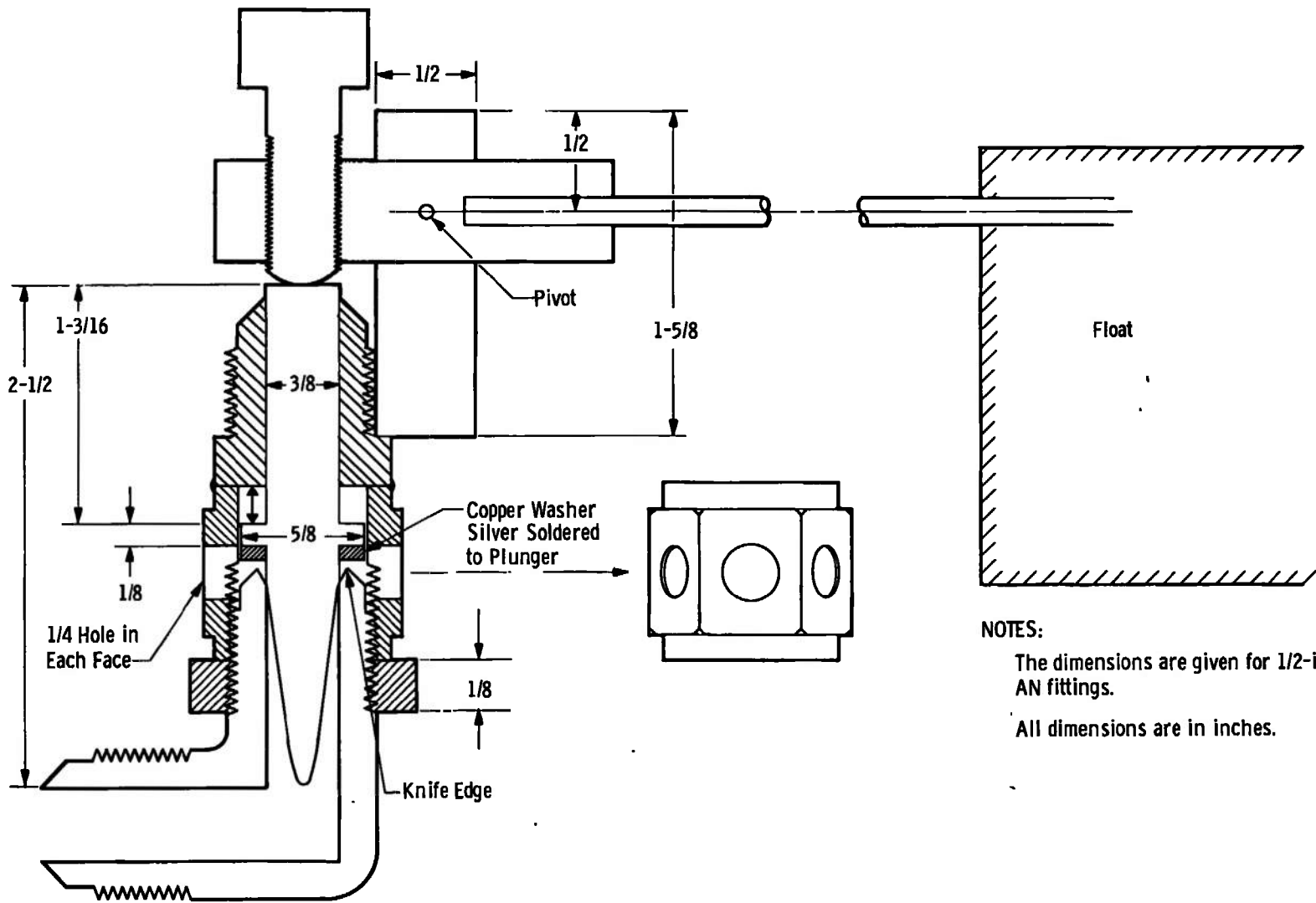
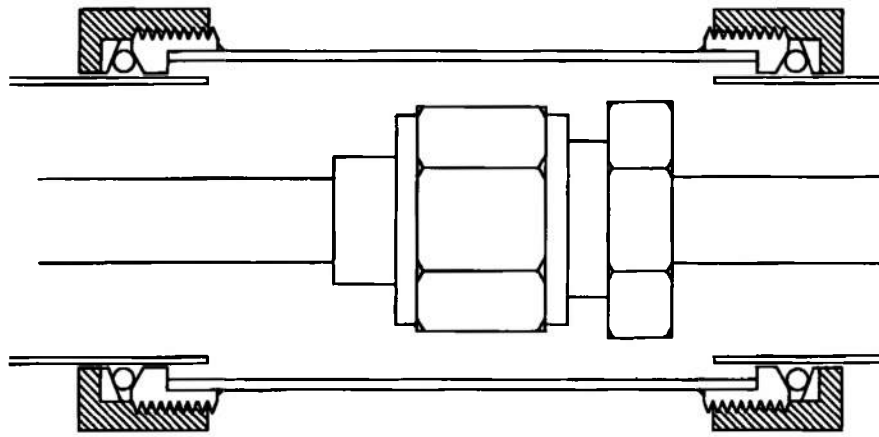
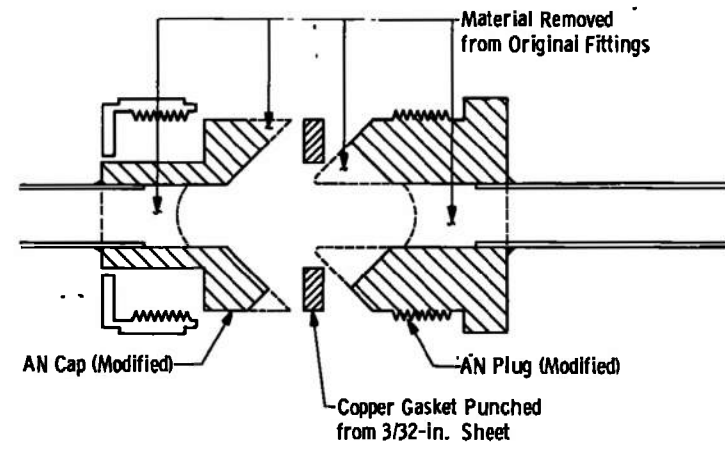


Fig. III-2 Valve and Float System in LN₂ Reservoir



a. Vacuum-Jacketed Disconnect



b. Shear Seal

Fig. III-3 Connector for LN₂ Lines

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13. ABSTRACT A small cryogenically cooled pump has been developed which is capable of pumping all gases including helium and hydrogen. Its low base pressure makes it suitable for use in ultrahigh vacuum systems, and its high pumping speed for all gases is maintained up to pressures in the 10^{-6} torr range. The pump operates by sorbing the normally noncondensable gases in a layer of argon cryofrost which is predeposited on its liquid-helium-cooled surface. As with any sorption pump, the sorbent eventually becomes saturated, and the sorbate partial pressure starts to rise. However, unlike most sorbent pumps, this one can be reactivated simply by recoating the surface with a fresh layer of sorbent in situ. Sorption isotherms have been obtained for He on 4.2°K cryodeposits of N_2 , A_2O_2 , and CO_2 over a pressure range from 10^{-8} to 10^{-3} torr.			

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
1 cryogenics						
2 pumps						
3 helium - Pumping						
vacuum technology						
4 Hydrogen - Pumping -						
5 Cryopumping						
1.5 - 2						