^{3}HELIUM/^{4}HELIUM DILUTION CRYOCOOLER FOR SPACE

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PROJECT SUMMARY

NASA Astrophysics programs require very sensitive sensors for detecting millimeter and sub-millimeter wavelength radiation. The noise equivalent power (NEP) of these sensors, called bolometers, can be increased by a factor of 1000 by operating at 0.1 K instead of 1.5 K.

Two potential methods for cooling sensors to 0.1 K exist; dilution refrigeration and adiabatic demagnetization refrigeration (ADR). The ADR cycle can work independent of a gravitational field. However, the weight, electrical requirements, and fringing magnetic field of an ADR system cause substantial disadvantages for spacecraft applications. A standard dilution refrigeration cycle is gravity dependent, but does not have the negative impact on spacecraft operation present with the ADR. NASA has funded ACE, Inc. under NASA contract no. NAS8-37260 to investigate dilution cooling cycles for low gravity environments. As a result of these studies, two potential zero gravity dilution refrigeration cycles were identified (Hendricks, et al. 1988).

The purpose of this effort has been to design, construct and test prototype dilution cryocoolers based on these two cycles. Although the devices we built and tested did not operate as fully functional dilution cryocoolers, important information was gathered. The porous metal phase separator was demonstrated to operate in the -1-g configuration; this phase separation is the critical element in a ^{3}He circulation dilution cryocooler. Improvements in instrumentation needed for additional tests and development have been identified.

INTRODUCTION

This section, and the section on the operation of dilution cryocoolers in zero gravity, serve as an introduction to the basic technical issues of this project. These sections are excerpts from Hendricks et al., 1988 and are included here for completeness.

Basic Principles of Dilution Cryocooler Operation

The "dilution" cryocooler is based on three basic principles. The first principle is the heat of mixing of ^{3}He in ^{4}He. The reversible dilution of ^{3}He into ^{4}He produces a cooling effect. This cooling is the basis of a "solution" refrigerator using the two liquid helium isotopes. The second basic principle is the phase separation of liquid ^{3}He and ^{4}He below 0.87 Kelvin. The third basic principle is that the "dilute solution" contains roughly 6.4 % of ^{3}He, even at zero temperature. Thus, the heat of mixing is available even at very low temperatures. The phase separation diagram of the two helium isotopes is shown in Figure 1.

A fourth principle, that is not used in the conventional ^{3}He circulation dilution cryocooler, is useful for alternate cycles. This is the fact that the two isotopes can be easily separated using a "superleak". This is only possible below the superfluid transition temperature of ^{4}He. The superleak can serve as a separation membrane in the solution refrigerator.

In this section we will review the basic operation of the solution refrigerator. We will then give a brief analysis of the two currently proven

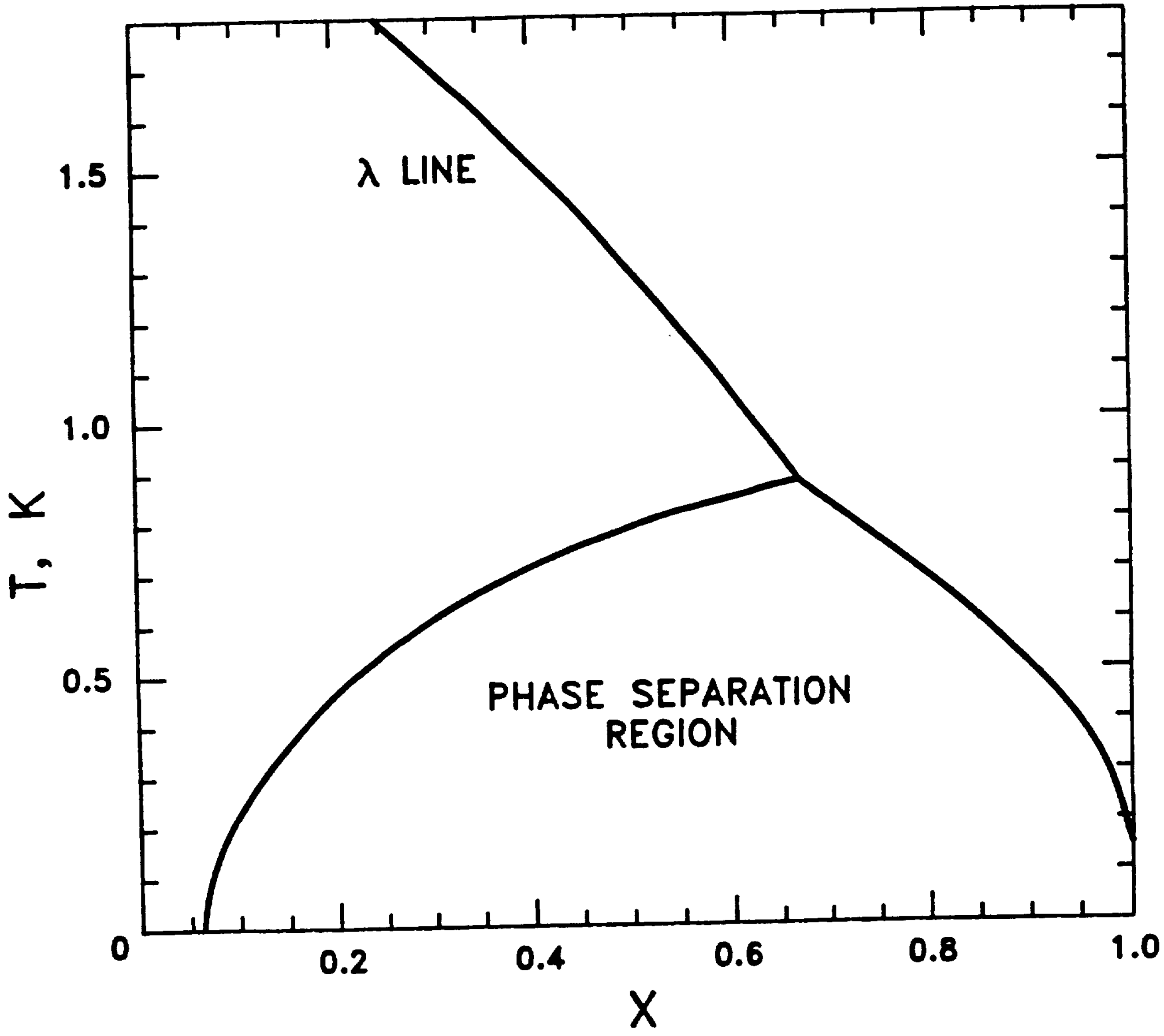


Figure 1 The ^{3}He/^{4}He Phase Separation Diagram
($x = n_3/n_3 + n_4$)

dilution cryocooler cycles. The ^{3}He circulation machine is the commercially available unit, and is almost universally used for laboratory studies in the 5 mK to 300 mK temperature range. The ^{4}He circulation, "Leiden" type dilution cryocooler has been developed in the laboratory. It can operate over the same temperature range as the ^{3}He circulation machine, but has not been commercially produced.

In the final section, a new dilution cryocooler cycle will be described. This machine will be called the "ACE, Inc." ^{4}He circulation, dilution cryocooler cycle. Even though it was specifically developed for zero gravity use, it can also operate in gravity, and is a possible replacement for the current commercial models.

The Solution Refrigerator. The solution refrigerator is discussed by Radebaugh in Chapter 11 of Walker's (1983) monograph on cryocoolers. The basic cycle is shown in Figure 2. The key to the device is availability of a permeable membrane (G) that will allow the free circulation of one component (A_1) by restricting the movement of the second component (A_2). The heating and cooling effects at the two membranes will be approximately

$$\dot{Q} = T\Delta S \tag{1}$$

As the solution refrigerator operates, there is a continuous injection of A_1 at the input and a removal of A_1 at the output. This means that if A_1 and A_2 are miscible, A_2 will take part in the mass transport from the input to output membrane. However, since the net mass flow of A_2 must be zero over

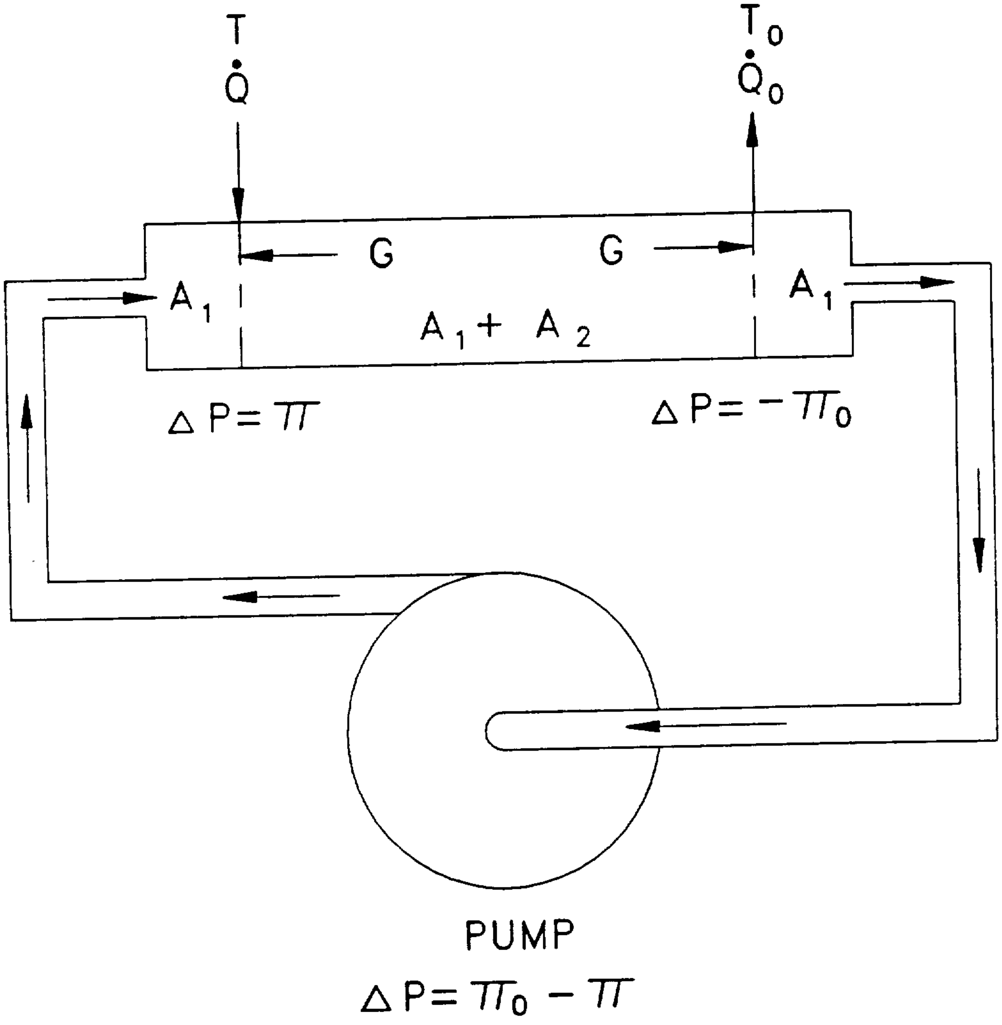


Figure 2 - Basic Solution Refrigerator Cycle.

time, there must be a countercurrent of A_2. This countercurrent will be by diffusion, through the moving mixture. The diffusion can be driven by:

i. concentration gradients,
ii. temperature gradients,
iii. osmotic pressure gradients, or
iv. a combination of the above.

If the circulating fluid is superfluid ^{4}He, and the stationary fluid is liquid ^{3}He, then the solution refrigerator is closely related to the dilution cryocooler. However, as stated above, the simple analysis is only applicable above the phase separation temperature (0.87 K). Therefore, we will restrict the liquid helium solution refrigerator to temperatures above 0.87 K.

Packed, fine insulating powders can separate the two isotopes easily. These superleaks transmit almost no ^{3}He, and can be used to purify ^{4}He. The superleaks are connected in series with a drift tube (T) as shown in Figure 3. A "fountain pump" or other circulator provides the pressure necessary to drive the superfluid through the device.

The ^{3}He concentration in the drift tube will change with temperature. Radebaugh (1967) has shown that the osmotic pressure in the drift tube will remain constant. This is true only for small relative velocities, however. For a discussion of cases where the mutual friction can become important, see deWaele et al. (1984). A chart of osmotic pressure vs. concentration and temperature is given in Figure 4.

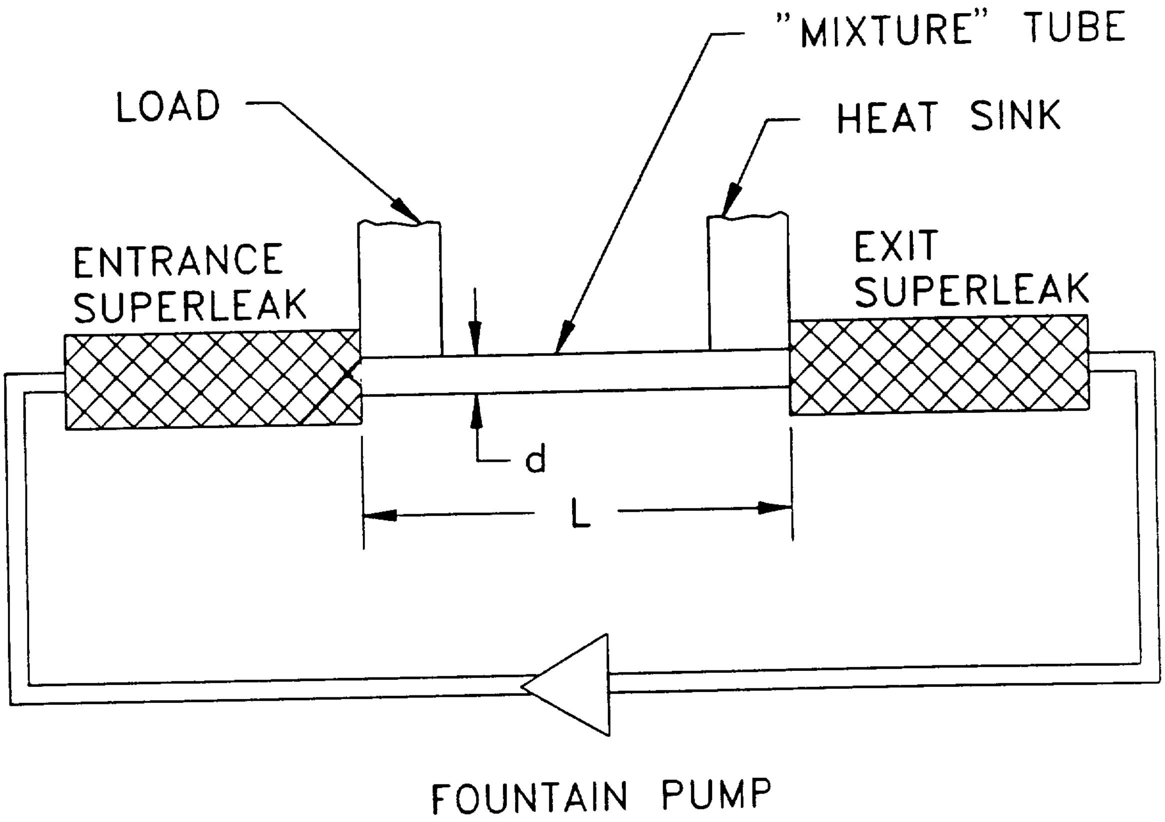


Figure 3 Solution Refrigerator Using Superleaks and He II Circulation.

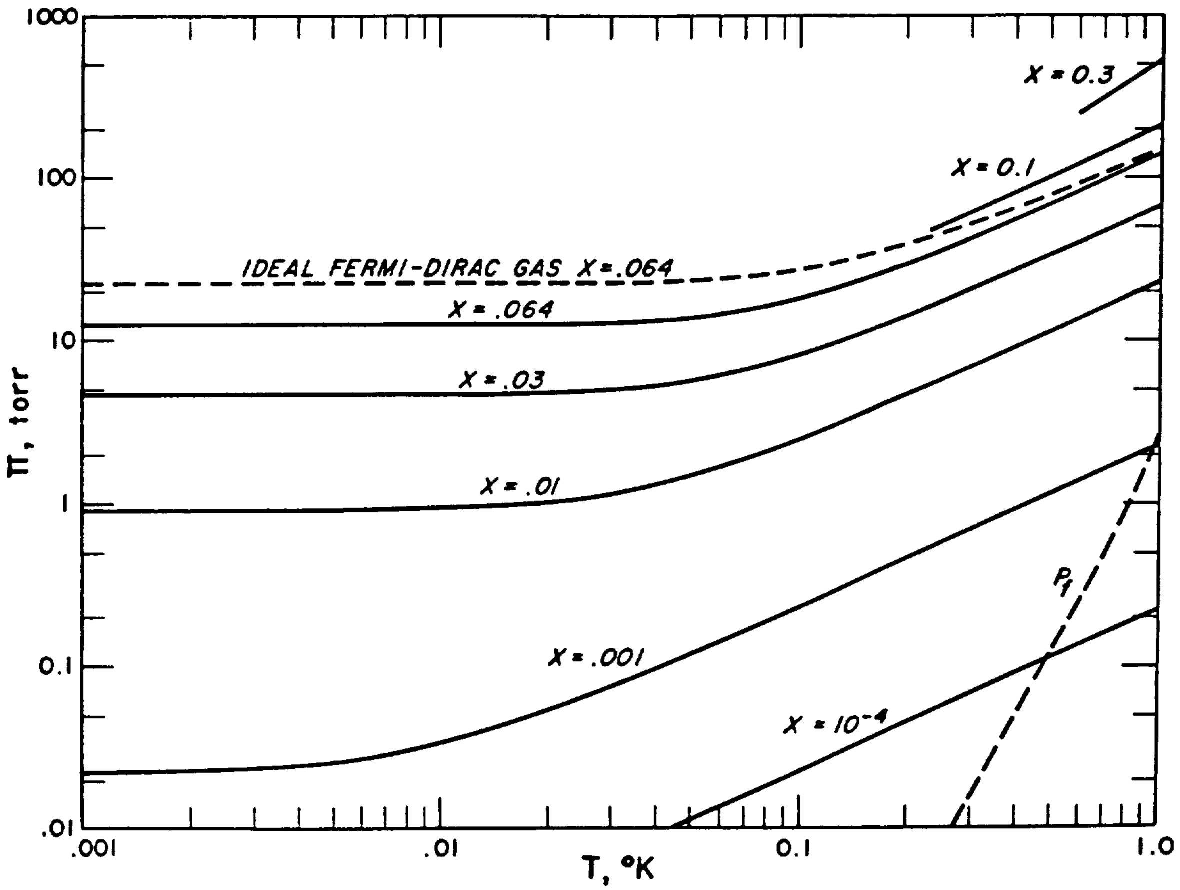


Figure 4 Osmotic Pressure as a Function of Temperature and Concentration

A demonstration calculation of a solution refrigerator will be given as an example. Assume end temperatures of 1 K and 1.5 K, and that the ^{3}He concentration at the 1 K end is 0.10. The osmotic pressure corresponding to T = 1 K and x = 0.1 is 208.6 Torr. The concentration corresponding to π = 208.6 Torr and T = 1.5 K is 0.064. Therefore:

$$\dot{Q}_1 = T_1 \Delta S_1 = (1)\ (3.247)\ \mathrm{J/mol} \qquad (2)$$

$$-\dot{Q}_2 = T_2 \Delta S_2 = (1.5)\ (3.315)\ \mathrm{J/mol} \qquad (3)$$

For a Carnot engine operating between the same two limits, we have

$$\dot{Q}_2 \,|_{\mathrm{Carnot}} = (T_2/T_1)\ \dot{Q}_1, \qquad (4)$$

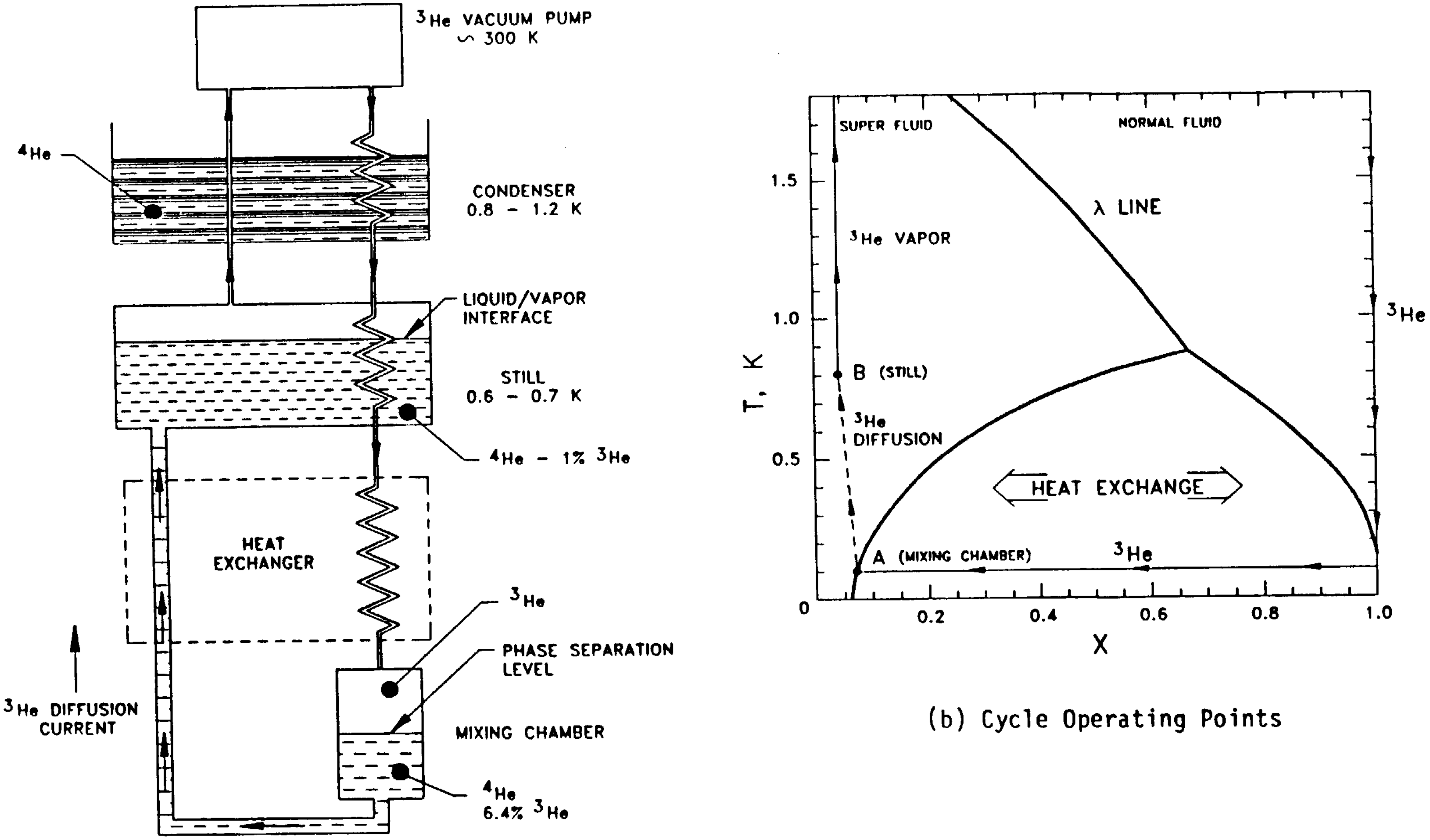
but $(1.5)(3.315) > (1.5/1)\ (1)\ (3.247)$
so the solution refrigerator is less efficient than a Carnot refrigerator, as we would expect. In the above we have used the tables given in Radebaugh (1967).

The ^{3}He Circulation Dilution Cryocooler. A detailed discussion of the conventional ^{3}He circulation dilution cryocooler is not necessary for the purposes of this report. For a complete discussion, the reader is referred to Lounaasma (1974), Radebaugh's chapter in Walker (1983), Betts (1976), or Richardson and Smith (1988). All these authors use the same basic techniques and only differ in small details. The theory of the ^{3}He dilution cryocooler is well established, and has been supported by over twenty years of laboratory experience.

The basic cycle of the ^{3}He circulation machine is illustrated in Figure 5(a). ^{3}He vapor is brought from room temperature and condensed to liquid in the condenser. The liquid is then cooled, first in the still, and then in the heat exchanger, before it is injected into the mixing chamber. The ^{3}He diffuses from the almost pure ^{3}He floating on the top of the mixing chamber into the dilute solution in the lower part of the mixing chamber. Below 0.1 K, the concentration of the dilute solution remains almost constant at 6.4% ^{3}He, independent of temperature. The "heat of mixing" of the ^{3}He into the dilute solution provides the cooling power of the refrigerator.

A continuous column of dilute solution connects the mixing chamber to the still. At the liquid interface, the ^{3}He is removed by evaporation. At temperatures below 0.7 K, almost pure ^{3}He is evaporated, with no ^{4}He removed in the vapor. Thus the ^{4}He can be seen as a fixed "mechanical vacuum", with the ^{3}He moving through it with no significant interaction. The dilute solution fills the other side of the heat exchanger, and the exchange of heat between the dilute solution and the incoming ^{3}He forms one of the most difficult technical problems in the design of the dilution cryocooler. A discussion of this problem is outside the purpose of this report, but is well covered in the previously cited literature.

The ^{3}He concentration in the dilute solution follows the constant osmotic pressure law. This was discussed in the section of the solution refrigerator. For a 6.4% ^{3}He concentration in the mixing chamber, we expect a 1% ^{3}He concentration in the still, for a still temperature of 0.7 K. Since there will be a deficit of ^{3}He in the still, due to evaporation, and an excess of ^{3}He in the mixing chamber due to inward diffusion; the ^{3}He will flow from the



(a) Cycle Schematic

(b) Cycle Operating Points

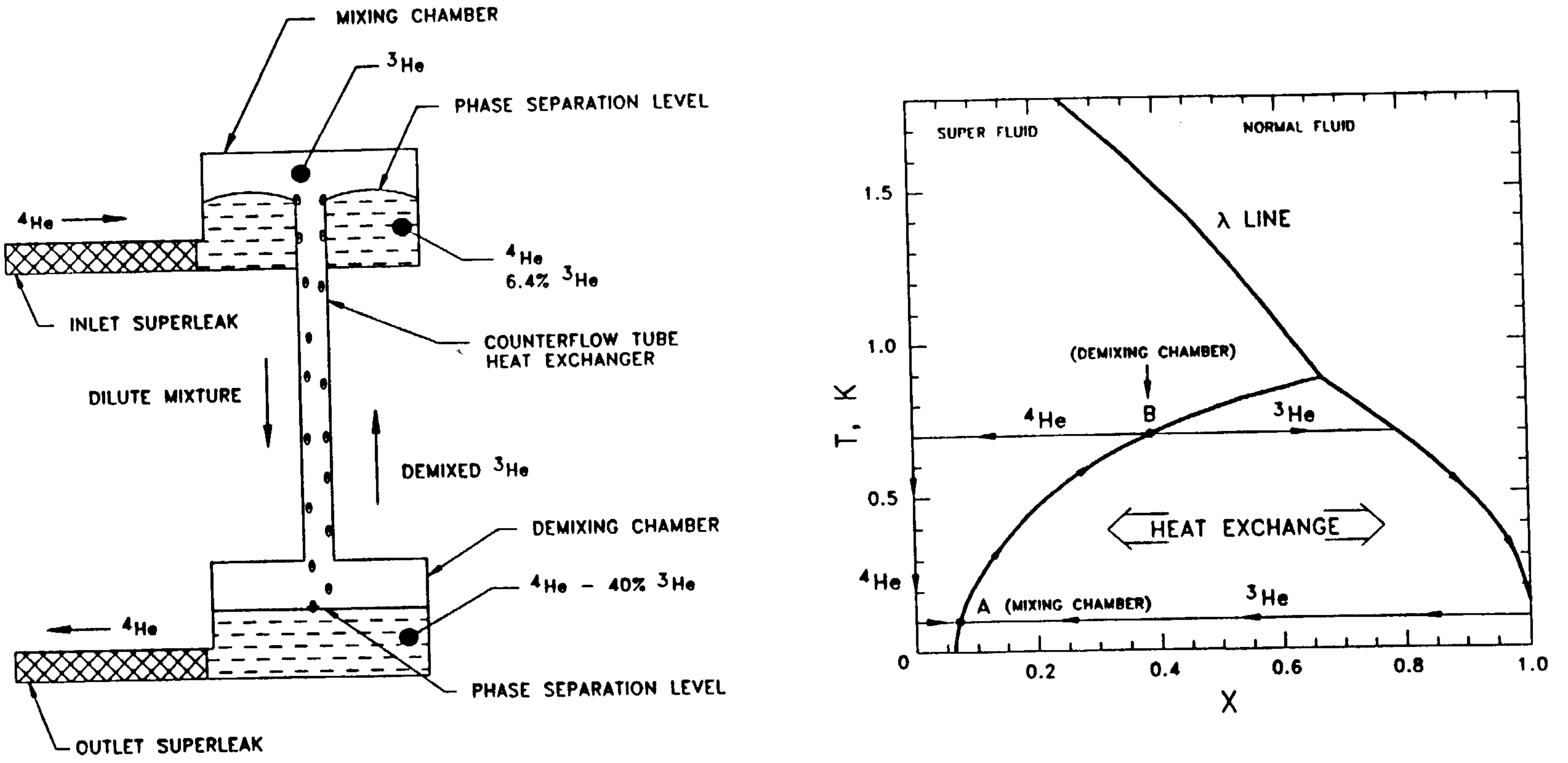
Figure 5 The ^{3}He Circulation Dilution Cryocooler mixing chamber to the still. This is a diffusion current, driven by the constant osmotic pressure requirement. The cycle is shown in Figure 5(b).

The cooling power of a ^{3}He circulation machine, with the flow of ^{3}He returning to the cryocooler shut off (non-continuous operation) is

$$\dot{Q}_m = 84\ n_3\ T_m^2 \qquad \text{J/sec} \tag{5}$$

Circulation of ^{3}He will reduce this cooling power, depending on the effectiveness of the heat exchanger. This can be used as a limiting value for comparison to other dilution cycles. Since the cooling power is directly proportional to the ^{3}He circulation rate, n_3, it is important to make this as large as possible. However, this is set by the capacity of the room temperature pump. Modest vacuum pumps will have a speed of 5 liter/sec., correspond to $n_3 = 30 \times 10^{-6}$ mole/sec. In order to get large flow rates, Roots blowers and other high capacity vacuum pumps are employed. Circulation rates of 2×10^{-3} mole/sec have been used, but these are extreme values due to the pumping problems.

The ^{4}He Circulation, "Leiden" Dilution Cryocooler. In the "Leiden" type dilution cryocoolers, liquid ^{4}He is circulated, rather than ^{3}He. The Leiden machine uses superleaks for injecting and withdrawing superfluid ^{4}He from the unit. A schematic of the cycle is shown in Figure 6(a). The system is arranged so that phase boundaries exist in both the mixing and the demixing chambers. The dilute solution in the mixing chamber is trapped below the "lip" of the counterflow tube. The ^{3}He floats on top of the dilute solution, and fills the counterflow tube down to the demixing chamber. In the demixing



(a) Cycle Schematic

(b) Cycle Operating Points

Figure 6 The "Leiden" Dilution Cryocooler chamber, the ^{3}He floats on top of the dilute mixture in the lower chamber. Pure ^{4}He is injected through the inlet superleak, and forms more dilute solution in the mixing chamber. This process is accompanied by cooling. As excess dilute solution is formed in the mixing chamber, it "pours" over the lip and falls through the counterflow tube as drops of dilute solution or as a sheet of dilute solution covering the walls of the tube. The cold dilute solution exchanges heat with the ^{3}He as the drops fall. The ^{4}He is removed from the tube by an exit superleak that is placed in the dilute solution in the demixing chamber. Heat is given off as the demixing takes place. This heat must be removed by an external cooler at a temperature below the phase separation point. This is typically done with a ^{3}He vapor cycle refrigerator.

The cycle is indicated on a phase diagram in Figure 6(b). The entire process in the counterflow tube is in phase equilibrium, so the state path is along the phase separation line. As the temperature changes, ^{3}He must diffuse into the falling drops to increase the concentration. As the ^{3}He rich solution increases in temperature, the ^{4}He fraction increases also. This means that the ^{3}He rich phase has an increasing density as we move down the tube. Therefore, there is no gravitational instability in this cycle.

One of the basic problems with the Leiden cycle machine is the outlet superleak. The superfluid ^{4}He is removed from a solution containing roughly 40% ^{3}He. Therefore, as the ^{4}He is removed, a relatively large amount of ^{3}He must be removed, and must "diffuse" away from the superleak. If ^{3}He builds up, it can form a blockage, and stop the superfluid ^{4}He removal. This is a limiting factor on the circulation rate of the machine.

The Leiden machine cooling power can be calculated from the ^{3}He circulation machine equation. For temperatures below 0.1 K, the ^{4}He rate can be written as:

$$n_4 = [(1.0 - 0.064)/0.064]n_3 = 14.6\ n_3 \tag{6}$$

Therefore, the maximum refrigeration rate according to Eqn. (5) will be:

$$\dot{Q}_m = 5.7\ n_4\ T^2_m \quad \text{J/sec} \tag{7}$$

It is much easier to get large ^{4}He circulation rates, as the entire circulation circuit can be maintained at superfluid temperatures, and the flow can be driven by a "fountain pump". Rates as high as 2 x 10^{-3} mole/sec are easily obtained using this method.

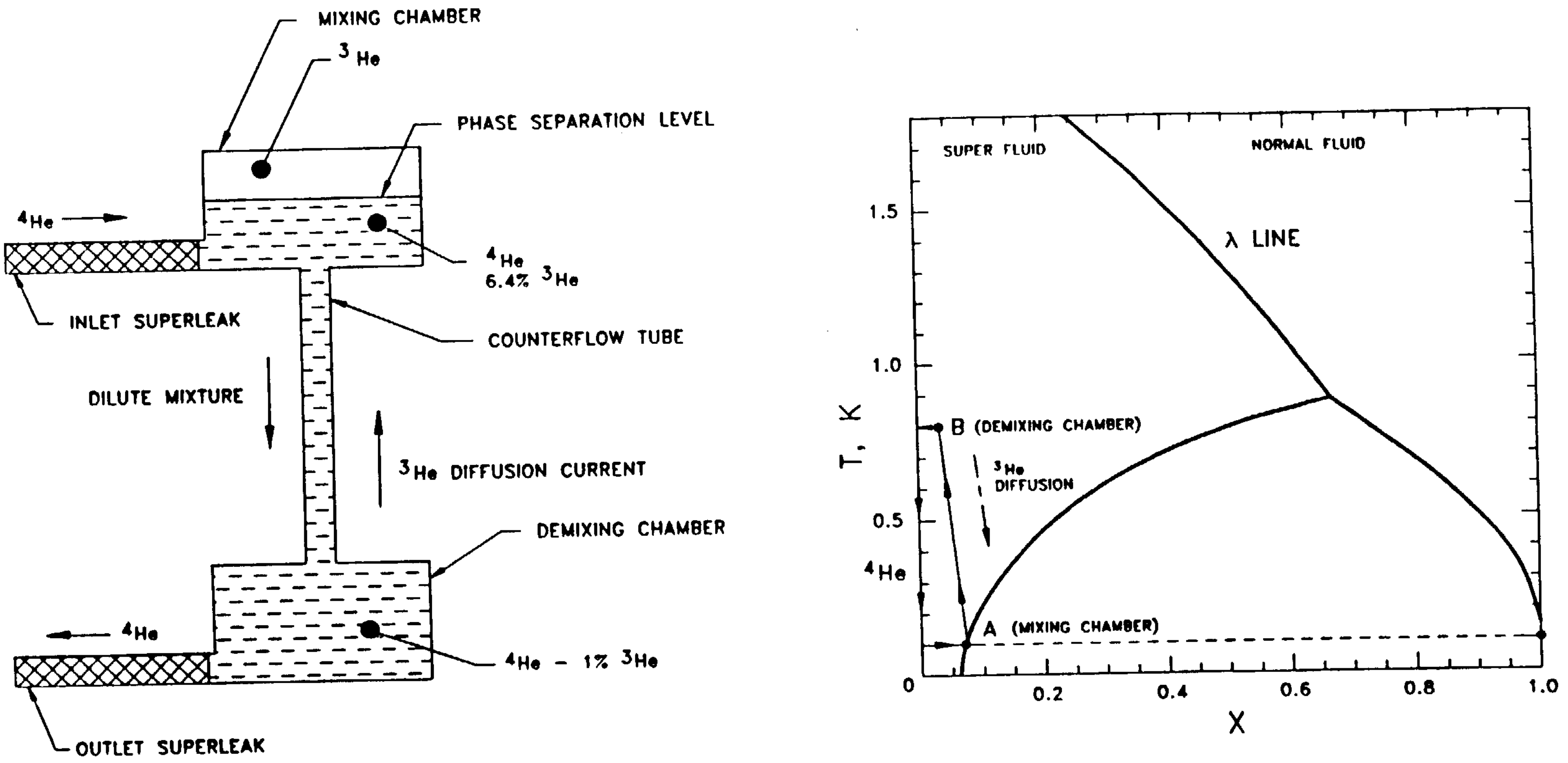
The main drawback to the Leiden machine is the precooler requirement. Since a ^{3}He vapor cycle is required, we have gained no real advantage over the ^{3}He circulation machine. All the external vacuum pumps are still required. The primary advantage in the Leiden cycle is the elimination of the recuperative heat exchangers in the low temperature section. This can lead to a considerable simplification in the design of the cryogenic section.

The Leiden dilution cryocooler is described in detail in the same references that were given for the ^{3}He circulation machine. A recent paper by Satoh, et al. (1987) contains a description of the best machine built to date. A base temperature of 3.4 mK was reached at a circulation rate of 3.5 x 10^{-3} mole/sec. This base temperature compares well with typical ^{3}He machines.

The ^{4}He Circulation, "ACE, Inc. Dilution Cryocooler". The schematic of the "ACE, Inc." cycle is shown in Figure 7(a). The cycle is similar to the Leiden cycle, as ^{4}He is circulated through superleaks. However, it is different because there is only one phase boundary, and that is in the mixing chamber. The principal differences between the Leiden and ACE, Inc. cycles can be seen on the phase diagram, Figure 7(b). Only the mixing chamber operating point is on the phase separation line. The rest of the dilute solution in the counterflow tube and the demixing chamber has sub-critical ^{3}He concentration. The relative concentrations of ^{3}He in the mixing and demixing chambers are set by the "constant osmotic pressure" requirement, as in the ^{3}He circulation cycle.

A substantial advantage of the ACE, Inc. cycle over the Leiden cycle is that the demixing chamber can operate at temperatures above the phase separation point. A higher demixing temperature will lower the overall cycle efficiency. However, if the requirement of a ^{3}He vapor cycle precooler can be eliminated, it can result in a much simpler machine.

The ^{3}He will be removed from the dilute solution in the demixing chamber. Therefore, an excess concentration of ^{3}He will exist there. This excess will diffuse up the counterflow tube, then replenish the ^{3}He being absorbed in the mixing chamber. This diffusion is similar to the diffusion that takes place between the mixing chamber and still of the ^{3}He circulation machine. It is in the opposite direction, however; and it moves against the mass transport velocity, v, of the dilute mixture. From the experiments of Satoh, et al. (1982) on vortex cryocoolers, we know that the ^{3}He will be swept out of the



(a) Cycle Schematic

(b) Cycle Operating Points

Figure 7 The "ACE, Inc." Dilution Cryocooler counterflow tube if the velocity exceeds the critical value. If we take the critical velocity to follow:

$$v_c = 10^{-6}/d \text{ m/sec.}$$

then: $n_4 \leq 2.8 \times 10^{-2}\ d$ (8)

where d is in meters and n_4 is in mole/sec. Therefore,

$$\dot{Q}_m \leq 0.16\ dT_m^{\ 2} \tag{9}$$

However, the counterflow tube also conducts heat. This is calculated from

$$\dot{Q}_c = k\ (A/L)\ \Delta T \tag{10}$$

Experimentally, the thermal conductivity values for solutions from 1.3% to 6.4%, lie in the k = 0.1 W/mK range. Also, $\Delta T = T_D - T_m \simeq T_D$, so we can write the available cooling power as:

$$\dot{Q}_a = \dot{Q}_m - \dot{Q}_c = 0.16\ d[T_m^{\ 2} - .49(d/L)\ T_o] \tag{11}$$

we can define a critical value when $\dot{Q}_a = 0$. The value of (d/L) corresponding to the critical value is:

$$\begin{aligned} (d/L)_c &= T_m^{\ 2}/.49T_D \\ &= 2.04 \times 10^{-2} \text{ for } T_m = 0.1 \text{ K and } T_D = 1 \text{ K} \end{aligned} \tag{12}$$

To insure that the conduction term is negligible, d/L should be less than one tenth $(d/L)_c$. Given this requirement, then the maximum cooling power is

$$\dot{Q}_a|_{max} = 0.16\ d\ T_m^2 \tag{13}$$

With a 4 mm tube, and $T_m = 0.1$ K, the maximum cooling power is 6.4 micro-watt. The drift tube length, L, is 2 meters.

The above analysis depends critically on the critical velocity equation. There is almost no data available on superflow at temperatures below 1 Kelvin. The temperature dependence is also unknown, except that theory indicates a constant value with temperature. Finally, the mutual friction between the superfluid and the ^{3}He is not well known. Castelijns, et al. (1984) has considered the effect of the ^{3}He velocity on the performance of the ^{3}He circulation dilution cryocooler. The velocity of ^{3}He in the ACE, Inc. cryocooler is not much larger than in the ^{3}He circulation cycle, so we suspect this effect is not important. If the critical velocity becomes the limiting element, the drift tube can be split into a number of parallel tubes having small diameters. This increases v_c for a given cross-sectional area.

Operation of Dilution Cryocoolers In Zero Gravity

In this section we will develop the theory of machines that will operate in zero gravity. One approach to the problem is to provide an artificial gravity. This could be done by rotation of the device (centrifugal forces) or by use of electric fields. The latter method has been demonstrated in models by the group at the Jet Propulsion Laboratory, Israelsson (1988). We have rejected this approach in this effort. We intend to design a machine for zero gravity operation without artificial gravity.

For the design studies we have selected the baselined specifications given in Table 1. The specifications are based on the Adiabatic Demagnetization Refrigerator (ADR) systems that have been developed for space based sensor systems and represent the current state of the art in detector technology. The three dilution cryocooler types that were presented earlier will be discussed in turn. The solution refrigerator does not depend on gravity in any way, so it will not be covered further.

Operation of a ^{3}He Circulation Dilution Cryocooler in Zero Gravity. The ^{3}He circulation dilution cryocooler, Figure 5(a), has three phase boundaries. These include:

i. The vapor/liquid interface in the ^{3}He condenser.

ii. The "dilute solution"/"concentrated solution" interface in the mixing chamber.

iii. The vapor/liquid interface in the still.

Each interface will be discussed in turn.

TABLE 1

Baseline Specifications for a Space Based ^{3}He/^{4}He Dilution Cryocooler

Operating Temperature	0.1 K
Operating Heat Load	10 μW
Heat Sink Temperature (Superfluid Helium Dewar)	1.5 K to 1.8 K

The vapor/liquid interface in the condenser is set by the thermal gradient in the condenser. Above the cold condenser section, the pressure is below that saturation pressure at the wall temperature. The tube is thus acting as a heat exchanger to cool the vapor. When the vapor reaches the condenser, the wall temperature falls rapidly, over a short section, to a value that is below the saturation temperature corresponding to the line pressure. Thus the fluid goes from single phase vapor to single phase, over pressurized liquid over a short distance. In addition, the tube has a small diameter so the fluid in the tube is roughly isothermal. Under these conditions gravity is not an important factor. The zero gravity heat transfer coefficients are probably different from the one g values, but not drastically so. We don't expect any free convective effects in such a confined space, so this indicates no important gravity effects. In conclusion, we do not expect the condenser to be a problem in a zero gravity ^{3}He circulation dilution cryocooler.

The next interface that we reach in the circuit around the refrigerator is the phase boundary in the mixing chamber. This boundary is where the actual cooling effect is produced, so in order to make use of the cooling power, we must make thermal contact to the phase boundary. The usual schematic of the ^{3}He circulation unit shows the ^{3}He being introduced into the concentrated phase, and indicates "diffusion" across the phase boundary. As a matter of fact, the ^{3}He is usually introduced into the dilute solution directly. This encourages stirring, and maintains concentration equilibrium in the mixing chamber. This experimental evidence supports the conclusion that a clear, defined phase interface is not a requirement for dilution cryocooler operation. The thermal contact requirement is the vital one.

The position of the phase boundary in the mixing chamber is set by the amount of ^{4}He in the system. To first order, the ^{4}He serves as a "mechanical vacuum". If the ^{4}He phase boundary in the "still" is fixed, then the position of the phase interface will be fixed, depending on the volumes of the parts. In this way we can insure that the still and connecting lines are full of dilute solution, and that the phase boundary is somewhere in the mixing chamber.

The phase arrangement in the dilute solution channel will be self correcting. The ^{3}He is being injected in the mixing chamber and removed in the still. Therefore, we expect the mixing chamber to be colder than the still with circulation on. Assuming such stable operation, now place a "blob" of pure ^{3}He in the dilute solution channel. The temperature in the channel will be somewhere between the still temperature, T_s, and the mixing chamber temperature, T_m. Therefore, the particular point should be somewhere between points A and B on the phase diagram, Figure 5(b). Now all these points are in the dilute phase, and are not in equilibrium with the concentrated phase. Therefore, we expect the "blob" to slowly "evaporate" into the dilute mixture and cease to exist. The only place where concentrated solution can exist is in the coldest part of the cryocooler; that is, in the mixing chamber.

The final phase boundary is in the still. Here, the problem is much more complex. In the still the dilute solution is a superfluid. This means that the walls of the still are coated with a superfluid film (Rollin film). This film can creep into the pumping lines and can contribute a substantial ^{4}He circulation to the cryocooler. This ^{4}He will reduce the cooling power of the machine, so some method of controlling the film is usually included in the still. In zero gravity the situation is much worse. The creeping ^{4}He film could reach throughout the cold section of the pumping lines and the ^{4}He level would be much higher than in one g operation. Therefore, the phase boundary in the still must be controlled for successful zero gravity operation.

A similar control problem with superfluid helium exists for dewars filled with He-II and operated in space. A device, called the superfluid porous plug has been developed that controls the superfluid. A device, based on similar principles can operate as a still phase separator (SPS). The SPS will be formed of a relatively high thermal conductivity material. This could take, the form of a plate perforated by small holes, or a pressed, sintered, porous metal disk. A schematic is shown in Figure 8. The passages in the SPS are represented by a uniform circular tube with diameter d. The matrix is represented by the disk, having an effective thermal conductivity, k. The disk will have n passages per unit area, and the effective length of the passages is L.

A qualitative description of the device follows. The dilute solution, at a temperature of 0.6 K to 0.8 K is on the upstream side to the plug. The downstream side is connected to a pumping line, and the pressure on the downstream side is reduced. Since it is a superfluid, a certain portion of the dilute solution will leak through the plug. The ^{3}He in the dilute solution will evaporate and the liquid will cool. A fountain pressure,

$$P_f = \rho S \, \Delta T \tag{14}$$

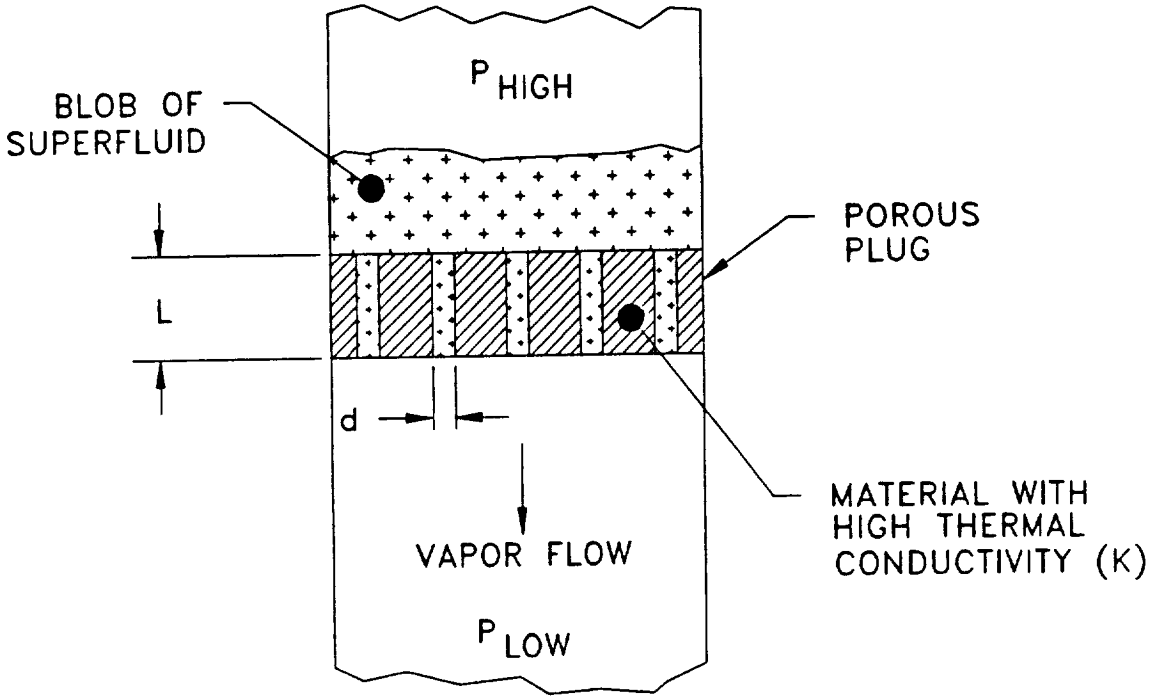


Figure 8 Schematic Diagram of the Superfluid Phase Separator (SPS). Surface tension in the pores prevents the liquid from escaping from the high pressure area P_{High} to the downstream low pressure P_{Low}.

will be generated that tends to drive the superfluid towards the hotter end of the plug; that is, back into the SPS. In steady state, the downstream end of the plug will be slightly colder, due to the ^{3}He evaporation. Heat will be removed from the dilute solution on the upstream end, and transmitted through the SPS to the evaporating interface.

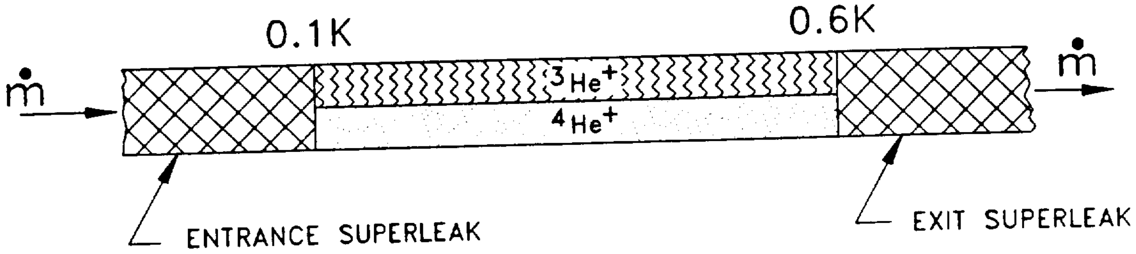
The holes in the SPS should have a small diameter, so that the surface tension force

$$P_{st} = \frac{2\sigma}{d} \tag{15}$$

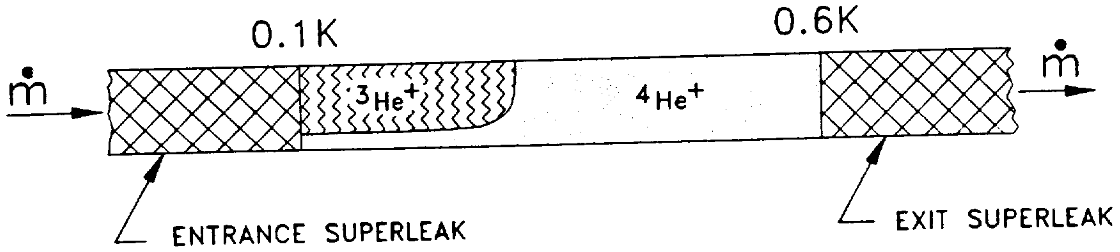
is relatively large. This will assist in the definition of the evaporation interface. However, the holes should be large enough to allow the ^{3}He to pass through with a relatively small pressure drop. According to the mechanical vacuum model, we can treat the ^{3}He as a vapor, except that we replace the free mass, m_3, by an effective mass m_3^*. For stable operation, the sum of the surface tension pressure and the fountain pressure should be much greater than the pressure drop of the ^{3}He passing through the SPS.

This device is related to the normal fluid phase separator that was developed by Alabama Cryogenic Engineering, Inc. in an earlier research effort.

Zero-Gravity Operation of the "Leiden" Dilution Cryocooler. According to our analysis performed during this research program, the "Leiden" cycle is not applicable in zero gravity. To illustrate, consider the situation shown in Figure 9(a). We assume that a solution type refrigerator device is filled



a) "Unstable" Phase Arrangement



b) "Stable" Phase Arrangement

Figure 9 Two Possible Arrangement of Phases in a Zero-Gravity Solution Refrigerator.

with $^3He/^4He$ mixture, and the end temperatures are set to the values shown. Is the indicated phase arrangement stable? There are two separate requirements:

i. The concentrations along the phase boundary must following the phase separation line.

ii. The osmotic pressure in the dilute solution must be constant.

Reference to the property tables indicates that these two conditions cannot be met at the same time. An additional indication that this is not an equilibrium state is a calculation of the cooling power with circulation of ^3He through the superleaks. We find that Q_{out} at 0.6 K is less than Q_{in} at 0.1 K. Thus, the second law is violated.

The solution to the problem is shown in Figure 9(b). An internal "convection" will take place, driven by the osmotic pressure. The ^3He will thus collect at the cold end of the tube, and the dilute solution will fill the remaining space. Therefore, the "Leiden" machine becomes the ACE, Inc. cycle. We conclude that the "Leiden" cycle is only possible in gravity, and is not a candidate for zero gravity operation.

Zero-Gravity Operation of the ACE, Inc. Dilution Cryocooler. The ACE, Inc. cycle avoids the problems of the Leiden cycle by having a phase boundary only in the mixing chamber. Since this is the coldest region in the unit, the ^3He phase can exist there stably at all temperatures. The arrangement of the phases is similar to the ^3He circulation dilution cryocooler. To simplify things, the amount of ^3He in the active region could be reduced, so that at the lowest temperatures there would be not concentrated ^{3}He phase. This is possible, but it will reduce the cooling power at temperature higher than 0.1 K. For maximum refrigeration power there should be enough ^{3}He to provide a phase separation at a relatively high temperature. This will speed up the cooldown process, and insure that the full cooling power is available at the lowest temperatures.

Since the ACE, Inc. cycle is new, the question of testing for zero gravity arises. As with all the dilution cycles there is a strong effect of gravity. The pure ^{3}He is much lighter than the dilute solution, so it tends to raise to the highest point in the machine. In addition, if the ^{3}He concentration changes along the counterflow tube, then the density also will change. A "gravitational instability" of this type must be suppressed in the ^{3}He circulation cycle. This is usually done by adding a "U-Tube" trap at the exit of the still.

For testing in gravity, the ACE, Inc. unit must be in one of two positions. Either the mixing chamber must be higher than the demixing chamber or they must be at the same height (horizontal operation). If the demixing chamber is above, a gravitational instability will occur, and the unit cannot operate stably.

TEST FACILITY

The test facility is comprised of several components. These components include; gas handling system, cryostat and instrumentation. A description of each component is included in the following sections.

Design

A major concern in the overall design is to ensure long term leak-free operation. Welded stainless steel construction was used wherever feasible. If welding was impractical, components were brazed, as when joining Cu to stainless steel. The use of PbSn solder was limited to electrical wiring.

Gas Handling System. This system is used to contain supplies of ^{3}He gas and ^{3}He-^{4}He mixture and to move them around where needed. As the cost of ^{3}He is approximately $150 per standard liter there is also a financial incentive for keeping the system leak tight. Figure 10 contains a schematic of the system. There are two gas panels, each panel controls the flow from a separate reservoir of ^{3}He. The two panels are connected together through a Metal Bellows Corporation model MBC-135 bellows pump, so that ^{3}He can be moved between reservoirs.

The valves on the gas panels are Nupro model SS-4D4L-V51 packless metal diaphragm valves with VCR fittings. When a valve fails, usually from an overtightened seat, it can be repaired and reinstalled without having to cut out any welded sections. We use Ag plated Ni gaskets and have had no trouble with the VCR fittings.

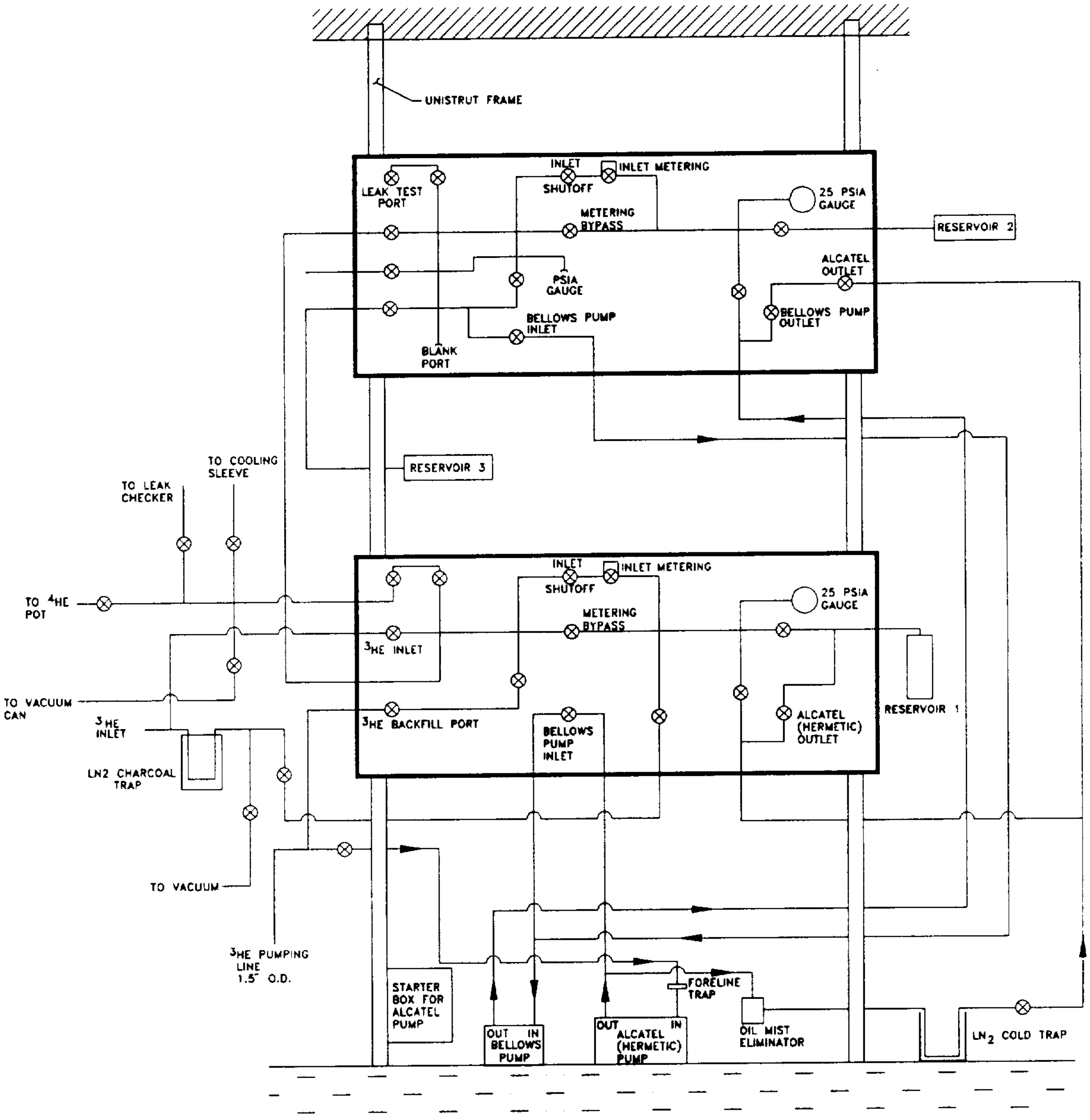


Figure 10 Gas Handling System

Gas panel No. 1 is used to control the flow of pure ^{3}He or ^{3}He-^{4}He mixture being pumped on. An Alcatel model 2033H hermetically sealed two-stage rotary pump is used to pump on the ^{3}He pot. During the latter part of the program, a failure in the 2033H pump required that it be temporarily replaced by a similar, smaller model (the Alcatel 2012AH). This pump was large enough to adequately serve as an emergency replacement. The pumps are operated at 6 psia or less for two reasons. If an Alcatel pump is operated above 7.3 psia, some leakage develops in the seals. Also, if a leak does develop, say for example in the reservoir, air leaks into the system and the ^{3}He does not escape. The resulting mixture can be purified by running it through a 4 K bath and freezing out all contaminants. Operating above atmospheric pressure would result in the ^{3}He venting to the atmosphere in the event of a leak.

The pumping line on the inlet of the Alcatel pump is a 1.5" O.D. stainless tube. There is a 1.5" high vacuum valve is on the inlet and outlet of the pump, to isolate it when performing maintenance or repairs. The demountable connections use conflats, to prevent unintended removal possible when using quick flange type fittings. The pumping system is provided with a foreline trap on the inlet and an oil mist eliminator filter and LN_2 trap on the outlet.

Gas panel No. 2 is used to meter quantities of ^{3}He and ^{3}He-^{4}He mixture into the dilution test cell. Only small amounts of ^{3}He are consumed per run. The resulting ^{3}He - ^{4}He mixture is allowed to vent to the atmosphere in the case of the ^{4}He circulation cryocooler as the dilution test cell warms up. ^{3}He or ^{3}He-^{4}He mixture is thus loaded into the test cell by the pressure differential between reservoir No. 2 and the test cell; loading occurs with the cell at a temperature of 1.0 K. Pressure sensors were installed on each of the gas panels. Both panels have one absolute pressure sensor (0-25 psia, Setra model 204).

Cryostat. The cryostat is designed to fit inside a 7.5" I.D. by 42" deep magnet dewar. A 3" thick Al hex flange mounts on top of the dewar, providing additional feedthroughs. A relief valve, cracking pressure set to 1 psig, is mounted on this flange along with a 3/4" ball valve. The cryostat mounts on top of the hex flange. An illustration of the cryostat is shown in Figure 11.

There are three main pumping lines, one each for the vacuum can, ^{4}He pot and ^{3}He pot, running through the top flange. Both lines for the helium pots are 1.5" O.D., the vacuum can pumping line is 2" OD. A hermetic 32 pin feedthrough is mounted onto the tee of the vacuum can line. Instrumentation leads are run through this line, avoiding cold feedthroughs. The pumping lines are graded inside the dewar; starting at 1.5" O.D. at the top flange, they are reduced to .75" O.D. on top of the vacuum can lid. As shown in Figure 11, the lines are offset through 45^0 ell fittings, forming a light trap to keep room temperature thermal radiation from impinging directly into the liquid pots and the vacuum can. Additional feedthroughs on the top flange are through Cajon Ultra-Torr fittings. The liquid helium level detector, ^{3}He filling capillary and liquid helium transfer line run through these fittings. Unused ports are blanked off with brass plugs. Four Al radiation shields are anchored to the pumping lines keep thermal radiation from the top flange from impinging on the liquid helium in the dewar.

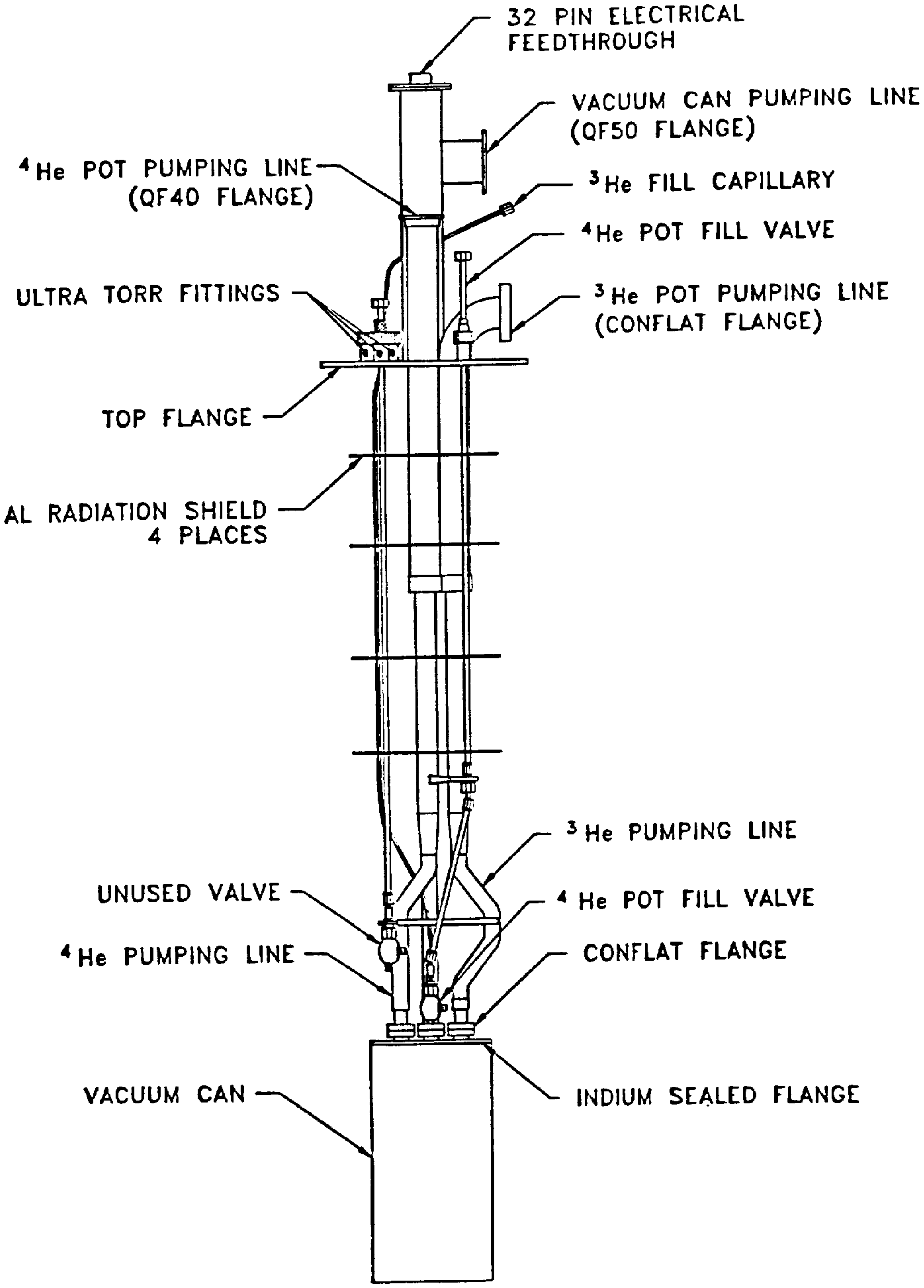


Figure 11 Schematic Layout of the Cryostat

The liquid pots are insulated from the 4 K bath by a vacuum can. Its dimensions are 6" O.D. by 11.5" high. Figure 12 shows the configuration inside the vacuum can. Copper cold fingers are welded into the vacuum can lid to provide 4 K heat sinks (not shown). The ^{3}He and ^{4}He pumping lines, 3/4" O.D. by .012" wall thickness, are attached by conflat flanges to the top of the vacuum can lid and to the top of the ^{4}He pot. The ^{3}He pumping line runs through the ^{4}He pot and out the bottom, where it is brazed in place. The ^{3}He pot connects to the pumping line with a conflat flange. Conflats and VCR fittings are welded to the top and bottom of the ^{4}He pot. The top and bottom of the ^{4}He pot are stainless steel plates, which have been brazed to the copper pot wall. These joints have been very robust, and no leaks have developed in the course of our work.

Both helium pots were fabricated with of 101 alloy Cu. The ^{4}He pot was built in three pieces; the top, bottom and body are separate. After welding the fittings in place in the top and bottom, the stainless steel lids were brazed in place to the body. The ^{3}He pot has a lid, into which was brazed a .75" O.D. stainless steel pumping line, and a chamber. The lid and chamber are sealed with In. The bottom of the chamber has tapped mounting holes for attaching and thermally anchoring the test cell to the ^{3}He pot. There have been no leaks associated with the ^{3}He pot and pumping line.

Instrumentation. Several thermometers are used when running the cryostat. The ^{4}He pot temperature can be regulated with a Lake Shore Cryotronics model DRC-81C temperature controller. Temperature measurements were made with calibrated Germanium thermometers manufactured by LakeShore Cryotronics, Inc. Three model GR-200A-50 thermometers were used throughout

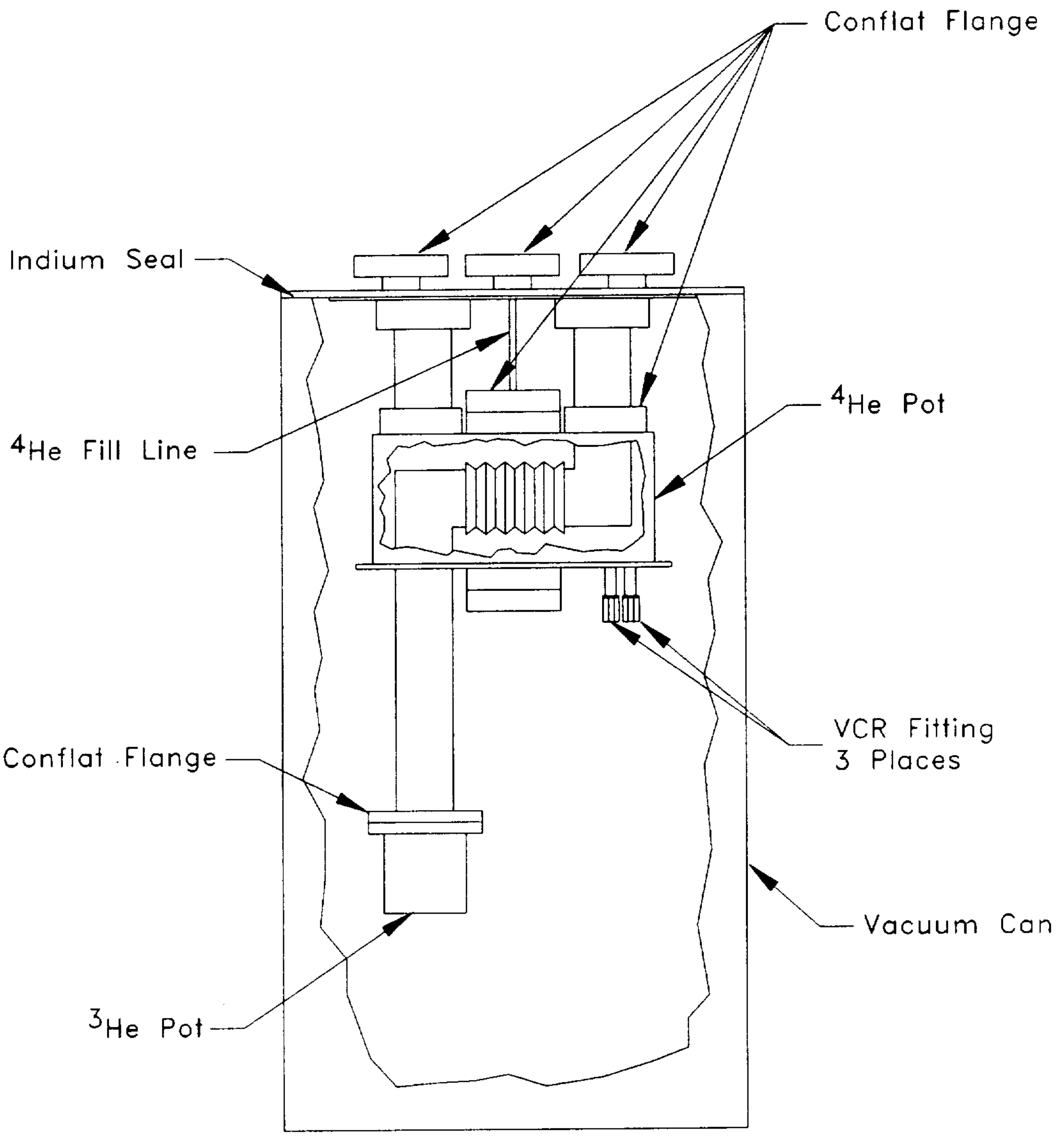


Figure 12 Configuration of ^{4}He Pot and ^{3}He Pot

the experimental program. These calibrated devices provide an experimental accuracy of approximately ± 1 mK over their entire calibrated range of operation. A mounting through hole was drilled into OFE copper, and the resistor was then inserted with a coating of vacuum grease, to insure a good thermal contact. The leads of each resistor were also carefully heat sunk for a length of ~ 50 cm at the resistor mounting location.

Wiring throughout the cryostat was done with 0.005" diameter Manganin wire. This choice of material results in very small heat leaks due to thermal conduction along the wires length. The lead wire had a typical electrical resistance of 11 ohm/ft.

At each thermal station, these lead wires were carefully heat sunk. This included heat sinking of the wires at the helium both temperature (4.2 K); at the ^{4}He pot temperature (1.0 K), and at the ^{3}He pot temperature (0.5 K - 0.3 K). Temperature sensors are read with a Biomagnetics Technology Inc. model 1000 conductance bridge. The excitation voltage is 100 μV. At this excitation level, sensor self heating has not been observed. All conductance measurements are made using the 4-wire method. The AC excitation voltage of the bridge eliminates contributions from thermal emfs.

OPERATION

The basic operation of the test facility is considered during leak detection and cooldown procedures.

Leak Detection

The most sensitive way to leak check the cryostat is with it mounted in the dewar. With the vacuum can in place (the indium seal having been previously leak checked), the ^{3}He pot, ^{4}He pot and vacuum can are evacuated with the high vacuum system. The leak detector is hooked to the ^{4}He pot and a small quantity of helium gas is admitted to the vacuum can. If the leak rate does not change over a period of 15 minutes, the ^{4}He pot is leak free. The leak detector is connected to the ^{3}He pot and helium is admitted to the ^{4}He pot. The entire ^{3}He pumping line is thus leak checked.

Having the vacuum can evacuated is essential for leak checking the ^{4}He circulation dilution test cell. In the test cell there are two superleaks in series, resulting in a very large flow impedance. During leak testing, the leak detector also pumps on the ^{3}He fill capillary. By evacuating the vacuum can, much lower background signal is present in the leak detector when a leak is present. Adding helium gas to the vacuum would then produce a noticeable increase in the leak detector signal. Unfortunately, this method does not pinpoint the leak location so further work is required. However, it is the best way to determine if a leak is present.

Cooldown

If no leaks are found in the cryostat, it is pre-cooled with LN_2. The helium gas in the vacuum can from the leak test remains and acts as a heat exchange medium. Once the ^{4}He pot temperature, as read from the temperature controller, is near 80 K, the remaining LN_2 is blown out with helium gas. The ^{4}He pot is pressurized with helium to about 4 psig and the fill valve is then left open. We then proceed to transfer LHe in the normal manner. When the ^{4}He pot temperature reads < 5 K, the exchange gas is pumped out. The vacuum can is pumped out overnight to ensure a good quality vacuum is attained.

DATA AND RESULTS

In the original design for the ^{3}He circulation dilution cryocooler, a vortex cooler was to be used to precool the incoming ^{3}He and to cool the dilution flow circuit to 1 K. Due to very good performance of our ^{4}He pot, which reached temperatures as low as 0.95 K, the vortex cooler system was not needed; the ^{4}He pot served very well as a precooler. However, a vortex cooler system was developed and tested, since it would be an important element in a system with a lower performance ^{4}He pot. A brief description of the operation of a vortex cooler system will now be presented, along with a summary of the ACE, Inc. vortex cooler design parameters and data showing the performance of this device.

Vortex Cooler Tests

The concept of forcing superfluid helium through a tightly packed solid powder to achieve cooling was first proposed by Kapitza in 1941. In 1967, Olijhoek performed the first experiments to verify the feasibility of this idea. The experiments were repeated by Staas and Severijns (1969), who named this type of device the vortex cooler. Other work followed (Olijhoek et al., 1973, 1974 and Satoh et al., 1982, 1983), but the device has not gained wide spread use simply because its operating temperature range, 0.7 - 2.2 K, can be covered easily in terrestrial laboratories by more standard means, such as with ^{3}He evaporative refrigeration. However, the vortex cooler has the unique advantage over other cooling cycles that it does not involve a liquid-vapor phase separation; it is therefore ideally suited for zero gravity work.

In Figure 13 a schematic diagram of a vortex cooler is given. Superfluid helium is forced to flow through a superleak made of very fine packed powder. According to the two fluid model, the super component of the fluid is inviscid and will pass through the superleak while the normal component will not. Driving superfluid through the superleak thus should result in lowering the entropy of the fluid in the cooling chamber; a cooling effect is observed. Excitations are swept away from the cooling chamber by the flow through the small diameter exit capillary. Cooling of the chamber at the end of the superleak is thus achieved.

In Figure 14 a drawing of the finalized ACE, Inc. vortex cooler is show. This figure is a cross-sectional cutaway representation; all of the parts used to assemble the vortex cooler have cylindrical symmetry.

Both the top and bottom plugs were made from OFE copper to insure low thermal gradients across them. VCR detachable fittings were installed at the fluid inlet and outlet to allow easy replacement of capillaries, vortex coolers, or heat exchangers. The superleak consisted of a 1/4" diameter stainless steel tube with 0.010" wall thickness. This tube was 1 3/4" long and packed with jeweler's rouge (iron oxide) powder. The vortex cooler top plug was attached to a mini-conflat flange that fastened to the underside of the ^{4}He pot. Superfluid helium could pass through the flange into a dead-end chamber in the top plug to assure that the top plug is adequately heat sunk to the ^{4}He pot. A tapped hole in the bottom of the bottom plug allowed screw attachment of heaters or other devices to the bottom of the vortex cooler. A 12" long, 0.028" I.D. stainless steel capillary served as the inlet to the vortex cooler.

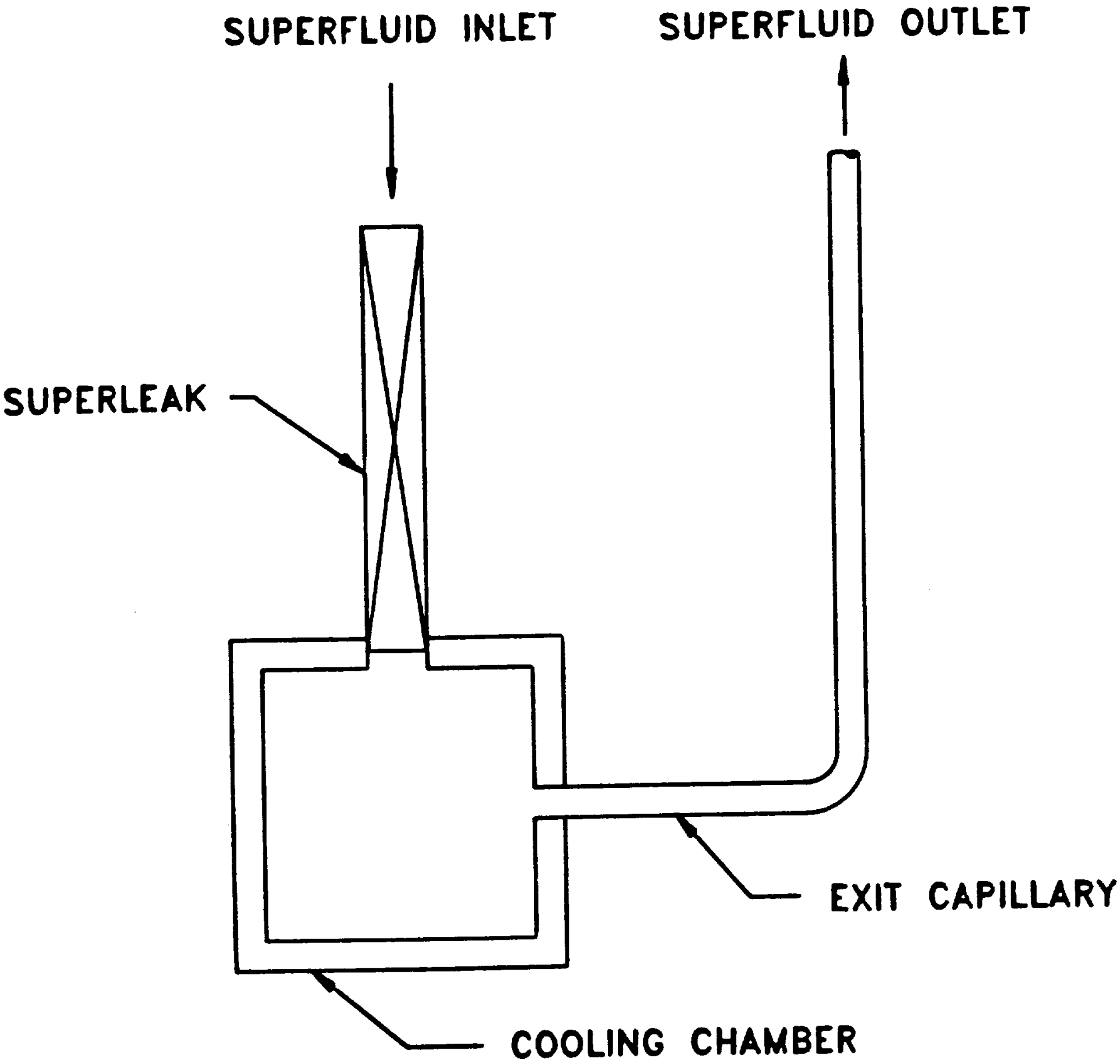


Figure 13 Schematic Representation of a Vortex Cooler

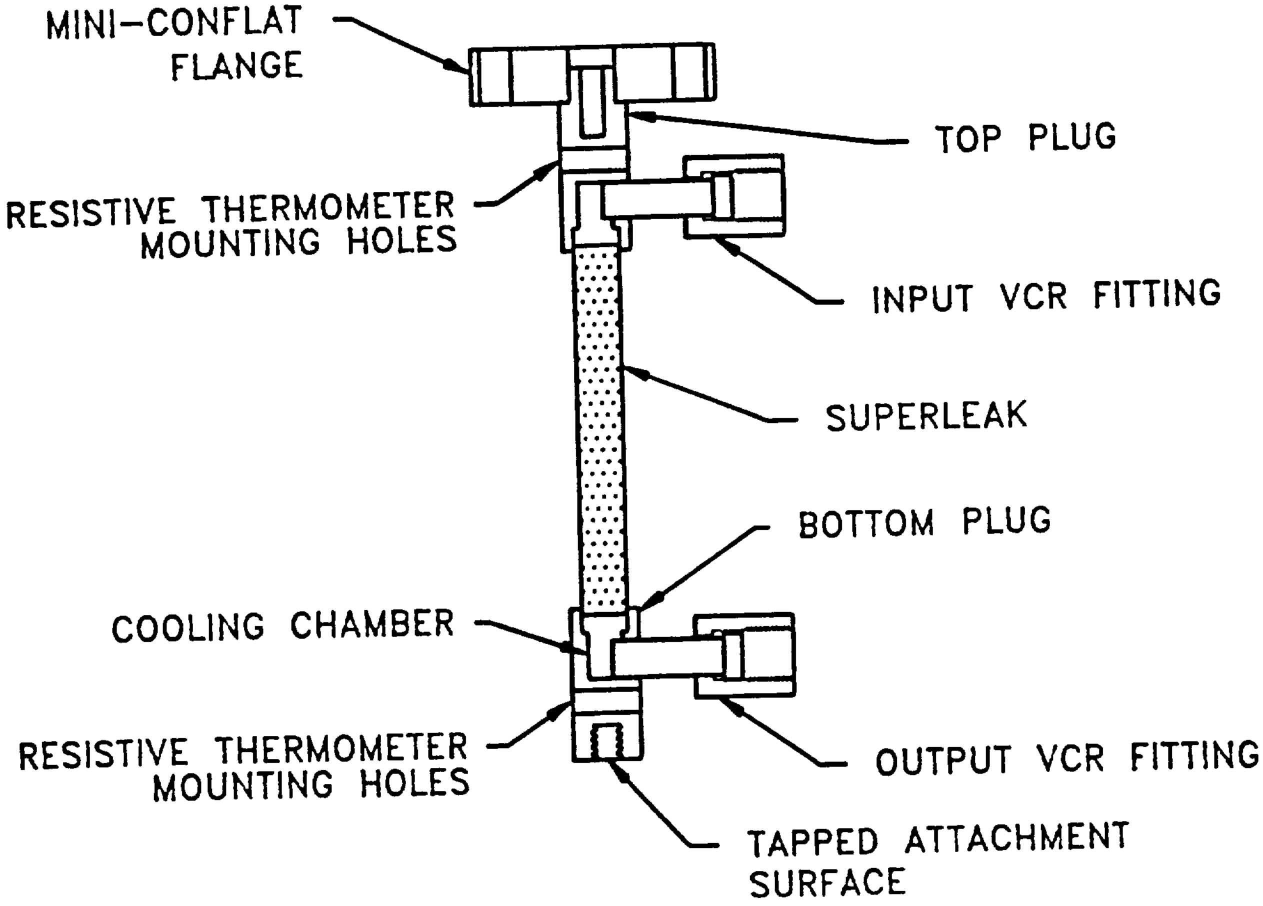


Figure 14 Scale Drawing of Finalized ACE, Inc. Vortex Cooler Design

In Figure 15 a drawing of the fountain pump is shown. It is structurally very similar to the vortex cooler previously described. In the fountain pump, the top plug and mini-conflat flange are completely drilled through, allowing superfluid to flow from the ^{4}He pot directly into the fountain pump superleak. A heater to run the pump was screwed to the fountain pump bottom. The fountain pump superleak was made from the same materials as the vortex cooler superleak, but was 2 1/4" long. A 12" long, 0.028" I.D. stainless steel capillary exited the fountain pump. For an introduction to the operation of fountain pumps, see Guenin and Hess (1980).

Figure 16 shows a cross-sectional view of the ^{4}He pot coiled heat exchanger. Fluid from the fountain pump is admitted to the coils in the exchanger, which are bathed in superfluid from the ^{4}He pot. After traveling through these coils, the fluid is cooled from the temperature at the exit of the fountain pump back down to the ^{4}He pot temperature. The heat exchanger coil consists of a 5 foot length of 1/16" diameter copper tubing. Although they are not shown in Figure 16, 1/8" VCR fittings are used for connection to the vortex cooler and fountain pump capillaries. In Figure 17 a schematic view of the vortex cooler/fountain pump/heat exchanger system is shown.

Figure 18 shows the temperature obtained by the vortex cooler as a function of input power to the fountain pump. The vortex cooler reached a minimum temperature near 0.8 K and had a power handling capacity of ~ 0.7 mW at 1.0 K. Figure 19 shows the equilibrium temperature of the vortex cooler as a function of load heat input, with a fixed fountain pump power input of 84 mW.

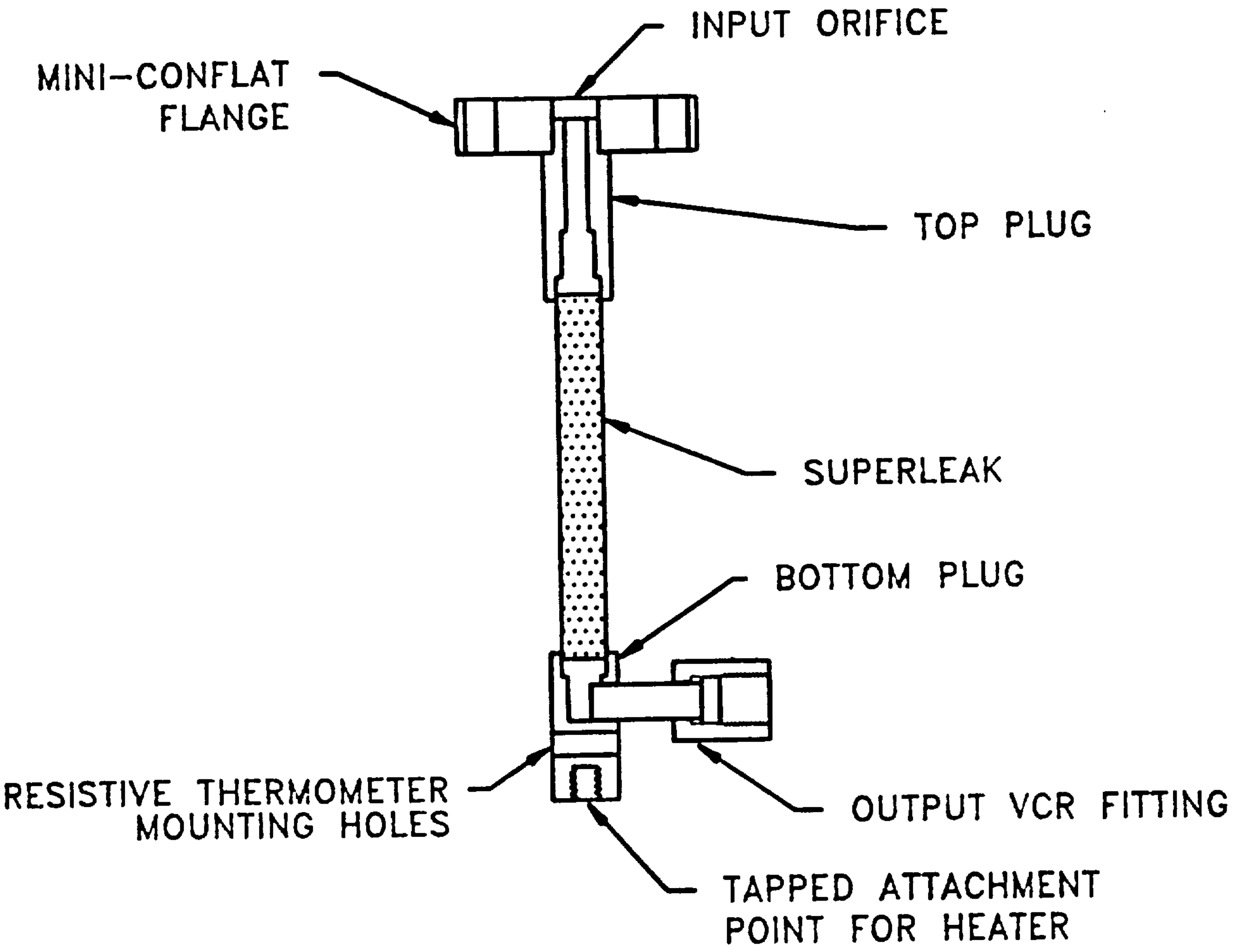


Figure 15 Scale Drawing of Finalized ACE, Inc. Fountain Pump

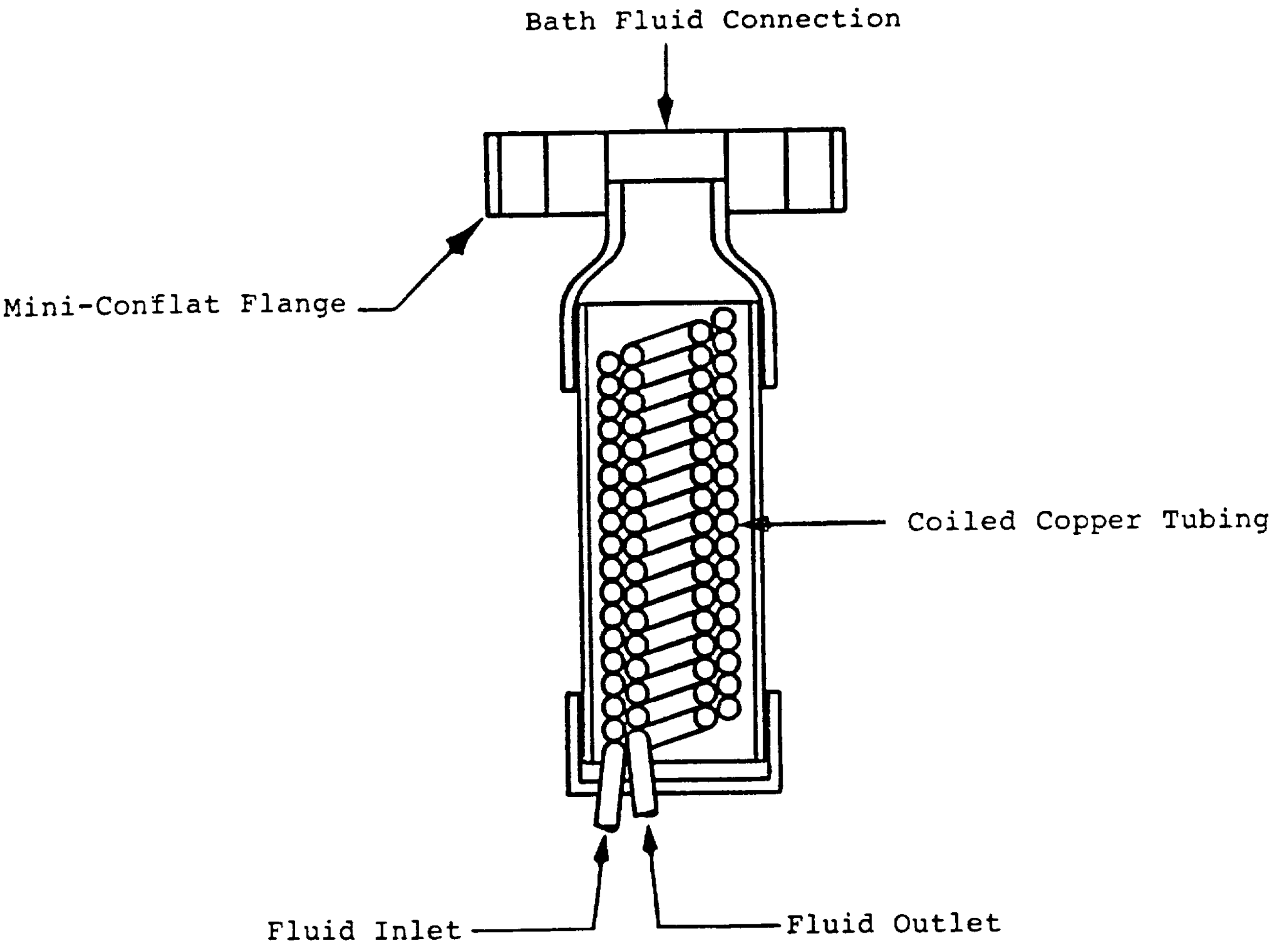


Figure 16 4He Pot Coiled Heat Exchanger

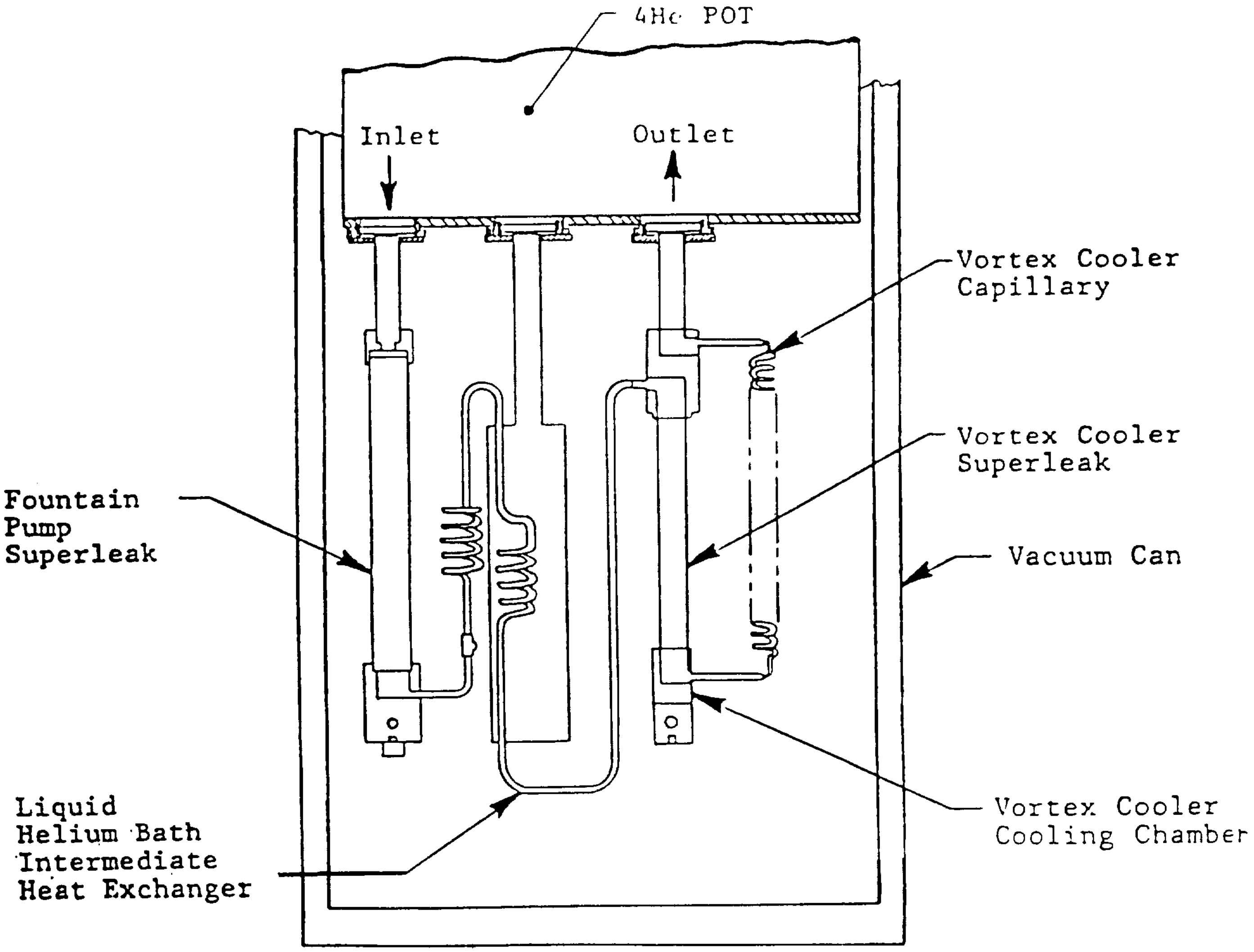


Figure 17 Fountain Pump/Vortex Cooler Configuration

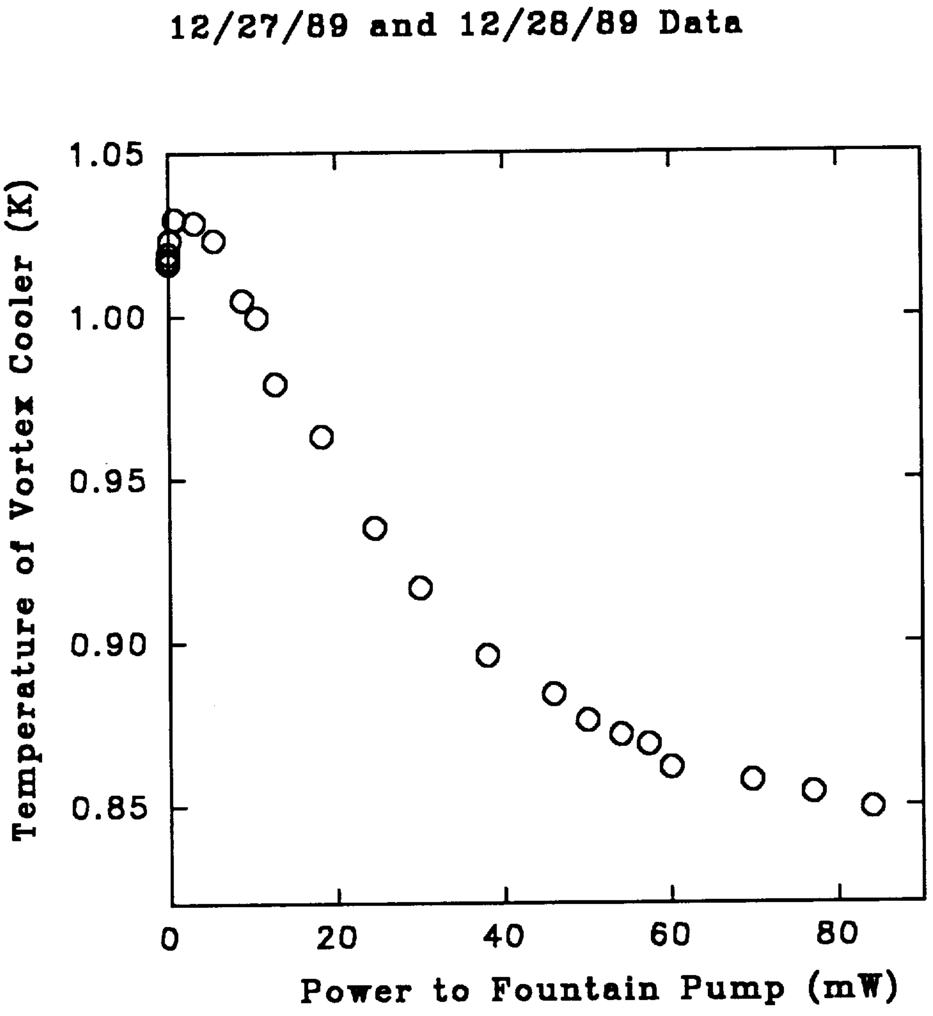


Figure 18 Temperature of Vortex Cooler vs. Power Input to The Fountain Pump

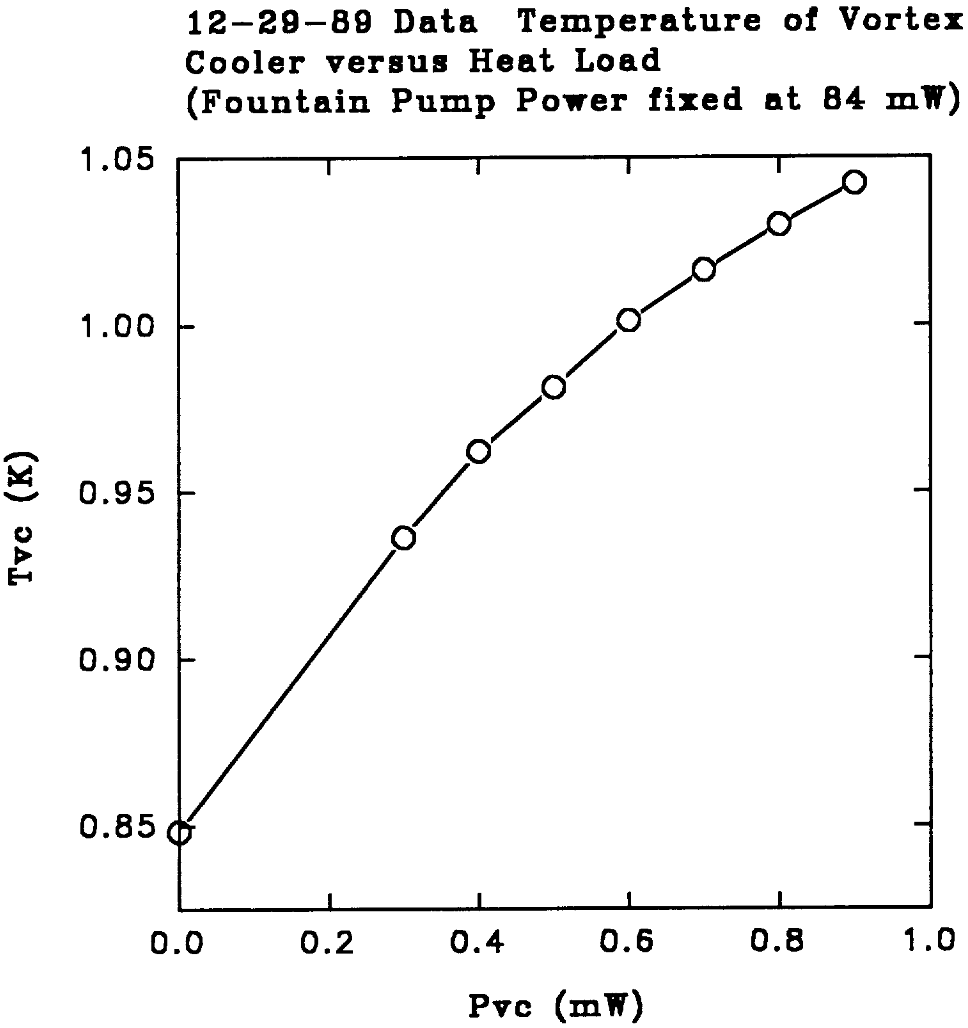
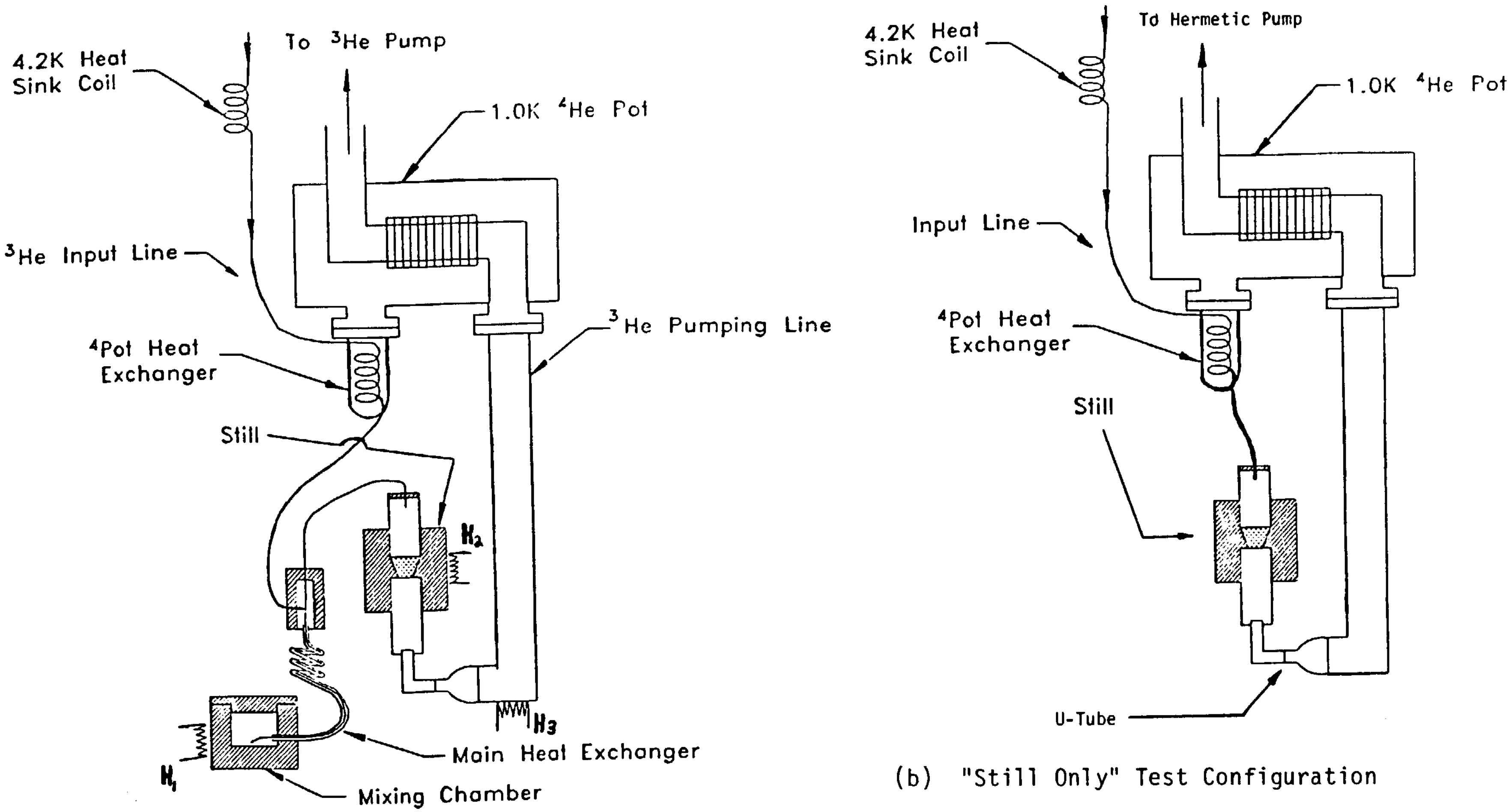

12-29-89 Data Temperature of Vortex
Cooler versus Heat Load
(Fountain Pump Power fixed at 84 mW)
1.05
1.00
0.95
0.90
0.85
Tvc (K)
0.0
0.2
0.4
0.6
0.8
1.0
Pvc (mW)

Figure 19

^{3}He Circulation Dilution Cryocooler Tests

The prototype dilution cryocooler for ^{3}He circulation tests was constructed as shown schematically in Figure 20(a). The idealized operating cycle of the refrigerator was as follows. ^{3}He from room temperature was returned by the hermetic pump, and passed through a liquid nitrogen temperature charcoal trap. This trap removed any impurities picked up by the ^{3}He gas from the pump. The gas then traveled down the dewar via capillary to the 4.2 K heat sink coil, which consisted of a 3′ length of 1/8" O.D., copper refrigerator tubing immersed in the 4.2 K helium bath. The gas then entered the ^{4}He pot heat exchanger. This exchanger consisted of a 1/16" O.D., 0.020" I.D. copper capillary 5′ long, immersed in a tube containing 1.0 K superfluid ^{4}He from the ^{4}He pot. The incoming ^{3}He then passed through one tube of the main heat exchanger. This exchanger consisted of two capillaries 18" long, with one nested concentrically inside the other. The outer capillary had an outside diameter of 0.060", with a 0.009" thick wall; the inner one had a 0.025" O.D., with 0.006" wall. Both capillaries were stainless steel, to cut down on longitudinal heat conduction. This exchanger was designed to approximate the specifications of the heat exchanger in the S.H.E. corporation mini-fridge (See Lounasmaa, p. 49). The inlet capillary then entered the mixing chamber, which was an OFE copper vessel with an internal volume of 0.79 cm^3. Here the ^{3}He mixes with the ^{3}He-^{4}He mixture to produce cooling. The ^{3}He then traveled back through the main heat exchanger up to the still. In the still, the liquid ^{3}He-^{4}He mixture was trapped by the porous silver trapping sponge. The trapping sponge was designed to hold the liquid via surface tension, thus preventing the liquid from falling through and thus establishing a phase boundary for evaporation of the ^{3}He. After evaporating from the



(a) ^{3}He Circulation Dilution Refrigerator Test Configuration

(b) "Still Only" Test Configuration

Figure 20

downstream side of the plug, the ^{3}He returned to room temperature via the ^{3}He pumping line. The trapping sponge was prepared similarly to those tested in the ACE, Inc. development program for a zero-g pure ^{3}He refrigerator (Hendricks and Dingus, 1987).

First, 400 Angstrom silver powder was compressed into the copper mount that made up the body of the still. Stainless steel pistons and a hydraulic press were used to accomplish this. Then , the plug was sintered in place in the copper mount by putting the entire assembly into a vacuum furnace. The plug was then heated to 200 C in forty minutes, and kept at 200 C for 1.5 hours. At this time, the oven was shut off and allowed to return to room temperature. During sintering, the plug was confined in a jig between two teflon cylinders. As the plug was heated, the teflon expanded, applying pressure to the plug material. This prevented the plug from breaking loose from the mount wall due to contraction during sintering. This method of sintering silver was similar to that used by Franco (1984) and resulted in a plug with a packing fraction of approximately 50%. The void volume of the plug was found to be 0.080 cm^3, and the volume of the still chamber above the plug was measured to be 0.709 cm^3.

Testing the ^{3}He Circulation Dilution Cryocooler

A 50% test mixture of ^{3}He-^{4}He was prepared to use in the cryocooler cycle. The sample mixture was condensed into the sample according to standard procedure for operation of dilution cryocoolers (See Lounasmaa, p. 23). However, several different test runs failed to produce cooling of the dilution chamber when heat was applied to the still. After these attempts were made, it was decided to look at the behavior of the separate components that made up the device, in order to understand what problems existed that were preventing operation of the whole system. The most complex part of the system was the still; thus, the decision was made to first carefully evaluate the performance of the still as a phase separator in an attempt to understand more about the behavior of the whole system.

Still Phase Separator Tests

To test still phase separator only, the apparatus was modified as shown in Figure 20(b). Essentially, the dilution chamber and main heat exchanger were removed. An input capillary 8" long, made from 0.004" I.D. stainless steel was installed between the ^{4}He pot heat exchanger and the still. Thermometers were used to monitor the temperatures of the ^{4}He pot, the still itself, and the lowest point at the bend of the pumping line below the trapping sponge, which we referred to as the u-tube. Heater coils were installed at both the still and u-tube locations, so that the effects of applying a known heat flux to these locations could be observed.

Figure 21 shows the results of a test run using this configuration. At time t=2.83, a sample mixture of 50% ^{3}He-^{4}He was admitted to the input capillary of the sealed and evacuated still. In ~10 minutes, the system reached equilibrium, with the sample condensed into the still. At t=3.40, the hermetic pump was engaged and opened to the still pumping line. Immediately, the temperature of the still and u-tube fell to ~0.5 K. During this cooldown, the input capillary to the still was left open. By t~4.0, equilibrium had been reached, with the still acting as a continuous mode 0.5 K cryocooler.

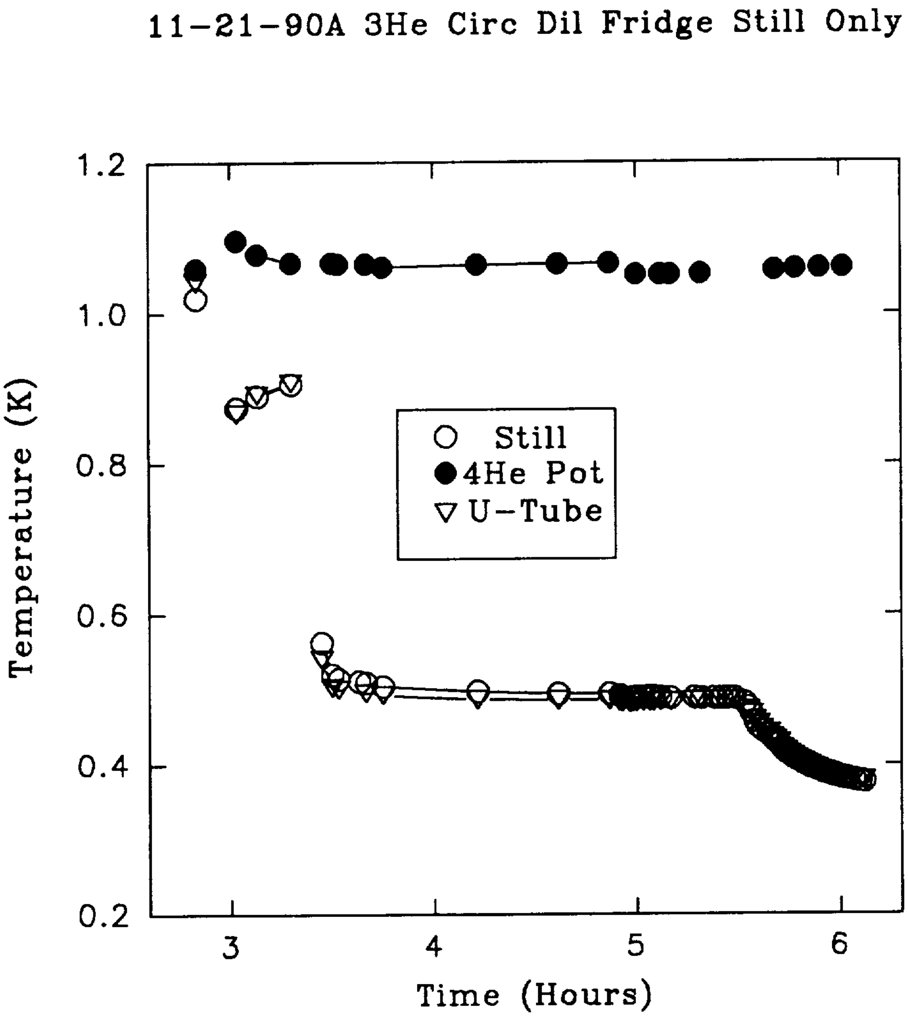

11-21-90A 3He Circ Dil Fridge Still Only
Temperature (K)
1.2
1.0
0.8
0.6
0.4
0.2
Time (Hours)
3
4
5
6
○ Still
● 4He Pot
▽ U-Tube

Figure 21

At 0.5 K the vapor pressure of ^{4}He is negligible, while the vapor pressure of ^{3}He is still significant (P_s = 0.2 Torr). With the still operating at this temperature, the pump should be continuously circulating almost pure ^{3}He. At t=4.92, the input capillary was closed off, so that no more circulation occurred. A very slight cooling of the still was then evident. This cooling should be expected, since "warm" 1.0 K ^{3}He was no longer being pumped into the still. Figure 22 shows the data during this part of the run on a more sensitive scale. Also, the ^{4}He pot was seen to cool slightly at t=5.00, since it no longer had to bear the burden of liquifying the incoming ^{3}He. The still stayed in rough equilibrium until t=5.50, at which time an unexpected decrease in the temperature of the still began. A decrease in the mass flow out of the system also occurs as is evident from Figure 23. In this figure, the pressure of the ^{4}He-^{3}He mixture contained in the fixed effective storage volume V=7.72 l is shown. The volume, V, is the sum of the storage tank volume and the volume of the output side of the pump and connecting lines, and was carefully measured and calculated. The product of the effective volume, V, and the pressure shown in Figure 23 give the total amount of gas in the system present at room temperature. Thus, as the amount of mixture present in the still decreases by being pumped away, the pressure in V is seen to rise. A decrease in the slope of the graph dp/dt means a slowing of the rise of mixture boiling off in the cell. A reduced boiloff rate of mixture in the cell implies a reduced heat load to the still.

This is true since the mass flow $\dot{m}$ of material evaporating is described by

$$\dot{Q}_{load} = \dot{m}L \tag{16}$$

where $\dot{Q}_{load}$ is the heat load and L is the latent heat of vaporization.

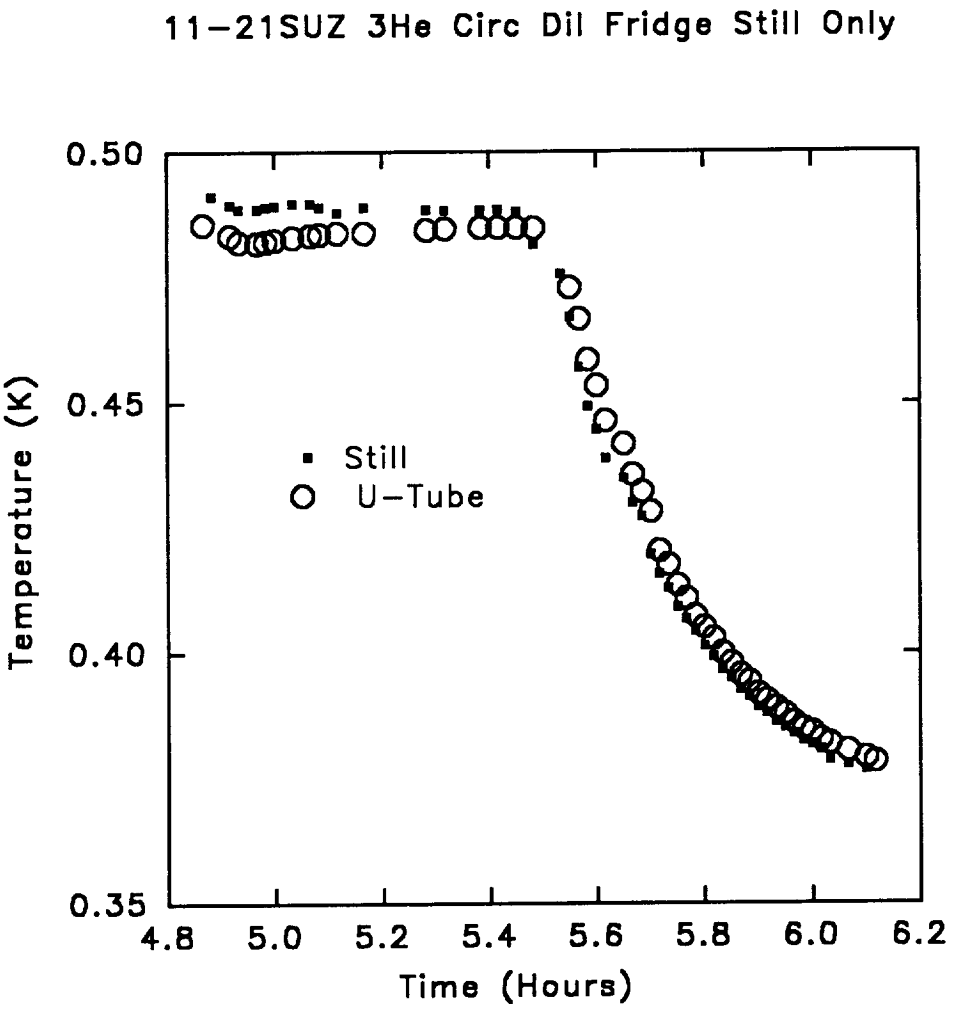

11-21SUZ 3He Circ Dil Fridge Still Only
0.50
0.45
0.40
0.35
Temperature (K)
4.8
5.0
5.2
5.4
5.6
5.8
6.0
6.2
Time (Hours)
■ Still
○ U-Tube

Figure 22

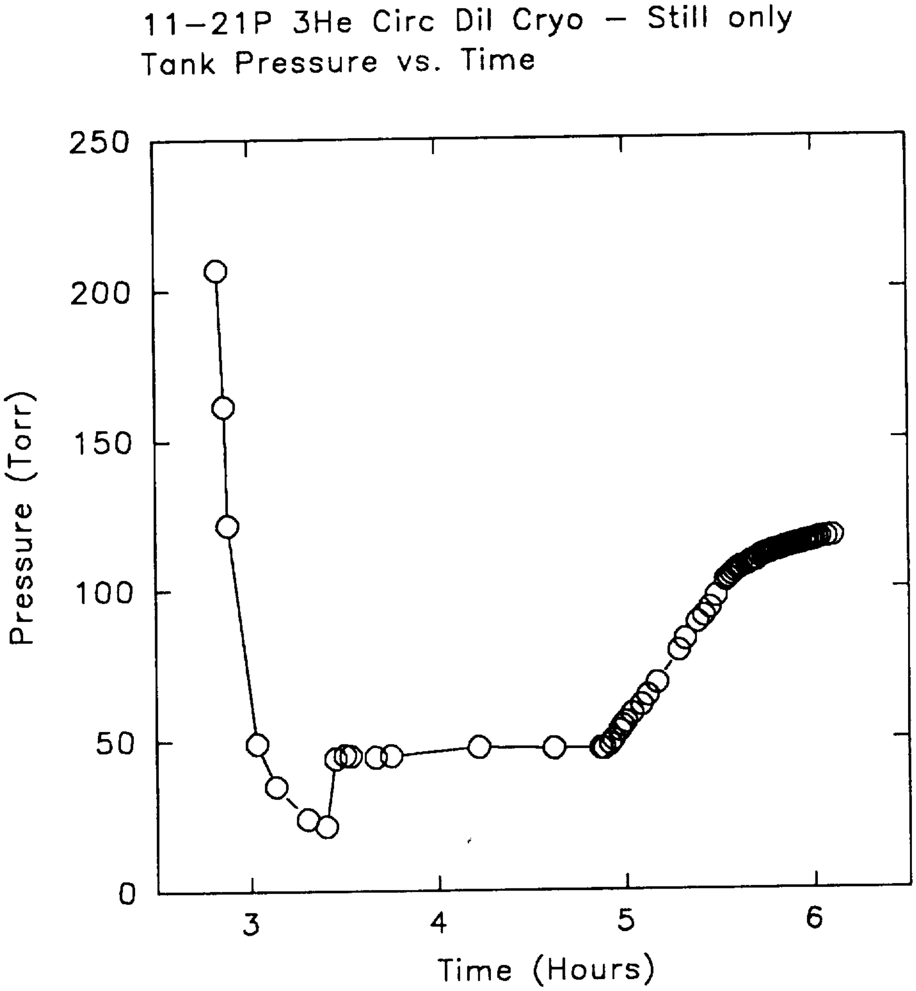

11-21P 3He Circ Dil Cryo – Still only
Tank Pressure vs. Time
250
200
150
100
50
0
Pressure (Torr)
3
4
5
6
Time (Hours)

Figure 23

From Figure 24 it can thus be seen that the drop in still temperature that occurs at t=5.50 is associated with a corresponding decrease in the heat leak to the still. Figure 23 shows pressure variations over the entire duration of the experiment, including the pressure drop associated with filling the still with liquid. It is not clear why the spontaneous cooling occurred at t=5.50. If the breakout pressure of the porous plug was exceeded, ^{3}He-^{4}He mixture could be forced through it. If this occurred, then one possible explanation for the apparatus staying at 0.5 K is that ^{4}He superfluid film traveled up the pumping line, evaporating at a much higher temperature. This phenomenon could limit the ultimate lowest pressure obtained at the evaporative interface with the ^{3}He in the still. ^{4}He at T=1.0 K has the same vapor pressure as ^{3}He at T=0.5 K. Thus, film flow up the pumping line to the ^{4}He pot could limit the temperature reached by the still to T_{still}=0.5 K until the ^{4}He disappeared from the system. Then, the still would be allowed to cool down further unencumbered. Verification of the above conjecture could be made simple if the ^{3}He concentration of the gas being removed by the pump at a given time could be measured. Unfortunately, the equipment necessary to make such measurement was not available.

Figure 25 shows temperatures of the still, ^{4}He pot and u-tube versus time for another run with the same experimental configuration. Initially, equilibrium existed at 4.2 K, before pumping on the ^{4}He pot began. At t=11.47, the ^{4}He pot had been pumped and had reached 1.0 K; the inlet capillary was then opened. Figure 26 shows the pressure of the sample, which was stored in the effective volume V, drop as the sample liquifies into the still. At t=13.58, pumping was applied to the mixture in the still and immediate cooling was observed. Figures 27 and 28 show this portion of the

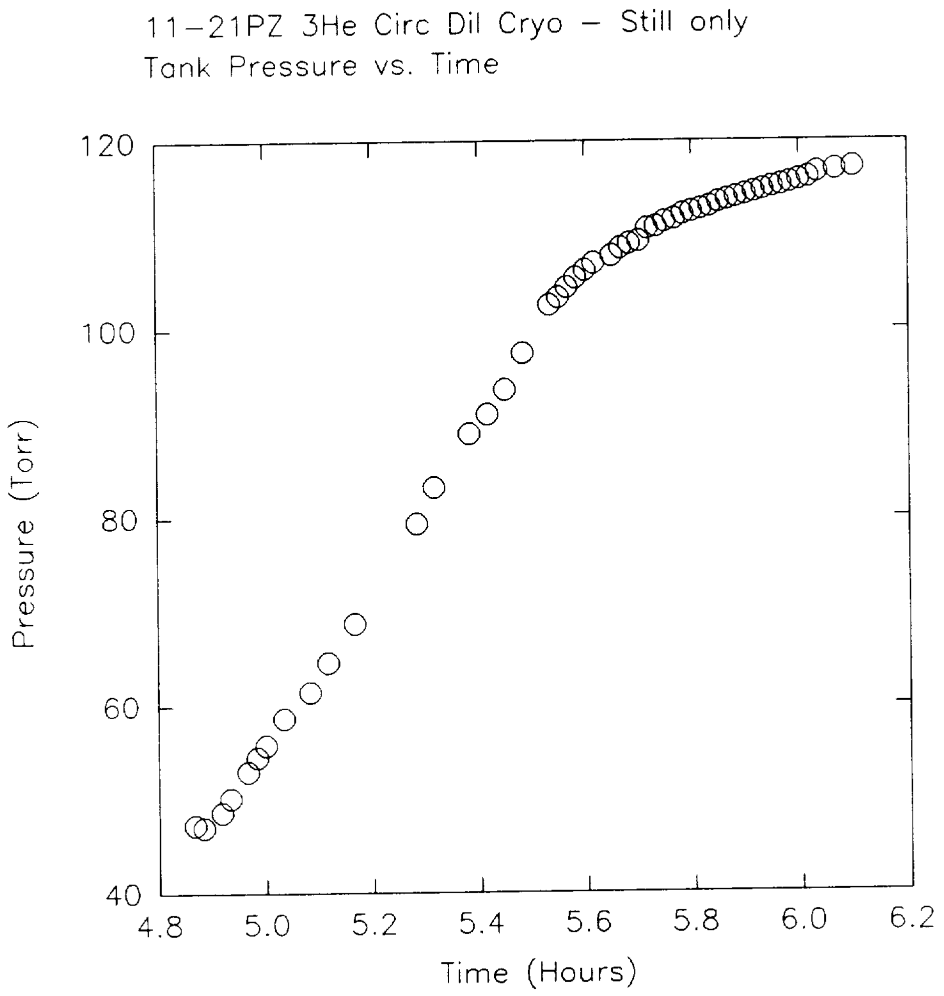

11−21PZ 3He Circ Dil Cryo − Still only
Tank Pressure vs. Time
Pressure (Torr)
120
100
80
60
40
4.8
5.0
5.2
5.4
5.6
5.8
6.0
6.2
Time (Hours)

Figure 24

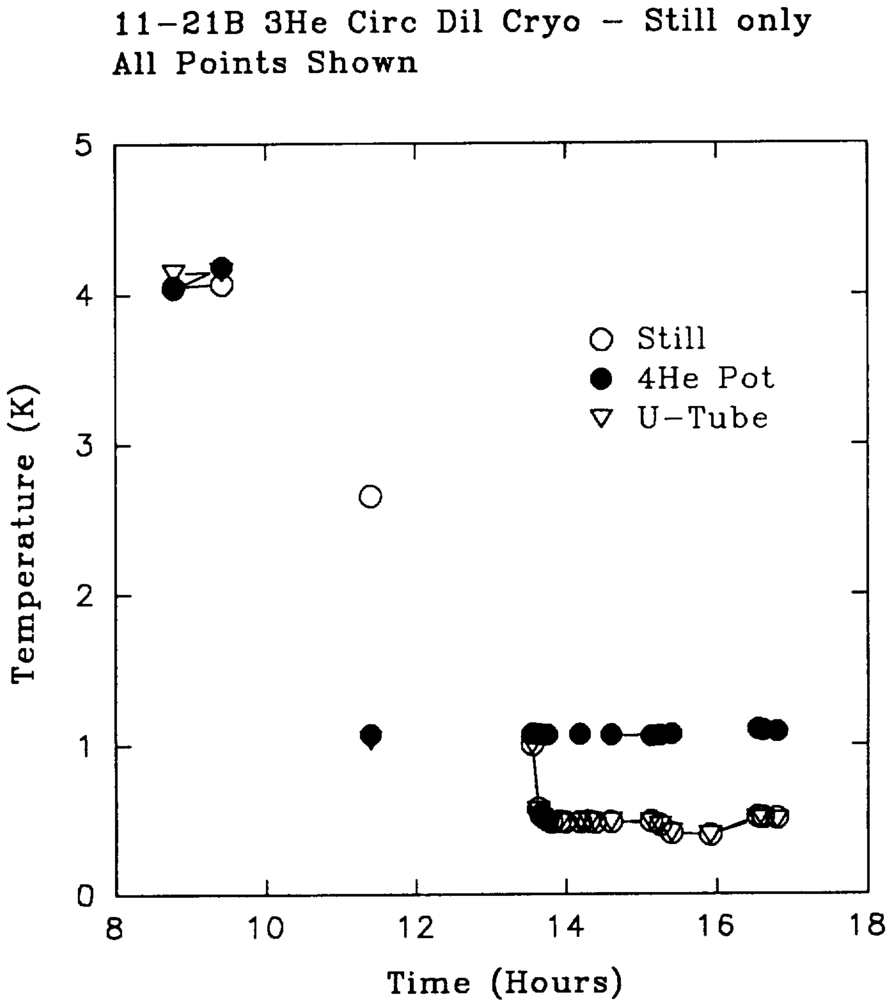

11-21B 3He Circ Dil Cryo - Still only
All Points Shown
Temperature (K)
Time (Hours)
0
1
2
3
4
5
8
10
12
14
16
18
○ Still
● 4He Pot
▽ U-Tube

Figure 25

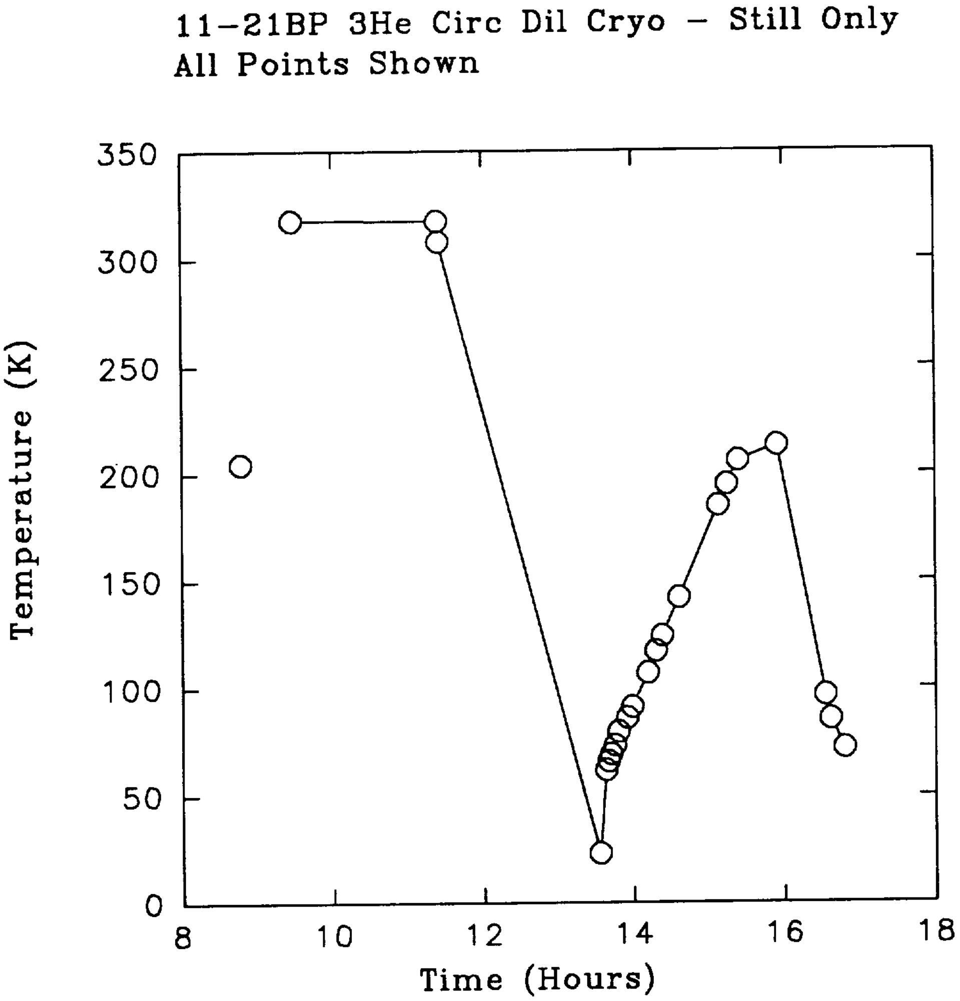

11-21BP 3He Circ Dil Cryo - Still Only
All Points Shown
Temperature (K)
Time (Hours)
350
300
250
200
150
100
50
0
8
10
12
14
16
18

Figure 26

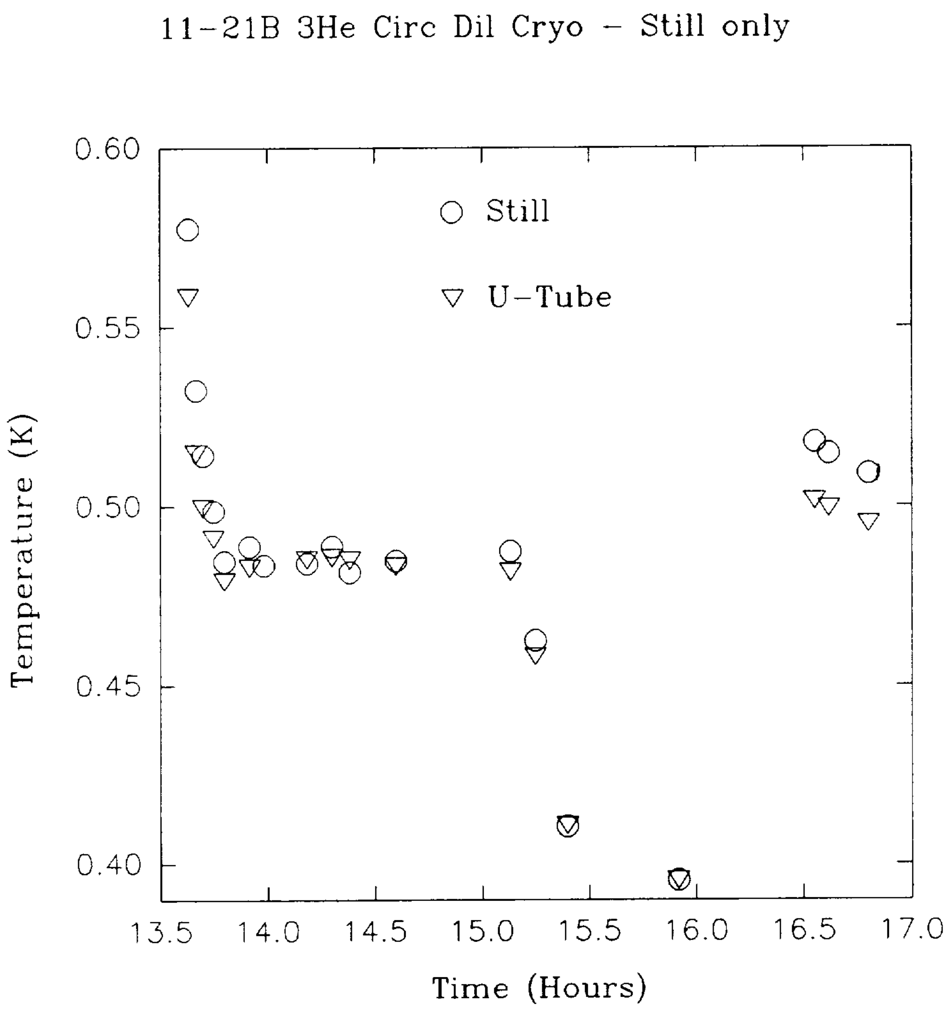

11-21B 3He Circ Dil Cryo – Still only
○ Still
▽ U-Tube
Temperature (K)
0.60
0.55
0.50
0.45
0.40
13.5
14.0
14.5
15.0
15.5
16.0
16.5
17.0
Time (Hours)

Figure 27

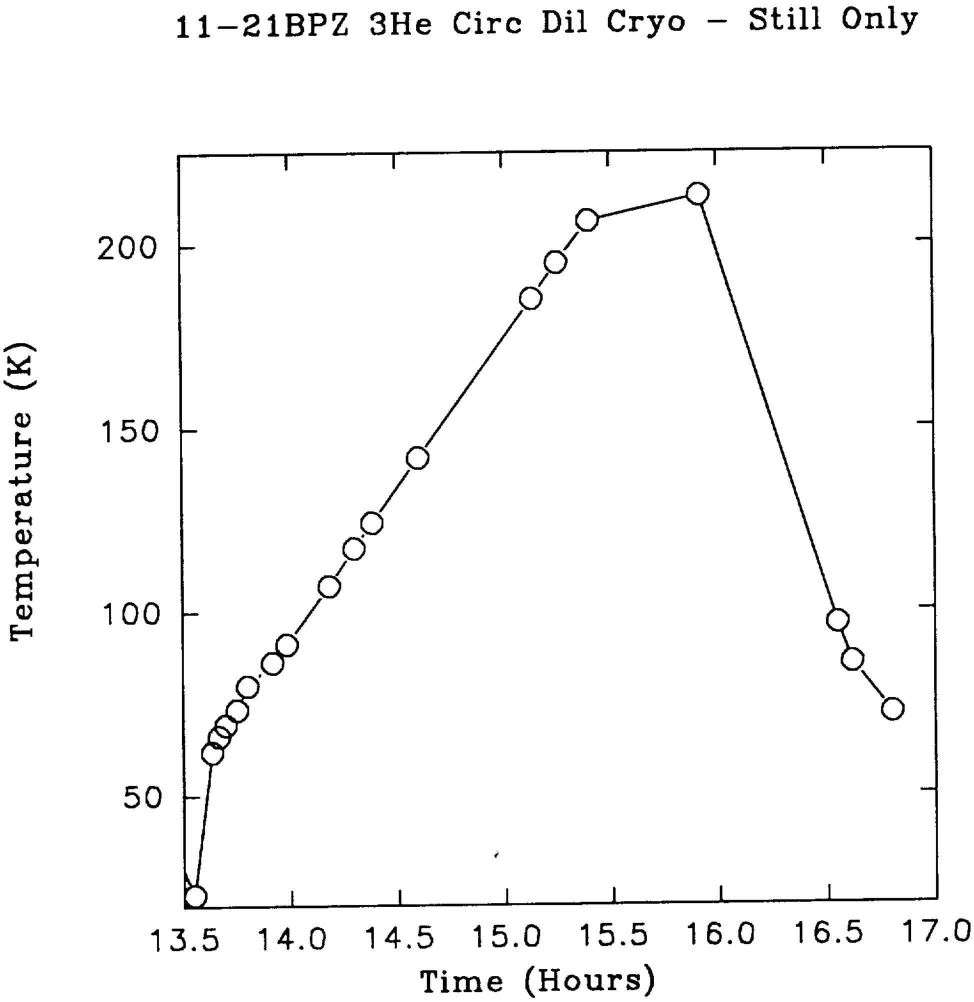
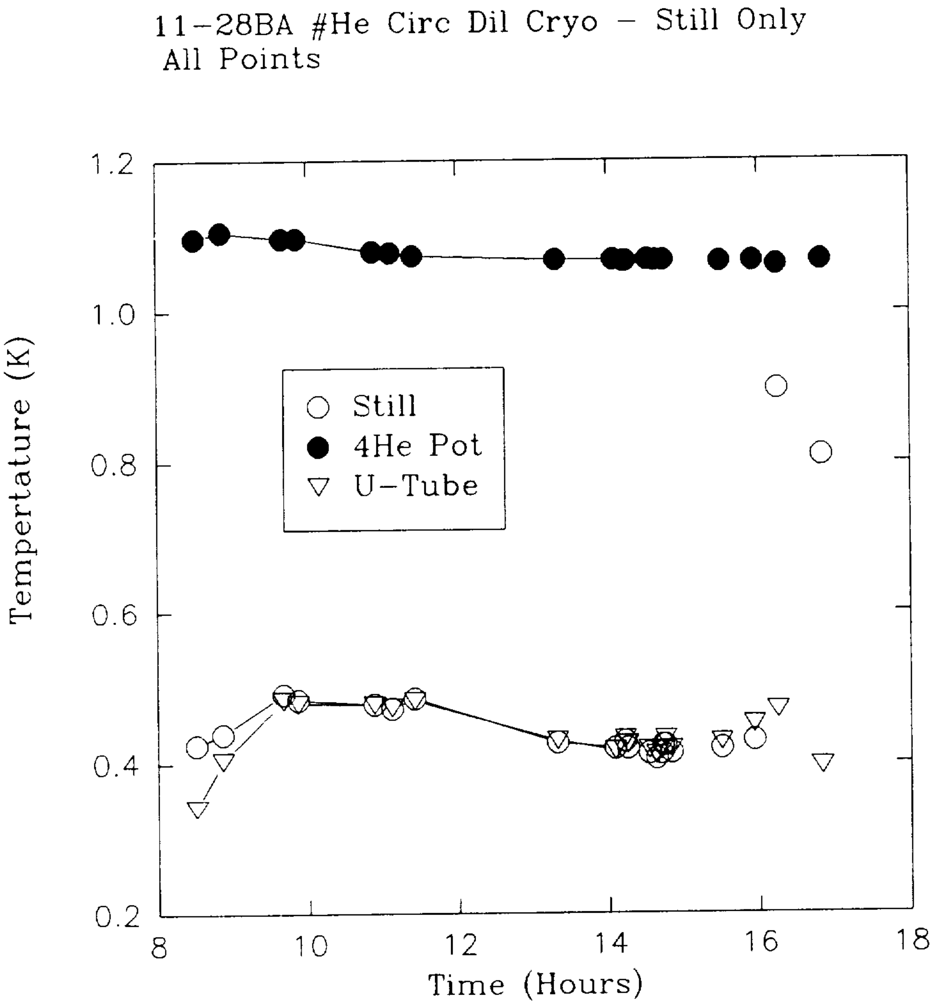

11−21BPZ 3He Circ Dil Cryo − Still Only
Temperature (K)
200
150
100
50
13.5
14.0
14.5
15.0
15.5
16.0
16.5
17.0
Time (Hours)

Figure 28

run on expanded scales. By t=13.75, equilibrium had been reached at roughly 0.48 K. This temperature equilibrium continued until t=15.40. During this time, the pressure versus time graph revealed that more material was leaving the still than was condensing down the input capillary, causing P to rise steadily. Suddenly, at t~15.40, a spontaneous cooling of both the sponge and u-tube occurred. At the same time, a decrease in the boiloff rate, which is proportional to dp/dt, took place. This trend continued until t~16.00, when a massive, spontaneous liquification of the sample occurred, as evidenced by the plunge in pressure. Correspondingly, both the still and u-tube warmed up dramatically.

The origin of the spontaneous cooling and liquifaction effects, which had been previously observed, remains unclear. This phenomenon is likely related to the time evolution of distribution of liquid in various parts of the system. The 1.0 K condenser chamber above the sponge, the sponge itself, the bend in the pumping line at 1.0 K, and in the u-tube below the sponge are all possible locations for liquid distribution. Without better instrumentation, it was very difficult to measure the quantity of liquid in any given place.

In Figure 29 and 30 another run of the still configuration is shown where heating tests were conducted in order to determine whether or not liquid was retained in the trapping plug. Before the test, the system was allowed to run overnight after filling the still with liquid, then applying pumping. The still was then operating in continuous mode when data taking began at t=8.5. Between t=8.5 and t=8.9. helium was transferred into the dewar. During the period between t=8.9 and t=14.25 the still and u-tube temperatures first rose slightly, then fell. During this time, the pressure of the helium in the



11-28BA #He Circ Dil Cryo - Still Only
All Points
○ Still
● 4He Pot
▽ U-Tube
Tempertature (K)
Time (Hours)
1.2
1.0
0.8
0.6
0.4
0.2
8
10
12
14
16
18

Figure 29

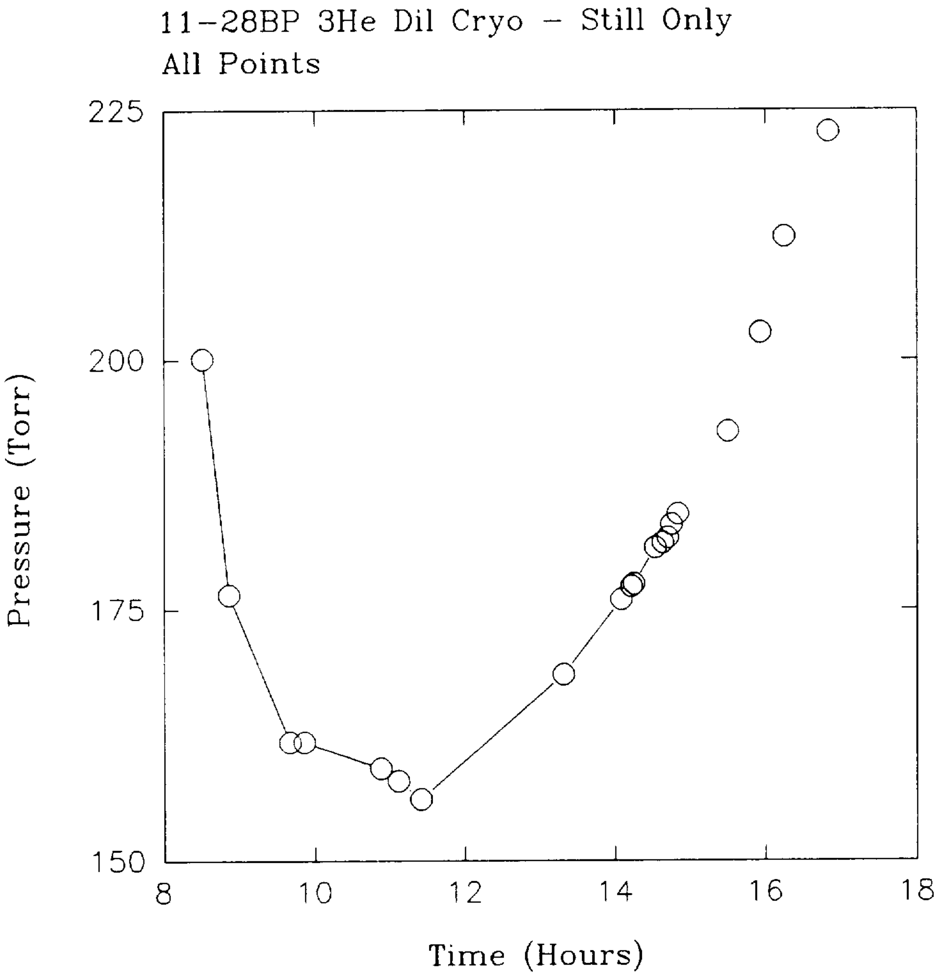
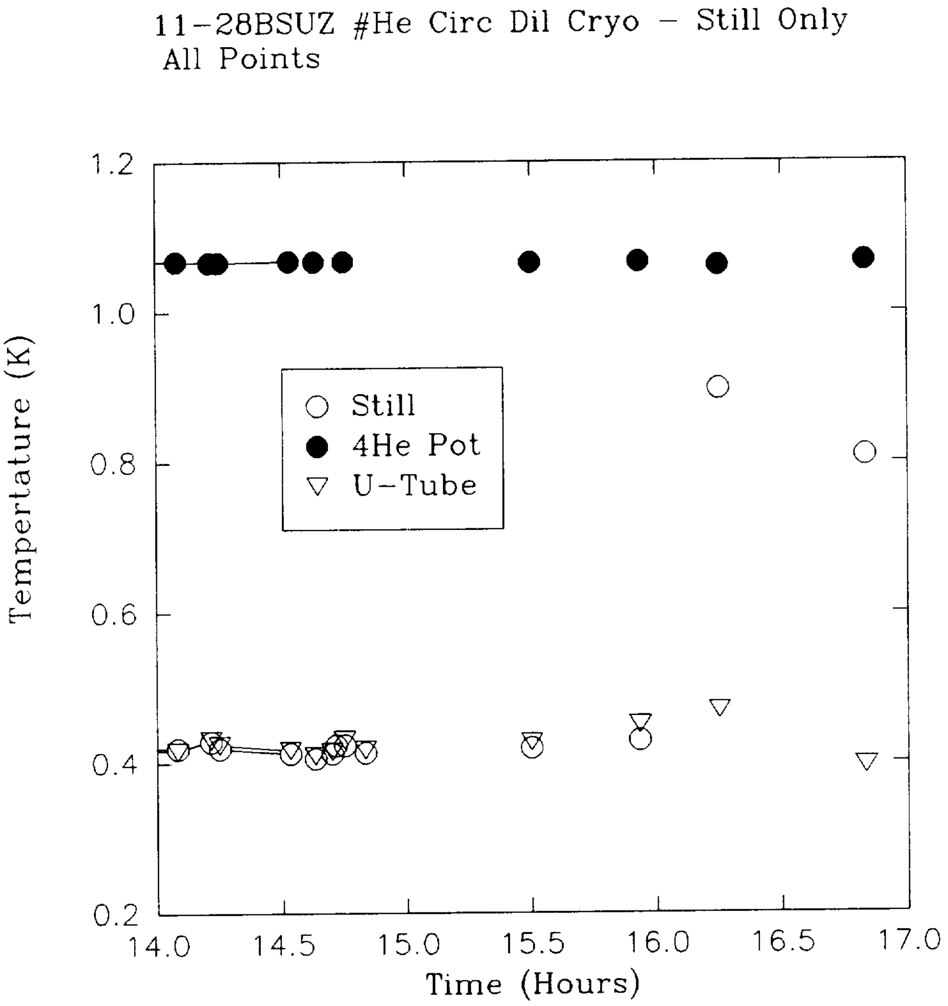

11-28BP 3He Dil Cryo - Still Only
All Points
225
200
175
150
8
10
12
14
16
18
Pressure (Torr)
Time (Hours)

Figure 30

fixed volume V first decreased, then increased. Both of these effects were on the order of 25%, and were possibly due to the thermal disturbance of the system caused by helium transfer. From t=14.25 until t=16.8, heat was applied first to the u-tube, then to the still. The temperature and pressure measurements taken during this period are shown in more detail in Figure 31 and 32. The purpose of these heating tests was to determine whether the liquid mixture in the still was being trapped by surface tension in and above the porous sponge, or forced through the sponge into the u-tube. If liquid is present where heat is applied, little temperature rise should be seen, since heat is taken away by the accelerated boiling of the liquid. However, if no liquid is present, a sharp temperature rise should be seen when low heats are applied. In Figure 33 the temperatures of the sponge and u-tube are shown as a function of input power to the u-tube. Little rise in temperature is seen in Figure 33 indicating the presence of liquid in the u-tube. In Figure 34, temperatures of the still and u-tube are shown as a function of heat applied to the still. Notice the marked rise in temperature of the still for relatively low input powers. This clearly illustrates that no liquid was present in the still at this time.

A very different situation is illustrated in Figures 35 and 36 when a different procedure was used to load the still. First, the still was completely evacuated. Then at t=10.03, the inlet capillary was opened to admit the sample to the still while the mixture pumping remained fully open. Figures 37 and 38 show temperature and pressure during this time on an expanded scale. Note that throughout the run, the temperature of the still remains substantially below that of the u-tube. This can only occur if liquid is present in the still only. Thus, these graphs represent proper continuous



11-28BSUZ #He Circ Dil Cryo – Still Only
All Points

Figure 31

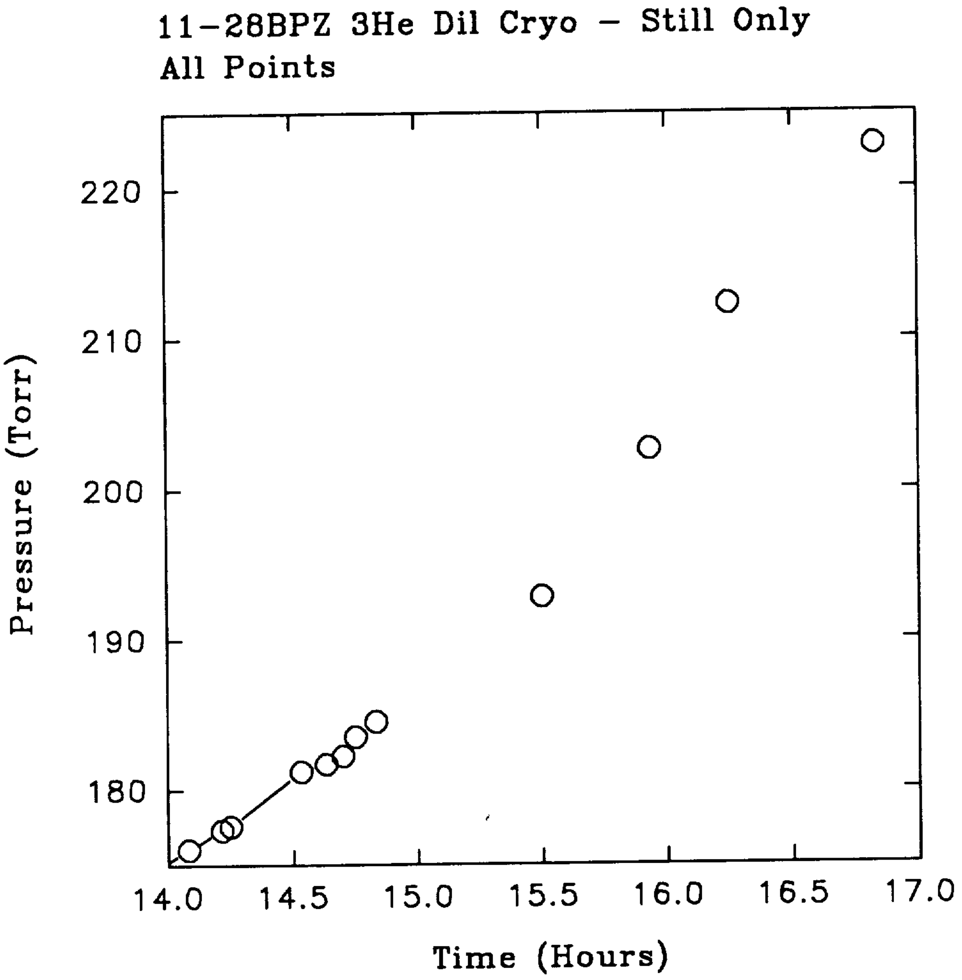
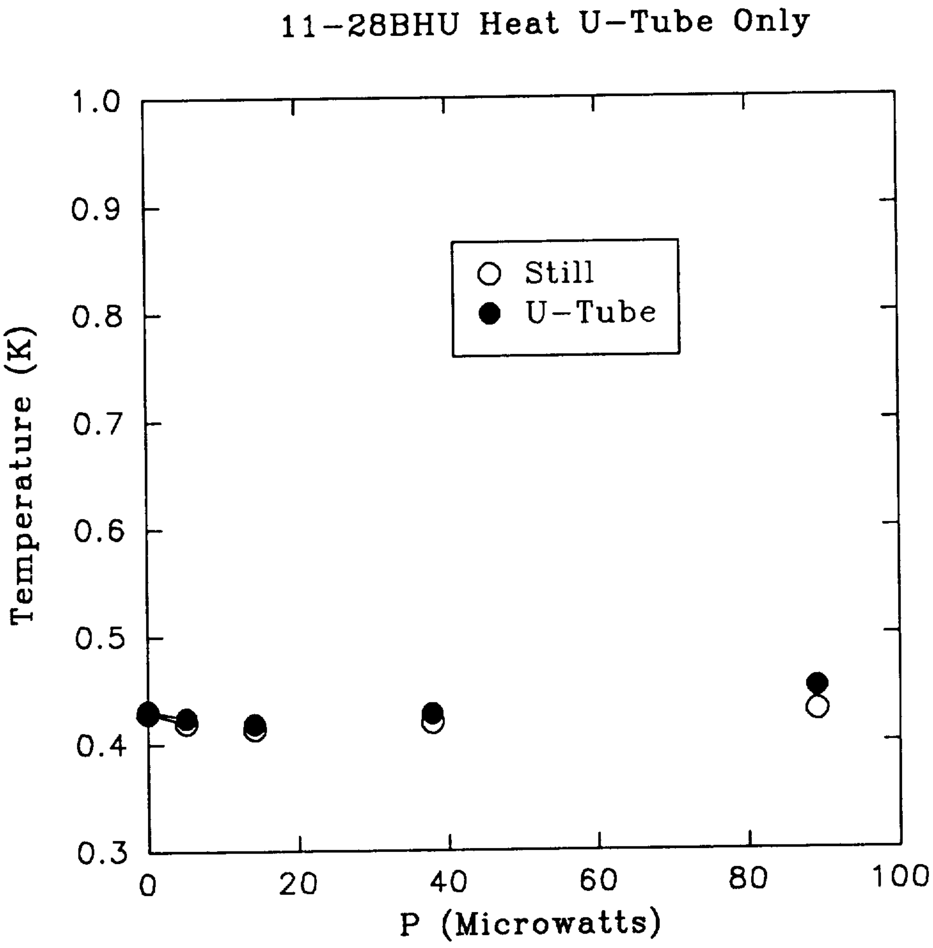

11-28BPZ 3He Dil Cryo - Still Only
All Points

Figure 32



11-28BHU Heat U-Tube Only
○ Still
● U-Tube
Temperature (K)
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0
20
40
60
80
100
P (Microwatts)

Figure 33

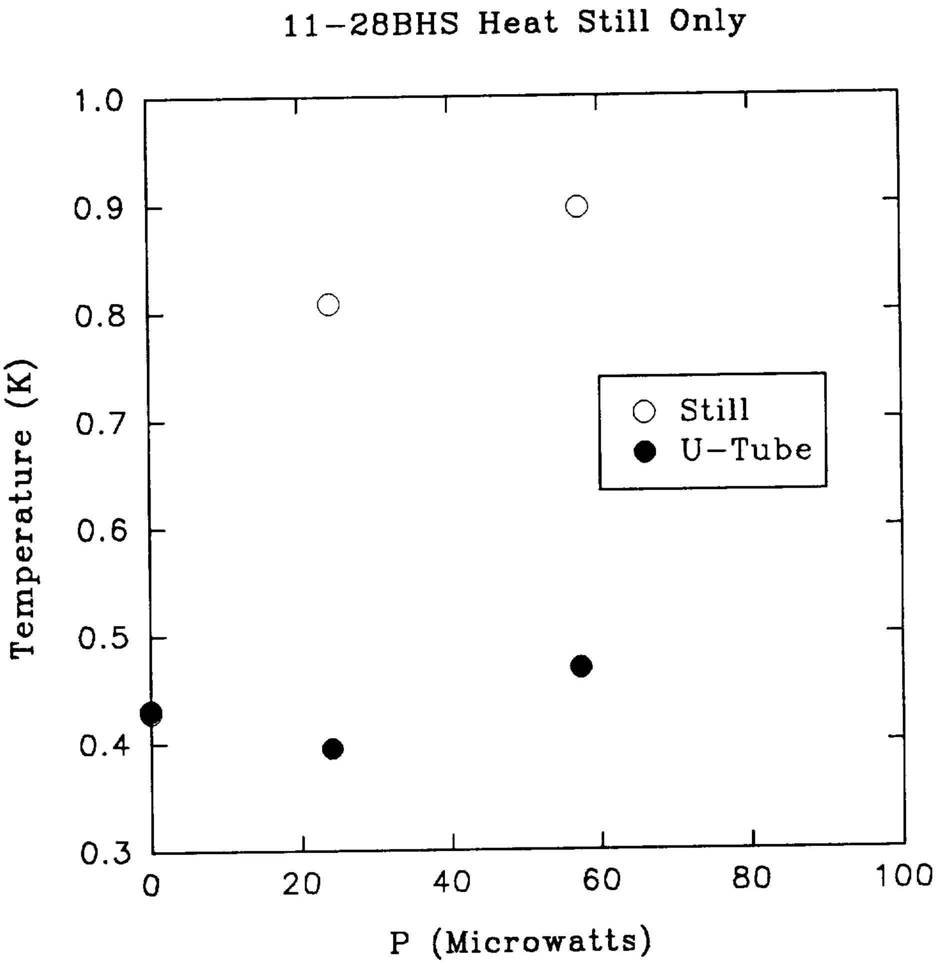

11-28BHS Heat Still Only
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
Temperature (K)
0
20
40
60
80
100
P (Microwatts)
○ Still
● U-Tube

Figure 34

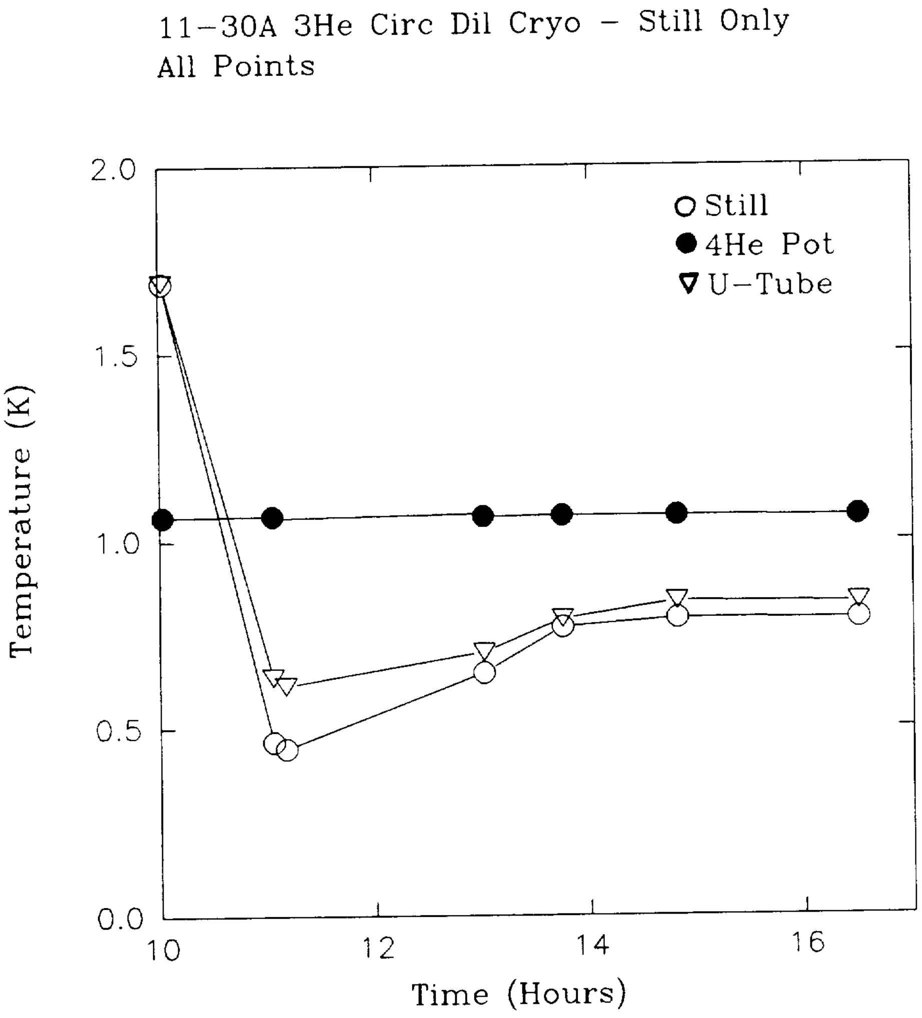
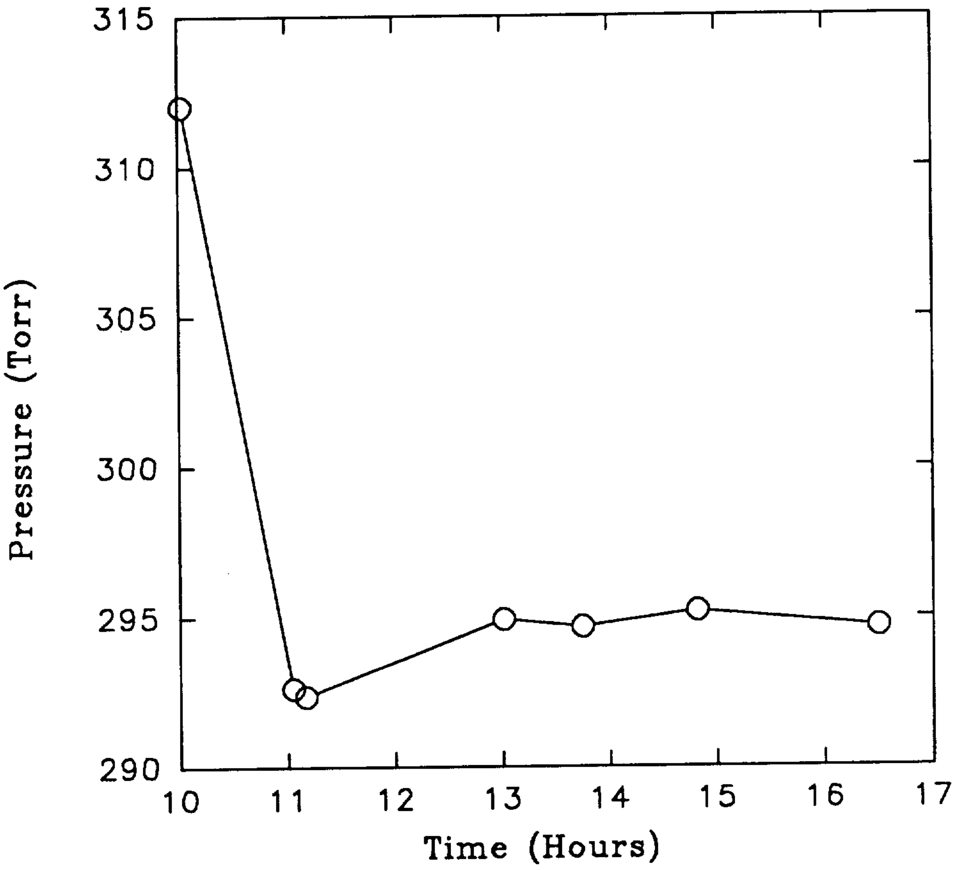

11-30A 3He Circ Dil Cryo - Still Only
All Points
○ Still
● 4He Pot
▽ U-Tube
Temperature (K)
2.0
1.5
1.0
0.5
0.0
10
12
14
16
Time (Hours)

Figure 35



11–30P 3He Circ Dil Cryo – Still Only
All Points

Figure 36

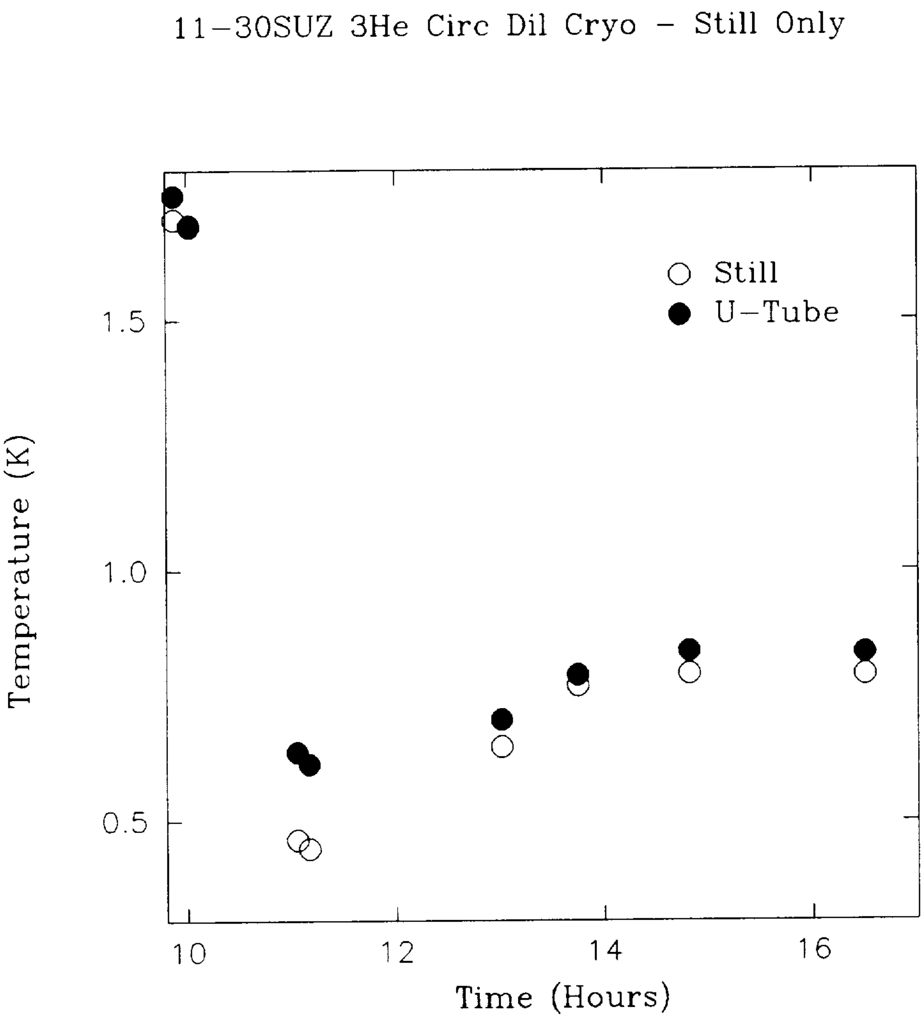

11-30SUZ 3He Circ Dil Cryo - Still Only
○ Still
● U-Tube
Temperature (K)
1.5
1.0
0.5
10
12
14
16
Time (Hours)

Figure 37

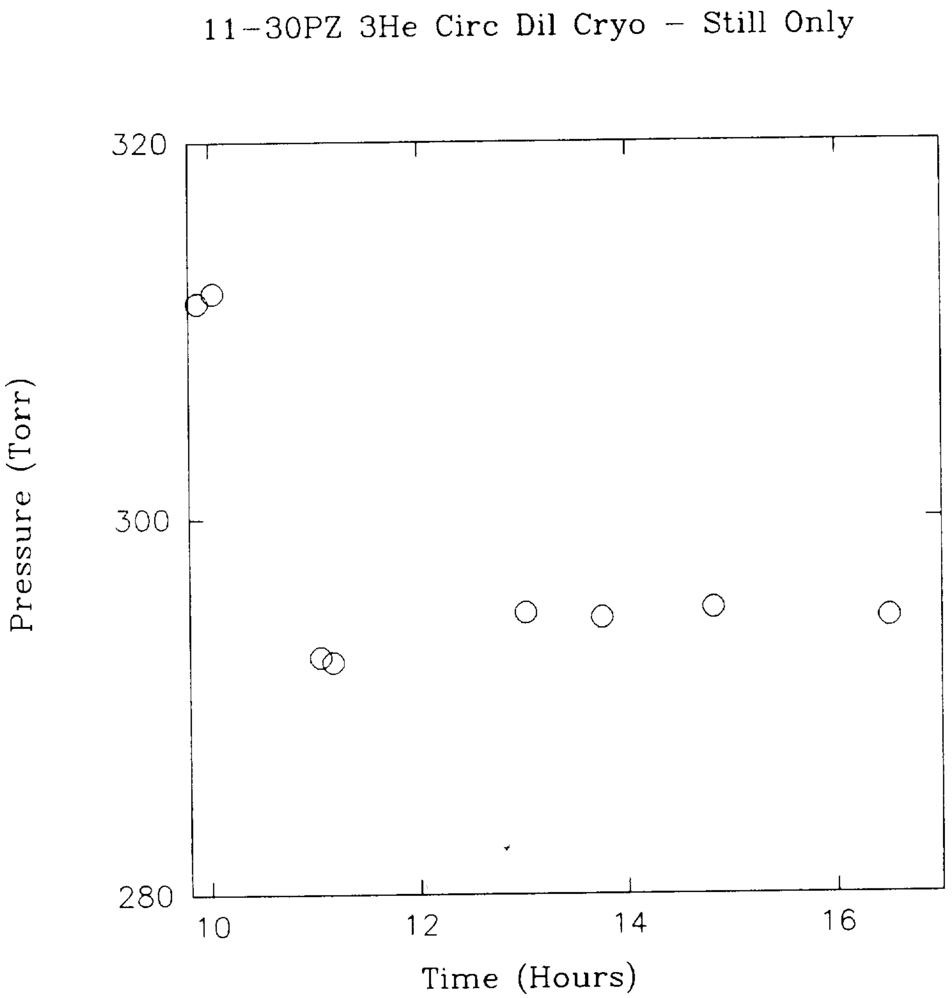

11-30PZ 3He Circ Dil Cryo – Still Only
320
300
280
Pressure (Torr)
10
12
14
16
Time (Hours)

Figure 38

mode operation of the porous sponge phase separator in the negative, or -1g configuration. The order of filling and pumping the cell seems to be very important in avoiding liquid breakout through the trapping plug.

^{4}He Circulation Dilution Cryocooler Results

The ^{4}He circulation dilution cryocooler configuration is shown in Figure 39. To prepare for a test run, the ^{4}He and ^{3}He pots were first cooled to 1.0 K and 0.4 K, respectively. Exchange gas was admitted to the exchange gas sleeve. This column of gas acted as a weak thermal link between the 1.0 K ^{4}He pot and the cooling chamber, and prevented rapid warmup of the cooling chamber when sample was admitted to it. ^{3}He was then condensed into the cooling chamber, filling the drift tube and other tubing between the superleaks. Since superleaks prevent ^{3}He flow, the sample was trapped. At this time, ^{4}He was admitted to the input, filling up the input and output capillaries and filling the remainder of the cooling chamber and drift tube with ^{4}He. The exchange gas was then pumped from the exchange gas sleeve, and ^{4}He could then be circulated through the device, with cooling of the dilution chamber expected.

Dimensions of the components used to construct the ^{4}He circulation dilution cryocooler are as follows. The inlet superleak was fabricated from a 2 1/4" length of 1/4" O.D., 0.010" wall stainless steel tubing packed with 0.5 micron alumina powder. The outlet superleak was made from the same materials, but was 2 1/4" long. The drift tube consisted of a 50cm length of 0.1mm I.D. stainless steel capillary. This tube could be easily interchanged with other drift tubes, if needed. 8" lengths of 0.1mm stainless steel

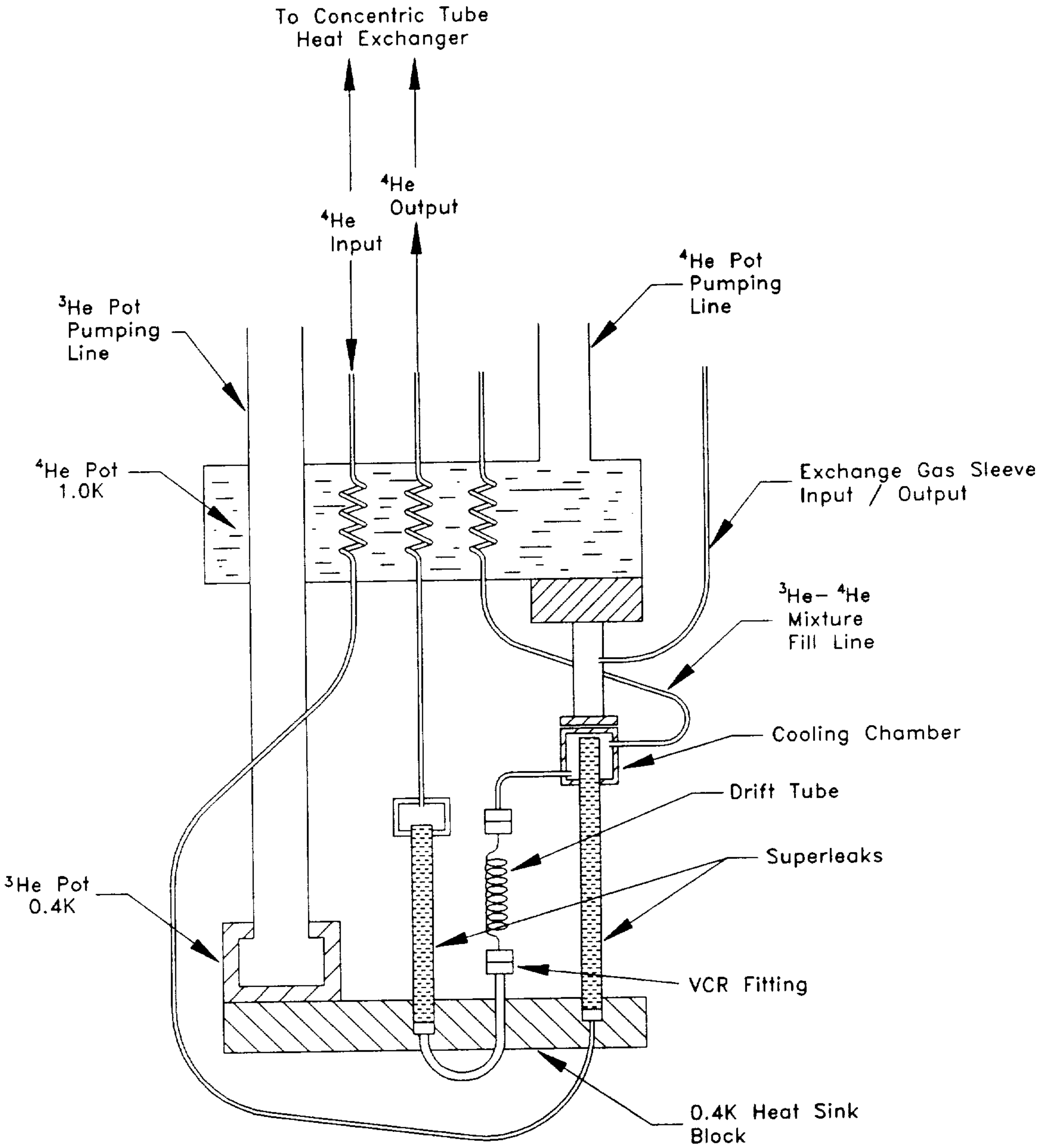


Figure 39 ACE, Inc. ^{4}He Circulation Dilution Cryocooler Prototype

capillary carried the ^{4}He from the ^{4}He pot heat sink into and out of the cryocooler.

In Figures 40 and 41 a preliminary test of the ^{4}He circulation dilution cryocooler is shown. In this test, the exchange gas sleeve, the cooling chamber and the input/output ^{4}He capillaries were evacuated. A 50% ^{3}H3-^{4}He mixture was used to fill the ^{3}He pot. This was done to observe the differences in mixture behavior between the -1-g configuration and normal evaporative cooling arrangements. The ^{3}He pot was a closed cylindrical copper vessel with the same silver powder used to make up the still trapping sponge sintered in a layer at its bottom. From the results, it is seen that an anomalous spontaneous cooling occurs at t=2.5 and that the boiloff rate in the pot suddenly decreases then. This is very similar to the anomalous behavior seen in the still tests conducted in the -1g configuration. This fact suggests that the source of the anomaly is not due to use of the phase separator in the -1g configuration. It may occur due to liquid present at 1.0 K inside the ^{3}He pumping line bend. When the ^{4}He disappears from its evaporation point at 1.0 K, the temperature of the device then falls.

In Figures 42 and 43, the results of a test with ^{3}He trapped between the superleaks is shown. ^{3}He was admitted between the superleaks in the form of 50% ^{3}He-^{4}He mixture. Then, ^{4}He was admitted to the ^{4}He inlet line. Pumping on the ^{3}He-^{4}He mixture in the ^{3}He pot at t=2.6 cooled the pot down to ~ 0.5 K. The dilution chamber then began to cool very slowly, but the time constant for this cooling was so large that the temperature of phase separation of the mixture was never obtained. Thus, the tests of the ^{4}He circulation dilution cryocooler apparatus were inconclusive.

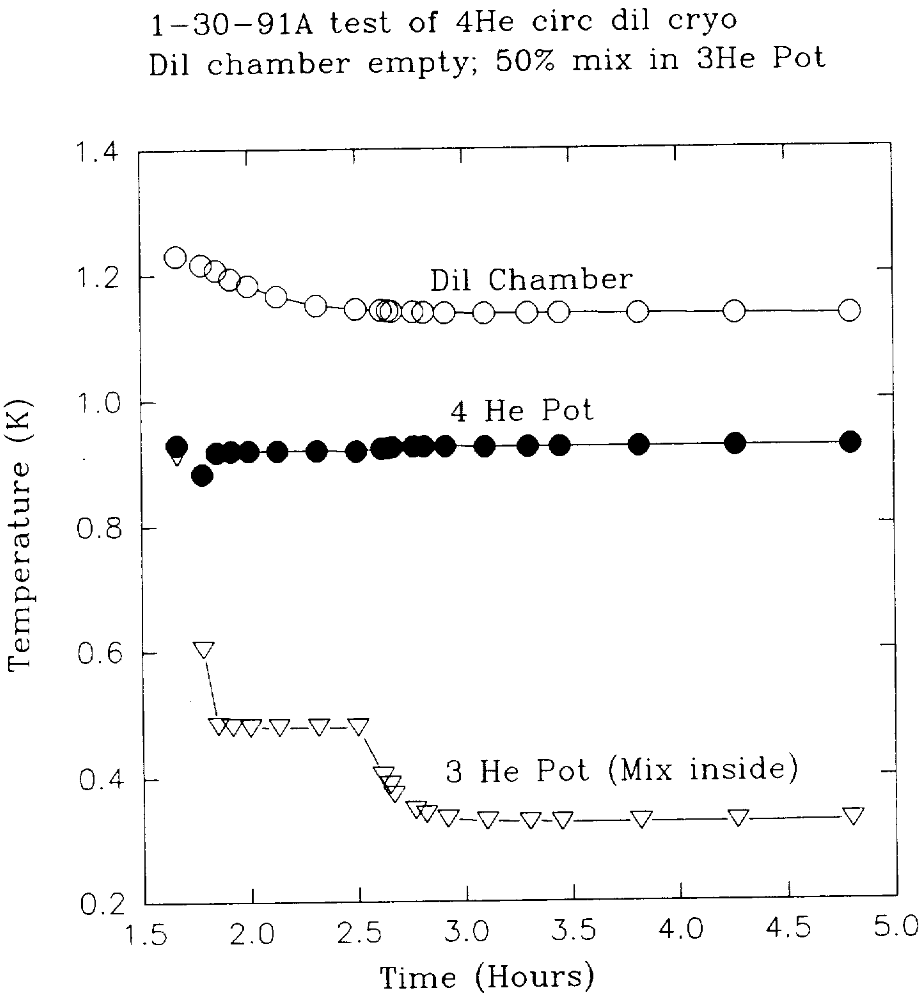

1-30-91A test of 4He circ dil cryo
Dil chamber empty; 50% mix in 3He Pot
Dil Chamber
4 He Pot
3 He Pot (Mix inside)
Temperature (K)
1.4
1.2
1.0
0.8
0.6
0.4
0.2
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
Time (Hours)

Figure 40

1-30-91A test of 4He circ dil cryo
Dil chamber empty; 50% mix in 3He Pot

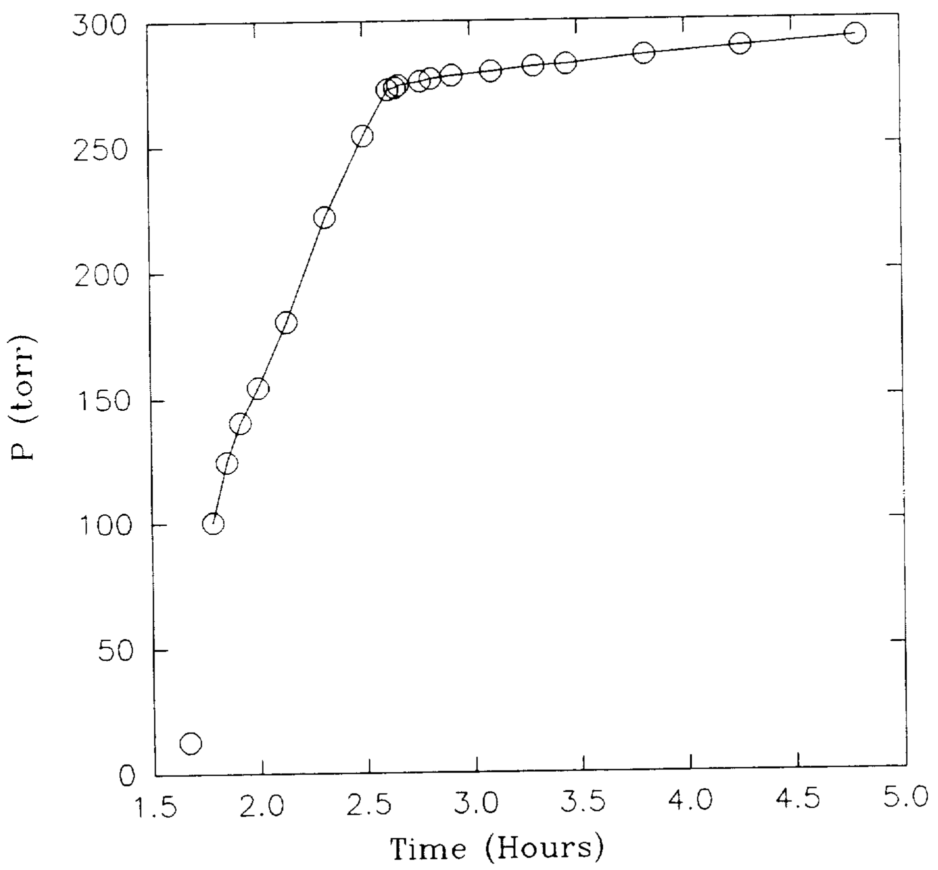


Figure 41

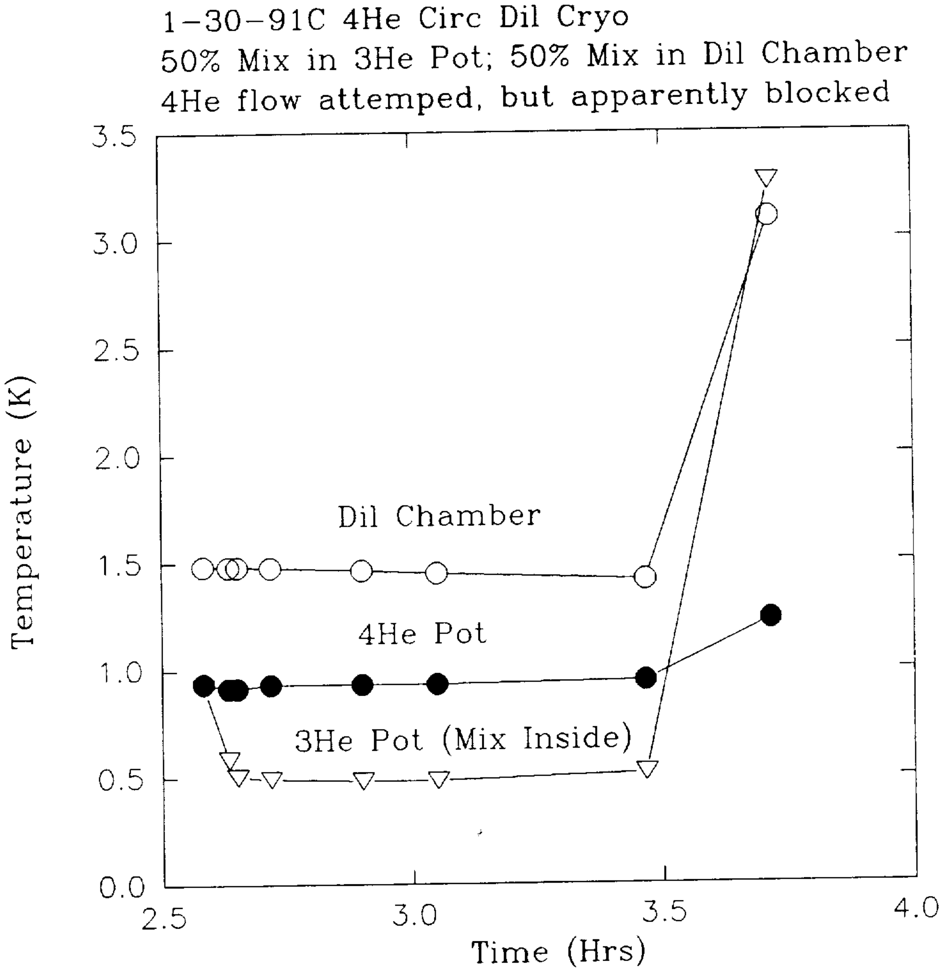

1-30-91C 4He Circ Dil Cryo
50% Mix in 3He Pot; 50% Mix in Dil Chamber
4He flow attemped, but apparently blocked
Temperature (K)
Time (Hrs)
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
2.5
3.0
3.5
4.0
Dil Chamber
4He Pot
3He Pot (Mix Inside)

Figure 42

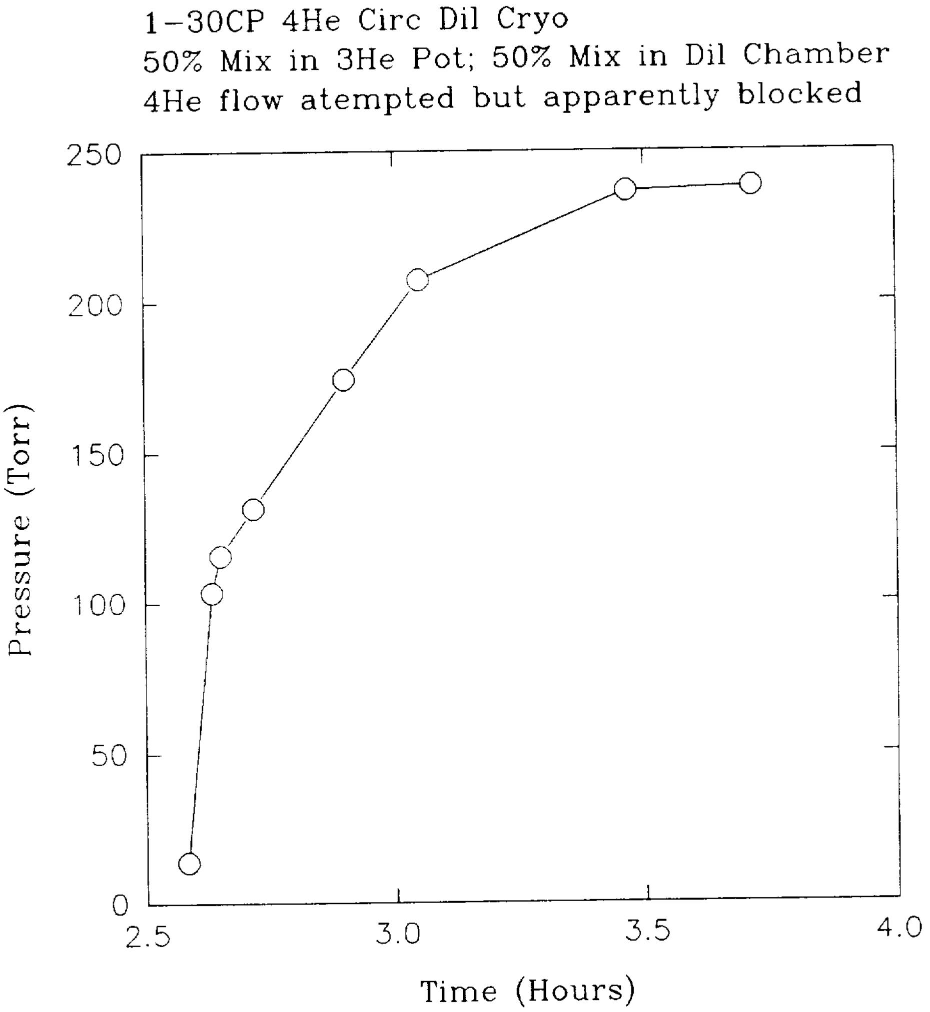

1-30CP 4He Circ Dil Cryo
50% Mix in 3He Pot; 50% Mix in Dil Chamber
4He flow atempted but apparently blocked
Pressure (Torr)
0
50
100
150
200
250
Time (Hours)
2.5
3.0
3.5
4.0

Figure 43

CONCLUSIONS

The primary conclusion of this research program is that a zero gravity cryocooler using the principle of the ^{3}He/^{4}He dilution refrigerator is feasible. Tests of the porous metal phase separator show that trapping of a ^{3}He-^{4}He mixture against gravity can be achieved.

It was shown by Hendricks, Nilles and Dingus (1988) that the critical phase interface that must be controlled in the ^{3}He circulation dilution cryocooler is in the still. It has been demonstrated in this effort that such phase interface control can be established since continuous operation of the still with ^{3}He-^{4}He mixture confined on or above the phase separator against gravity has been observed.

It has also been shown that a good deal of experimental complexity is involved in conducting experiments with time dependent concentrations of ^{3}He mixtures as the test fluid. A need exists for apparatus permitting the real time evaluation of mixture concentration during circulation tests.

RECOMMENDATIONS

^{3}He Circulation Dilution Cryocooler

The feasibility of a ^{3}He circulation dilution cryocooler for zero gravity has been demonstrated by tests that show a porous still phase separator that operates in -1g. Development of a fully functional dilution refrigerator for zero gravity could provide substantially improved performance over the currently available adiabatic demagnetization refrigerator (ADR). To fully understand the operation of the elements that make up a ^{3}He circulation dilution cryocooler, and to successfully coordinate these parts into a working whole requires more tests with enhanced instrumentation. Additional instrumentation required is as follows.

Thermometry. To evaluate temperatures along pumping and input lines, in order to detect changes in thermal profile that might indicate film flow and liquid level changes, more thermometers are required.

Liquid Level Sensors. The level of liquid helium in a test cell can be monitored via open gap capacitors (See Dingus, 1986). A capacitance bridge with resolution of one part in 10^5 is required to measure capacitance changes as liquid moves between the capacitor plates. This high resolution is needed due to the small dielectric constant of liquid helium.

Real Time Evaluation of Sample Concentration. It is highly desirable to be able to measure the relative amounts of ^{3}He and ^{4}He circulating in the system at a give time. Also, a measurement of the ^{3}He concentration of the sample left at room temperature would reveal the ^{3}He concentration of material down inside the cryocooler, since the total net sample concentration is fixed.

A mass spectrometer is needed to perform analysis between the isotopes ^{3}He and ^{4}He. As an example, a helium leak detector sensitive to both ^{3}He and ^{4}He could be used. Calibrated standard leaks are available that can be filled with pure ^{3}He, pure ^{4}He or mixtures to provide fixed points. With these tools, a definitive study of the ^{3}He circulation dilution cryocooler can be made.

^{4}He Circulation Dilution Cryocooler

Further work is also needed on the ^{4}He circulation dilution cryocooler. A thermal switch is needed between the dilution cooling chamber and the ^{3}He pot to allow the system to reach equilibrium in a reasonable time and to permit cooling tests to be carried out. More detailed testing of this cycle would also benefit from the additional instrumentation described in the previous section.

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APPENDIX A

DILUTION CRYOCOOLER PHASE SEPARATORS FOR ^{3}HE

Below 0.86 K, ^{3}He - ^{4}He mixtures undergo a phase transition that results in the creation of a ^{3}He rich and a ^{4}He rich phase. This transition is illustrated in the phase diagram presented in Figure A-1. For the traditional earth-based dilution refrigerator to operate gravity is used to separate these two phases. The ^{3}He rich phase floats on top of the ^{4}He rich phase due to the mass density difference between the two isotopes. In space based applications, gravity will not be available to provide this needed phase separation.

A possible alternative method would be to utilize surface tension to separate the two phases. The surface tension of ^{4}He is approximately 2.3 times as large as that of ^{3}He at 0.5 K (Wilks, 1967, pg. 422). As the temperature goes toward absolute zero, the ^{3}He rich phase approaches pure ^{3}He, and the ^{4}He rich phase decreases the concentration of 3He asymptotically to 6.4% ^{3}He. Thus, separation of these two phases via their differences in surface tension should be achievable.

A promising method for achieving phase separation for both binary liquid and liquid-vapor systems involves using a porous metallic matrix to retain liquid in zero gravity. Such a trapping method has been successfully demonstrated by ACE, Inc., for pure ^{3}He for the -1-g "inverted" configuration (see NASA contract # NAS8-35254 Final Report, Long Lifetime, Spaceborne, Closed Cycle Cryocooler). In this application, the porous matrix or "sponge"

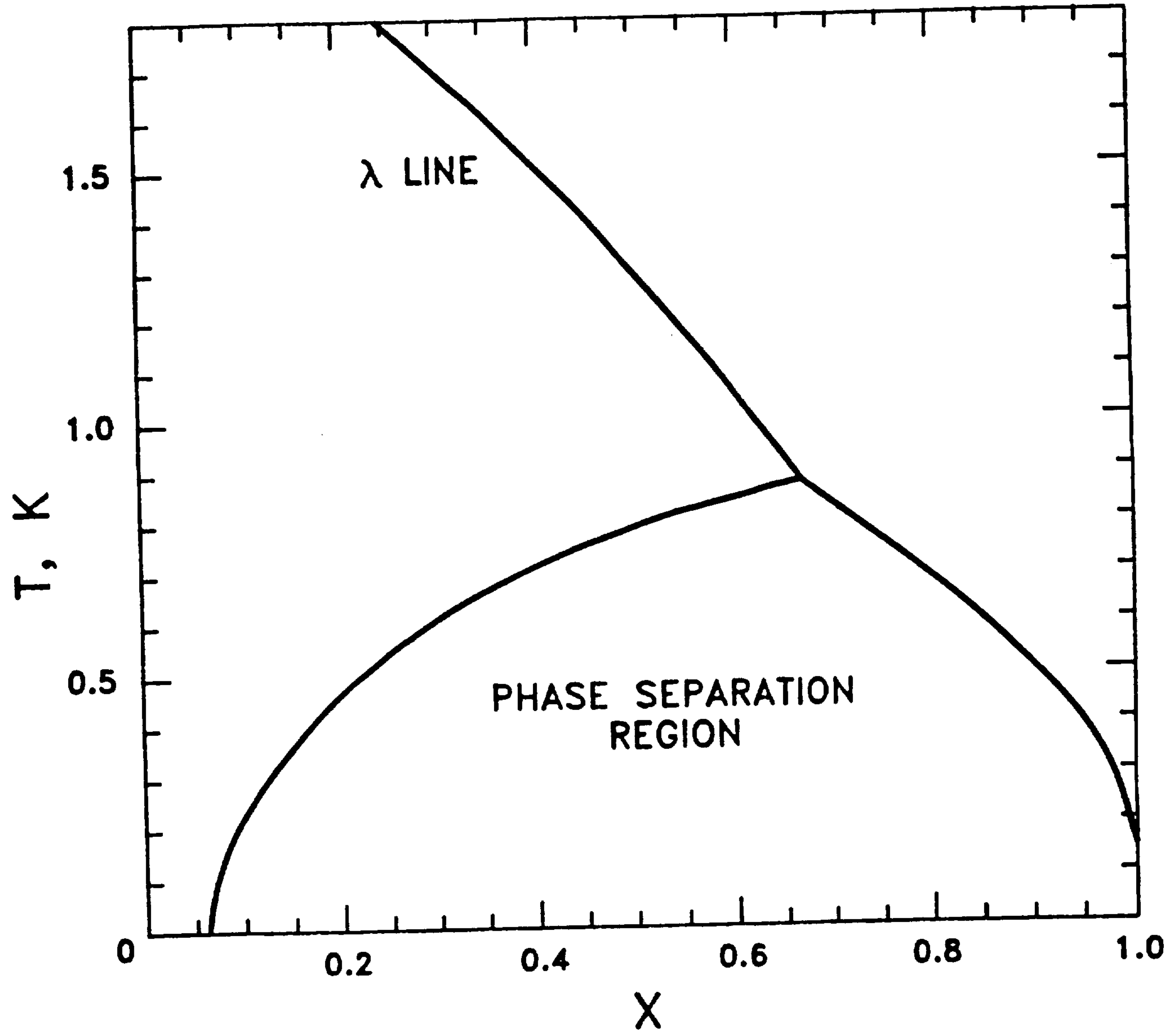

Figure A-1 The ^{3}He/^{4}He Phase Separation Diagram
($x = n_3/n_3 + n_4$)

was used to trap liquid in a liquid-vapor phase separator. This sponge corresponds to the still phase separator (SPS) discussed earlier.

Since ^{3}He has the lowest surface tension of the two isotopes, successful trapping of ^{3}He indicates that trapping should be achievable for either the ^{3}He rich phase or the ^{4}He rich phase. Since the ^{4}He rich phase has the higher surface tension, it should cling more strongly to the porous material. The exact distribution between the ^{3}He rich and ^{4}He rich components within the pores is difficult to predict beyond the expected preferential attraction of ^{4}He to the pore walls.

Phase separation between liquid and vapor must occur in the still of the dilution refrigerator. In the still, pumping is applied to the liquid mixture of ^{3}He-^{4}He to remove the ^{3}He from the still for recirculation. Because of the high partial pressure of ^{3}He at this temperature, nearly pure ^{3}He is removed from the still. This ^{3}He is circulated and introduced to the mixing chamber to provide the cooling action of the refrigerator. In zero gravity, the liquid must be prevented from escaping the system through the pumping lines. Furthermore, the method used to achieve liquid-vapor phase separation in the still must not interfere with the evaporation of the ^{3}He. To explore the idea of using the porous matrix to trap the mixture in the still, experiments with ^{3}He-^{4}He mixtures as the working fluid were conducted using the -1-g test apparatus that was designed for the pure ^{3}He experiment. What follows now is a description of this test apparatus, followed by a presentation of the mixture test data.

Test Facility and Instrumentation

This section will describe the test facility that was used for the surface tension phase separation tests for both pure ^{3}He and ^{3}He-^{4}He mixtures and for the solution refrigerator tests. A description will be given of the basic cryostat and dewar configuration, as well as the electronic instrumentation.

Basic Cryostat. Figure A-2 shows a schematic diagram of the basic cryostat configuration. The cryostat consisted of a 6 liter liquid helium pot which was suspended in a liquid nitrogen cooled Cryofab, Inc., model CSM-85 dewar. The space around the ^{4}He pot was supported from the dewar top flange. A thin walled stainless steel pumping line served as a helium vapor exhaust port. A large capacity mechanical pumping system was used to pump the ^{4}He pot down below the lambda transition to a minimum temperature of 1.4 K. At the bottom of the superfluid pot 4 mini-conflat connectors made access to the liquid helium in the pot possible. These connectors were welded to the pot and provide access to the liquid. The ^{4}He pot had a removable copper radiation shield attached to it to protect the experimental space from radiation leaks due to the dewar's 77 K walls. Copper radiation baffle plates attached to the pumping line reduced radiation leaks to the pot from the room temperature dewar top flange.

All electrical leads were of 0.005" manganin wire, and were passed through the dewar top flange via room temperature ceramic feed-throughs. These leads were well heat sunk on the pumping line. This made use of the cold helium gas being removed from the system to minimize the heat leak to the

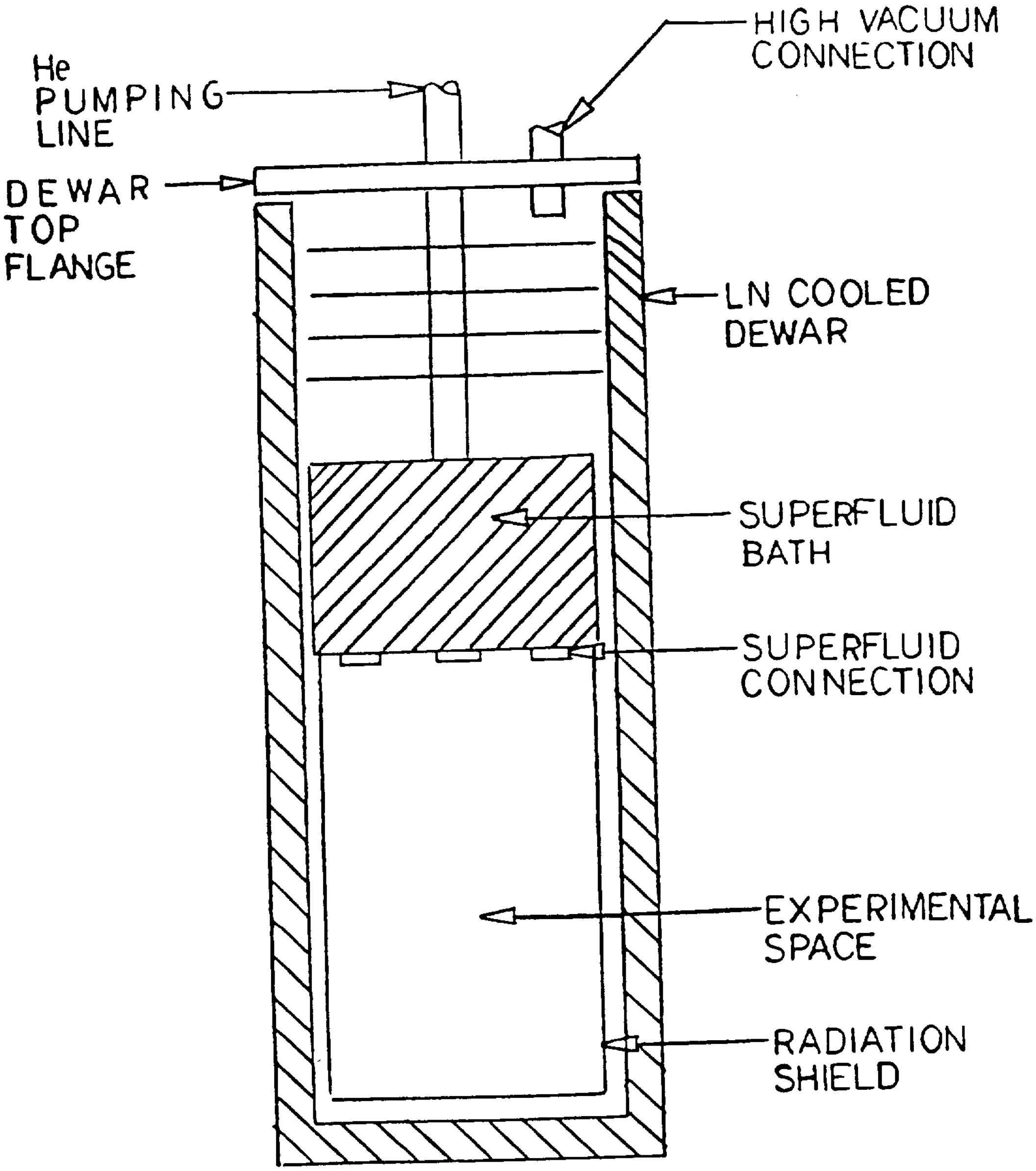

Figure A-2 Schematic view of cryostat.

pot via the leads. All capillaries and electrical leads were also well heat sunk to the superfluid pot itself.

When the cryostat radiation shield was in place, a thermal blanket consisting of 20 layers of NRC-2 superinsulation was wrapped around the pot and radiation shield to reduce the radiation leak to the pot from the 77 K dewar walls. The pot walls and radiation shield were also covered with a single layer of 3M No. 425 aluminum tape. Shu, Fast and Hart (1986) have shown that this combination of superinsulation and aluminum tape can significantly decrease heat leaks in cryogenic environments. With these precautions taken, the 6 liter helium pot could hold liquid for up to 24 hours.

The experimental space inside the copper radiation shield was a cylindrical chamber 7" in diameter and 11" in length. This space provided adequate room mounting the cryocooler and associated hardware.

One final feature of the cryostat design that facilitated modification of the apparatus was that the entire cryostat could be decoupled from support vacuum lines and electrical leads and be lifted from the dewar. Also, the dewar could be lowered as an optional method of obtaining access to the experimental space.

Electronic Instrumentation. The heart of the electronic instrumentation system is a Biomagnetic Technologies Potentiometric Conductance Bridge (PCB). This bridge was used to measure the resistance of Cryocal Model CR100 and Lake Shore Cryotronics Model GR-200A-100 Germanium Thermometers. The PCB applies very small load currents (picowatts) to the resistors, and thus avoids self-heating in the thermometer elements.

Hastings ST Series mass flowmeters were used to measure the helium gas flow rates. These gauges give a 0-5 volt D.C. output that is linear with mass flow over their calibration range; also, these devices are pressure independent. Setra Pressure gauges were used to monitor pressures to the system. These gauges give out a 0-5 volt D.C. voltage that is linear with pressure.

Pumping and Gas Handling System. In Figure A-3 a schematic representation of the pumping system is given. In normal operation, the nearly pure ^{3}He vapor was removed by the pump via the "out" port of the sponge chamber. The vapor then passed through a low impedance cold trap designed to prevent back streaming of pump oil into the chamber. The pumping speed is then regulated by the block and metering valves shown at the pump inlet. After passing through the Alcatel Model 2012H hermetically sealed pump, the ^{3}He vapor passes through an oil mist eliminator, and into a charcoal cold trap. The output of the pump is then measured with a Hastings ST-10 mass flowmeter. The gas then enters the main body of the gas panel, where pressure monitoring is done with Setra pressure gauges. After passing through a needle metering valve, the ^{3}He enters a Hastings ST-10 mass flowmeter, and then back into the cryocooler via a return line. This is the typical configuration used during a continuous cycle run.

Other important features of the system are a 37.4 liter storage volume, where the ^{3}He-^{4}He sample is stored. A Metal Bellows hermetically sealed pump

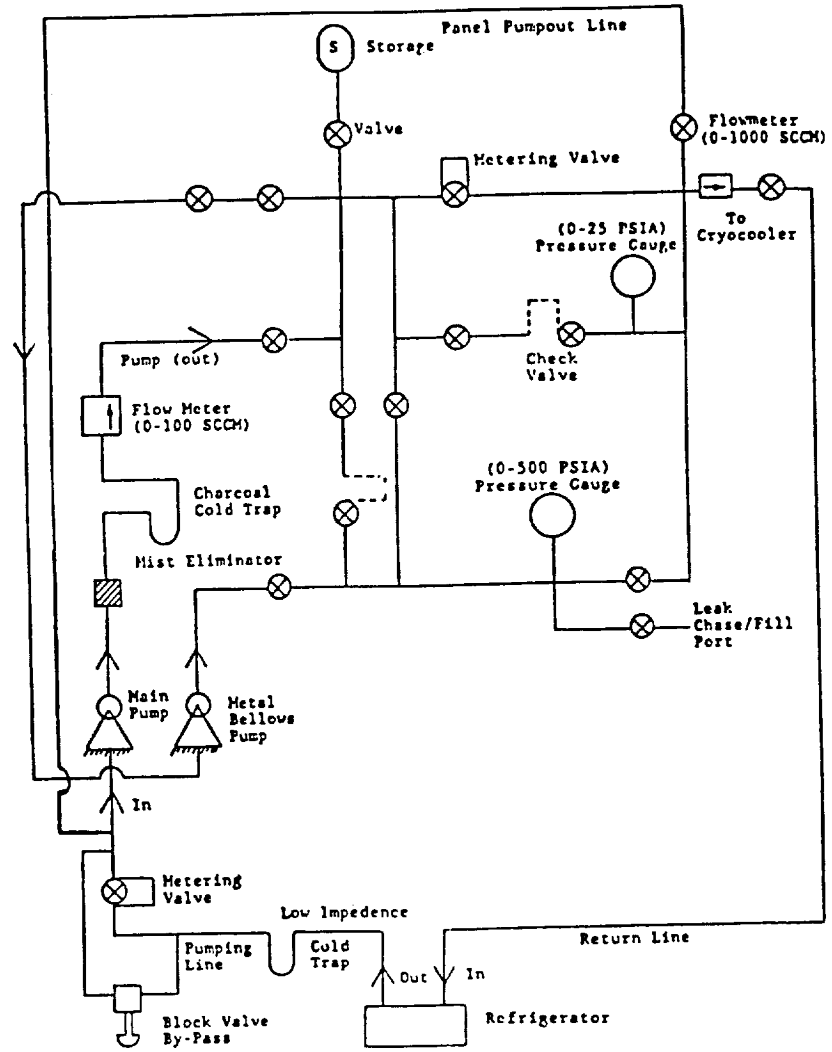

Figure A-3 ^{3}He-^{4}He mixture gas handling and pumping system.

is also attached to the gas handling panel to facilitate removal of the gas from the storage can during its condensation into the trapping sponge. This gas handling and pumping system offered great flexibility in controlling the refrigeration cycle and in monitoring system parameters.

Porous Metallic Sponge Assembly

Figure A-4 shows a scale drawing of the trapping sponge, pumping line, and return capillary. ^{3}He-^{4}He mixtures entered the system via a 10 feet length of coiled stainless steel capillary that passed through one of the superfluid pot pumping lines. This scheme is designed to make use of the cold ^{4}He vapor coming out of the superfluid pot to precool the incoming gas in the capillary. The capillary is coiled to increase its total surface area, thus improving heat transfer.

After it passed through the pumping line, the capillary entered the ^{4}He bath, where it was well heat sunk to the bath temperature by contact with the high conductivity superfluid. In this region of the capillary, the incoming gas condensed to form liquid, which trickled down below the bath.

After passing through the bath, the liquid then dripped into the upper chamber, where it was trapped by the porous sponge. A 1/2" diameter thin walled stainless steel pumping line was used to remove the vapor from the cryocooler. This line was firmly heat sunk to the 1.5 K bath at ^{4}He pot level, and had bends to eliminate radiation leaks from room temperature. Finally, the 1.5 K radiation shield described in the cryostat section surrounded the entire sponge assembly to block radiation leaks from the 77 K

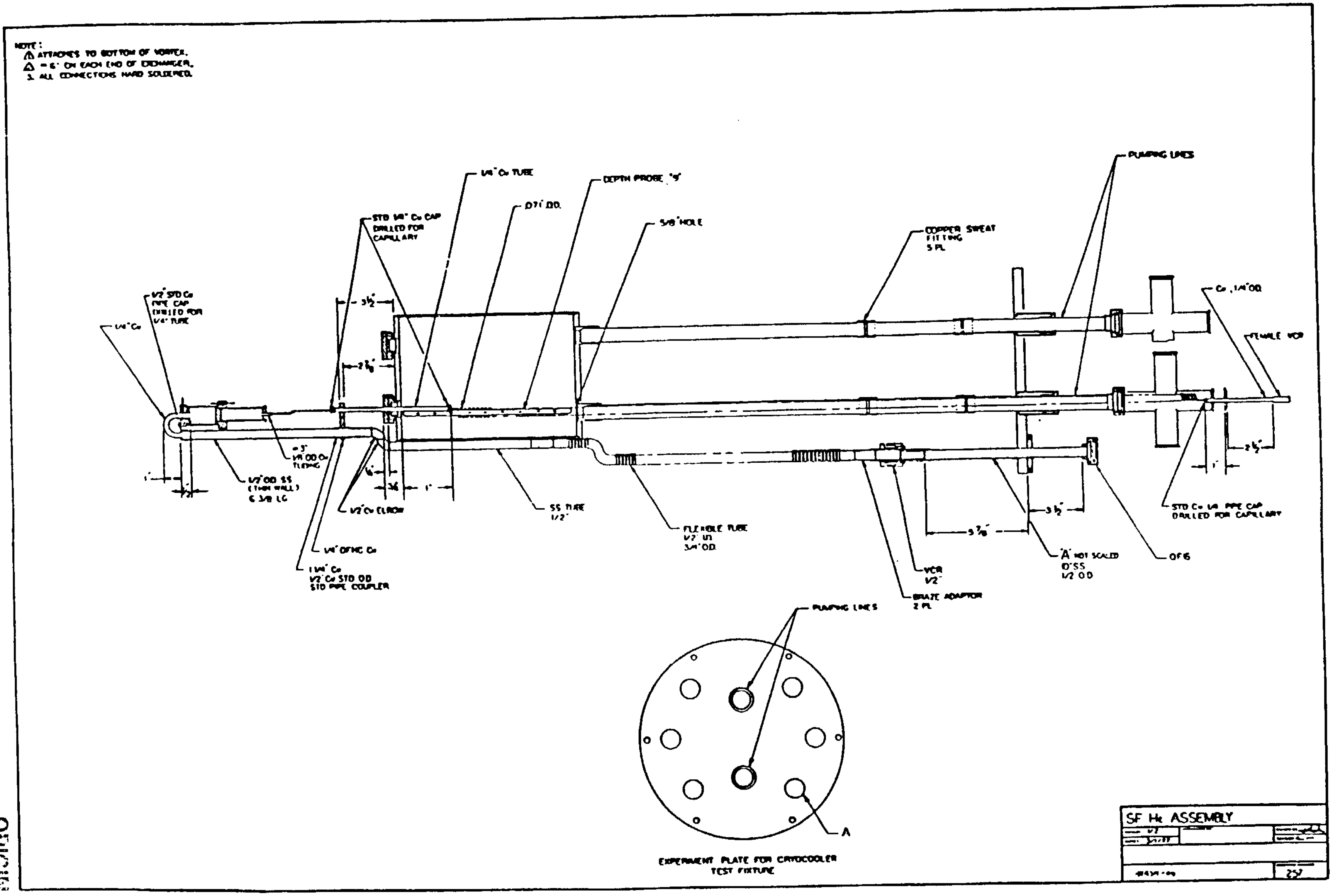

Figure A-4 Scale drawing of ^{3}He trapping sponge test cryostat.

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walls of the nitrogen dewar after moving through the bath. Then the fluid collected in the chamber above the sponge. This fluid was drawn into the porous silver trapping sponge by capillary action. Evaporation then occurred at the lower face of the sponge.

If a loss of trapping occurred, some or all of the fluid above the sponge would fall into the lower chamber. This chamber was thermally isolated from the liquid reservoir held above the sponge by the low thermal conductivity of its stainless steel walls. The lower chamber and U-shaped tube below the chamber were all made of copper and were isothermal. Thus, the device is schematically represented as previously shown in Figure A-5. Presence of liquid in either the upper or lower chamber is determined by applying heat loads to heaters shown and measuring the temperatures of the chambers with resistive thermometers.

Porous Trapping Plug

The porous sponge used in the device was prepared in the following fashion. First, 400 Å silver powder was compressed to 3000 psi with a hydraulic press. The powder was contained in a stainless steel jig and compressed with a stainless steel piston. This jig produced compressed cylindrical silver plugs approximately 1/4" in thickness and 3/4" in diameter. These plugs were then placed in a vacuum oven, which was evacuated and then backfilled with helium gas. The plug was then heated to 200 C in forty minutes and kept at 200 C for 1-1/2 hours. At this time, the oven was turned off and allowed to cool slowly to room temperature. This method was similar to that used by Franco (1984), and resulted in plugs with a packing fraction

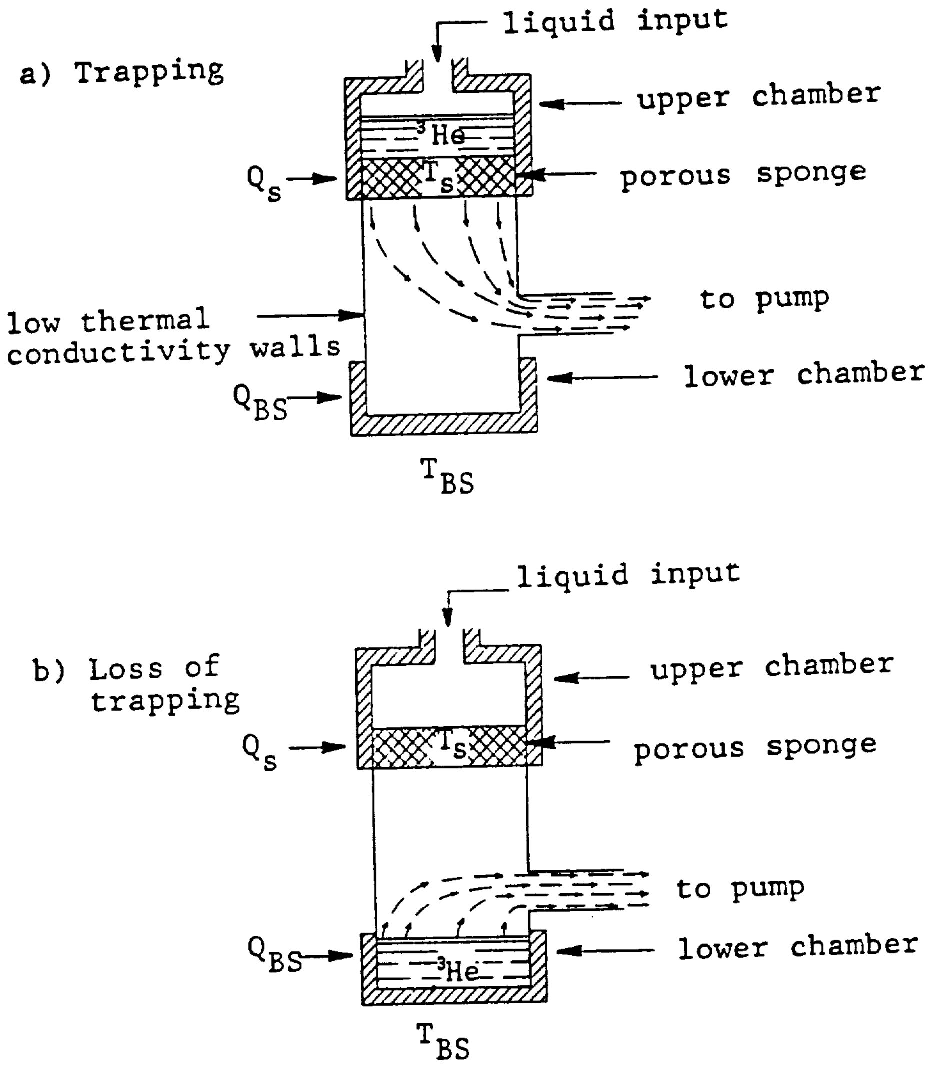

Figure A-5 Schematic view of trapping sponge holder
(a) retention of liquid against gravity (-1-g) is shown.
(b) depicts the situation occurring with a loss of liquid trapping.

of approximately 50%. The resulting plug was then epoxyed into an OFHC copper plug holder with Stycast 2850GT epoxy.

Results and Discussion

In this section, the results of the -1-g trapping tests of ^3He-^4He mixtures in the porous silver sponge will be discussed. After the data is presented, conclusions drawn from the tests will be given.

Preliminary Test: Pure ^3He. To verify that the trapping plug apparatus was working properly, tests were conducted with pure ^3He. These results agreed well with previous tests that demonstrated the sponge's ability to trap ^3He against gravity (See NASA Contract No. NAS8-35254). After condensing liquid ^3He above the trapping sponge, pumping from below the sponge was applied, and cooling occurred until a steady equilibrium was reached at $T \approx 0.6$ K. This temperature was maintained until the ^3He was completely exhausted from the sponge. The mass flow into and out of the sponge assembly was measured, along with the pressure above the sponge and the temperature of both the sponge (T_S) and the chamber below the sponge (T_{BS}). Loss of trapping could be detected by observing the drop of T_{BS} below T_S and by applying heat to each station. If liquid was present in either station, the mass flow was seen to follow the latent heat relation

$$\dot{Q} = \dot{m}L \qquad \text{(A-1)}$$

where $\dot{Q}$ is the heat input, $\dot{m}$ is the mass flow out, and L is the latent heat of vaporization. Using this technique, loss of liquid trapping or film flow through the plug could be easily detected.

Trapping Plug Results for ^{3}He -^{4}He Mixtures. In Figure A-6, the temperature of the sponge station and the temperature of the station below the sponge are shown as a function of time for a test run with molar concentration X=0.5. Here we define the molar concentration x as

$$x = N_3/(N_3 + N_4) \tag{A-2}$$

where N_3 is the number of moles of ^{3}He in the sample and N_4 is the number of moles of ^{4}He. First, the sample was admitted above the sponge. Care was taken to avoid fractionation of the mixture so that the concentration of the sample would be well defined. After pumping was applied below the sponge the system rapidly cooled to $T \approx 0.6$ K and stayed constant in temperature for approximately one hour. During this time, nearly pure ^{3}He was being pumped from the system due to the high partial pressure of the ^{3}He in the mixture. It should be noted that the same minimum temperature was reached during this part of the run as was achieved using pure ^{3}He as the working fluid. This is because the ^{4}He acts like a mechanical vacuum due to its low partial pressure, and does not appear to interact with the mechanism of transport of ^{3}He through the sponge. During this phase, the sponge temperature is somewhat lower than the temperature below the sponge, indicating that liquid had not broken through. Liquid trapping was confirmed using the mass flow test described previously.

At $T \approx 160$ minutes, a sharp rise begins in the temperature of both the sponge and below the sponge. This temperature rise is due to the exhaustion of the ^{3}He from the liquid mixture, leaving only ^{4}He trapped by the sponge. Since the vapor pressure of ^{4}He is much less than that of ^{3}He at 0.6 K, the

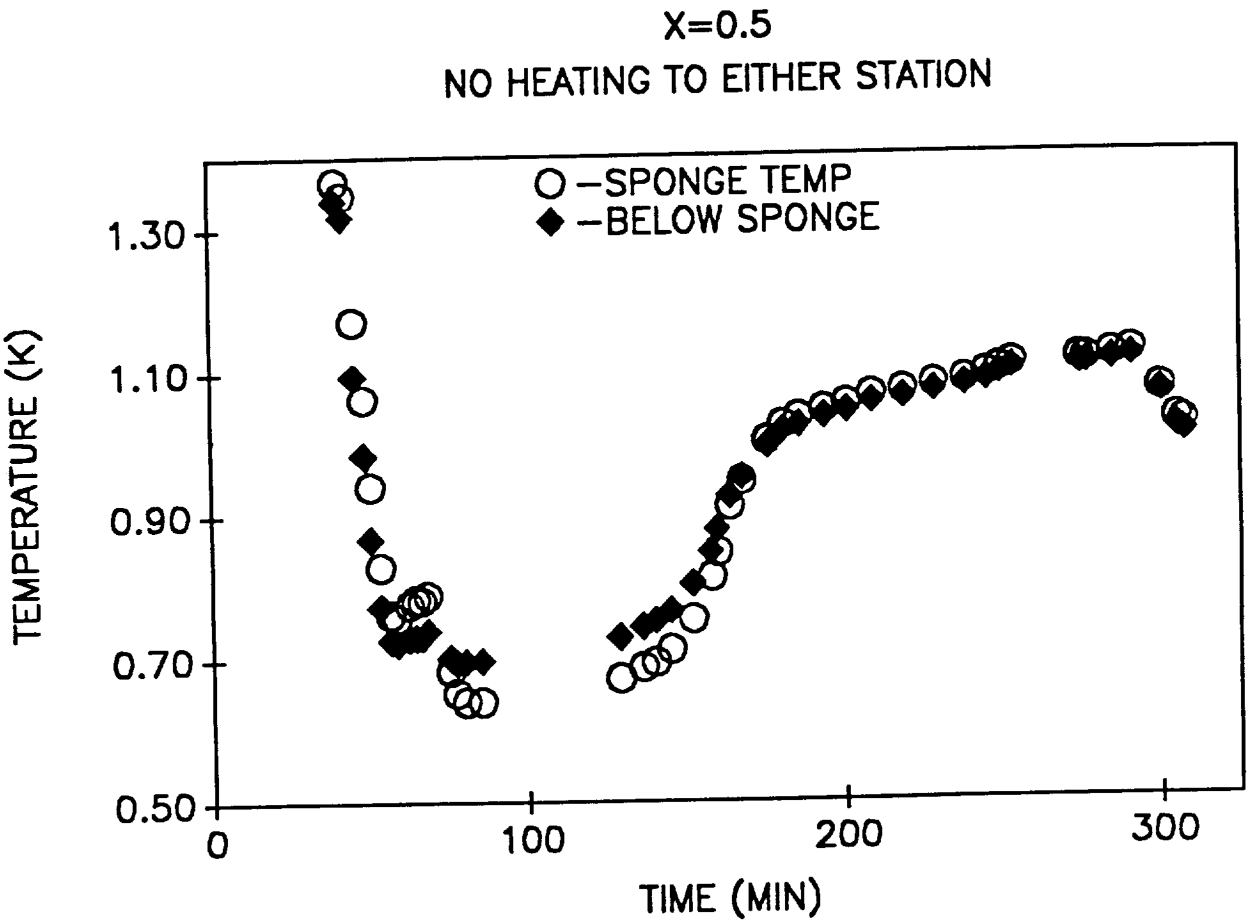

Figure A-6 Temperature of sponge and below the sponge versus time for a 50% ^{3}He-^{4}He mixture run.

system temperature rises due to the insufficient cooling power of the ^{4}He. Temperature equilibrium is again reached at $T \approx 1.0$ K, which is the often observed practical minimum cooling temperature of ^{4}He evaporative coolers. The slight drop in temperature at the end of the run is not well understood, but is thought to be due to a thin layer of ^{3}He which phase separated when the mixture was colder, and was excluded from the plug due to the surface tension of the ^{4}He rich phase. It should be noted that after the ^{3}He is exhausted from the system, the temperature of the station below the sponge decreases to a lower value than the sponge. Also, heat tests then reveal the presence of liquid in the station below the sponge. We believe that superfluid film flow accounts for these effects. When ^{3}He is present, the thermal conductivity of the liquid mixture is greatly reduced, and the temperature gradient across the sponge is relatively large. Thus, the sponge acts like a porous plug phase separator (See Hendricks and Karr, 1986) when ^{3}He is present, and all liquid is retained by the sponge. It is important that no film flow is seen when ^{3}He is present because downstream film flow in the still of dilution refrigerator serves to introduce heat leaks into the system and decreases the rate of ^{3}He being pumped from the still.

In Figure A-7, the pressure above the sponge is shown as a function of time for the same run illustrated in Figure A-6. After initially pumping down the system, the pressure reaches a minimum value and stays at that minimum throughout the duration of the run. This minimum pressure is somewhat higher than the vapor pressure at T_s due to the pressure drop across the sponge. The fact that the equilibrium temperature rises when the pressure remains constant is another direct indication that ^{3}He is pumped away from the mixture first.

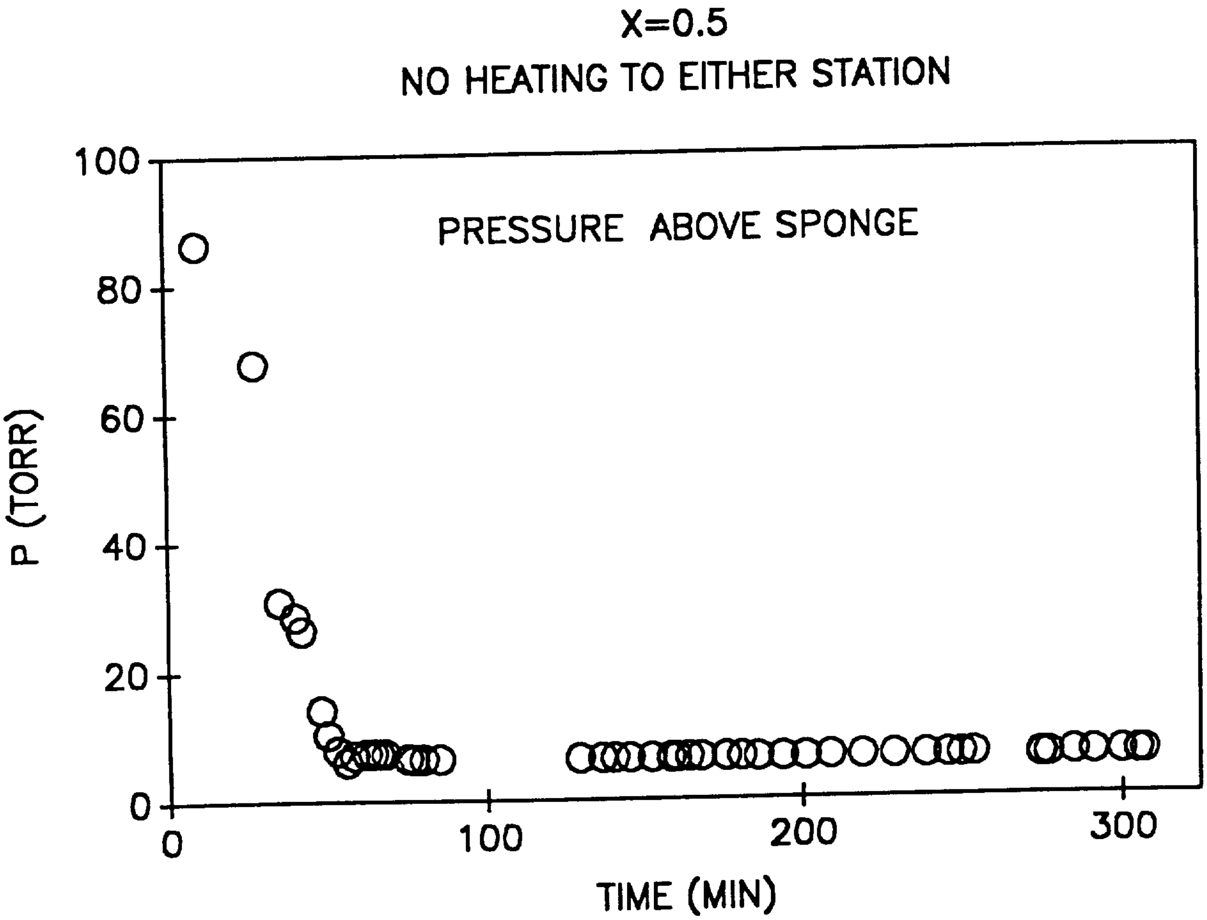

Figure A-7 Pressure above the sponge as a function of time for the 50% ^{3}He-^{4}He mixture test shown in Figure A-6.

Figure A-8 shows the results from another test of the X=0.5 mixture. Here, the general performance is the same as in Figure A-6, but a lower minimum temperature is reached. This graph is on a shorter time scale than Figure A-6; equilibrium at a higher temperature is not shown. Figure A-9 shows the pressure above the sponge as a function of time for this test run.

Additional tests were conducted with a mixture concentration of 75% ^{3}He (X=0.75). Very similar results were obtained with this concentration to the data from the X=0.5 test runs. The only difference in the results was that the portion of the run with $T \approx 0.6$ K lasted much longer, since more ^{3}He was available. These results substantiate the observation that all the ^{3}He is preferentially removed from the sponge before the ^{4}He in the mixture is expelled.

The previous results were obtained by filling the chamber above the sponge, shutting off the input mass flow, and then pumping on the sponge from below. In an actual dilution cryocooler application, continuous circulation of ^{3}He through the system is required to provide constant operation. To test the feasibility of continuous operation, the trapping sponge was filled with mixture, and pumping started from below while the output flow from the pump was brought to room temperature, sent back into the cryostat to recondense, and fed back into the chamber on top of the sponge. This arrangement led to a steady minimum temperature of the sponge that could be maintained indefinitely, since the ^{3}He was not depleted. In Figure A-10, typical results for such a continuous cycle run are shown. Equilibrium temperatures for each station are plotted as a function of heater power applied to that station for both the sponge and below the sponge. The rapid increase in the temperature

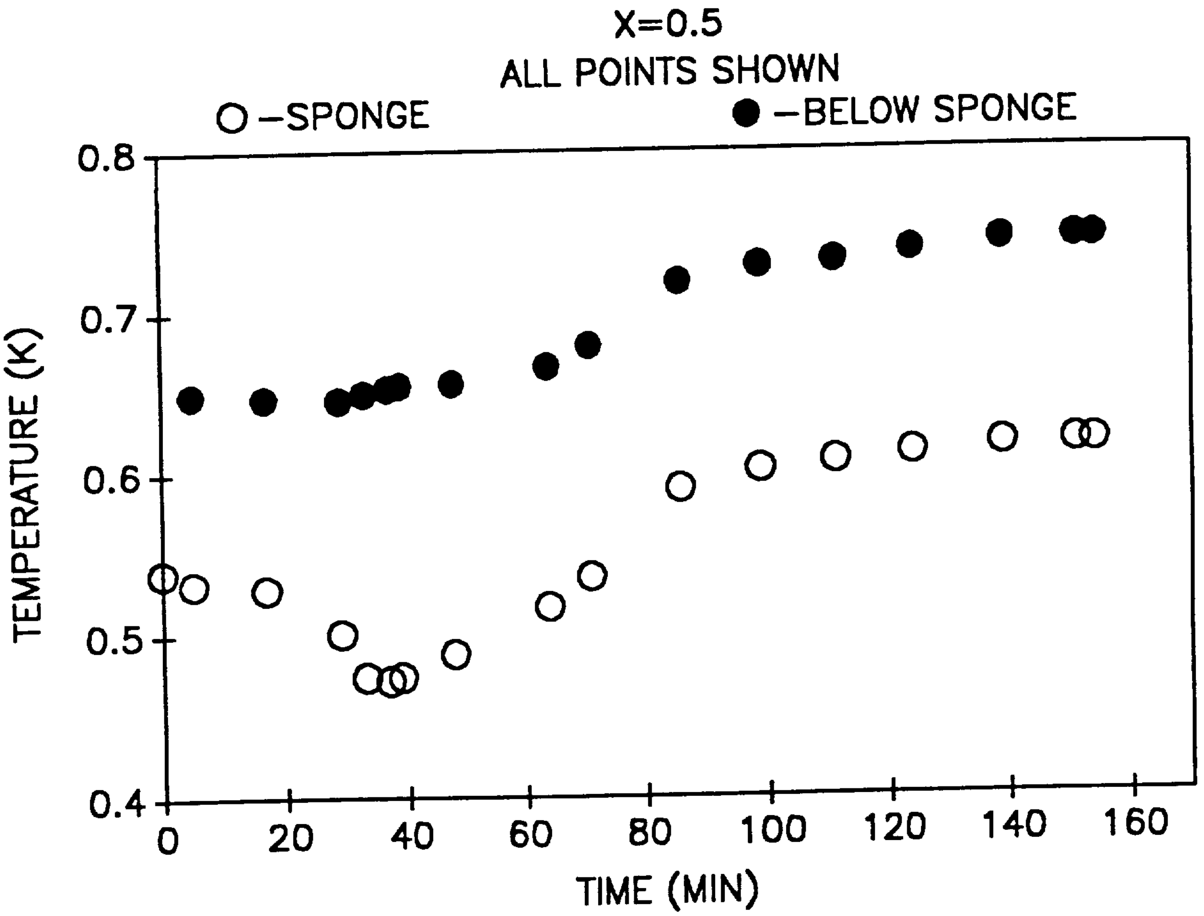

Figure A-8 Temperature vs. time for another 50% ^{3}He-^{4}He mixture test run.

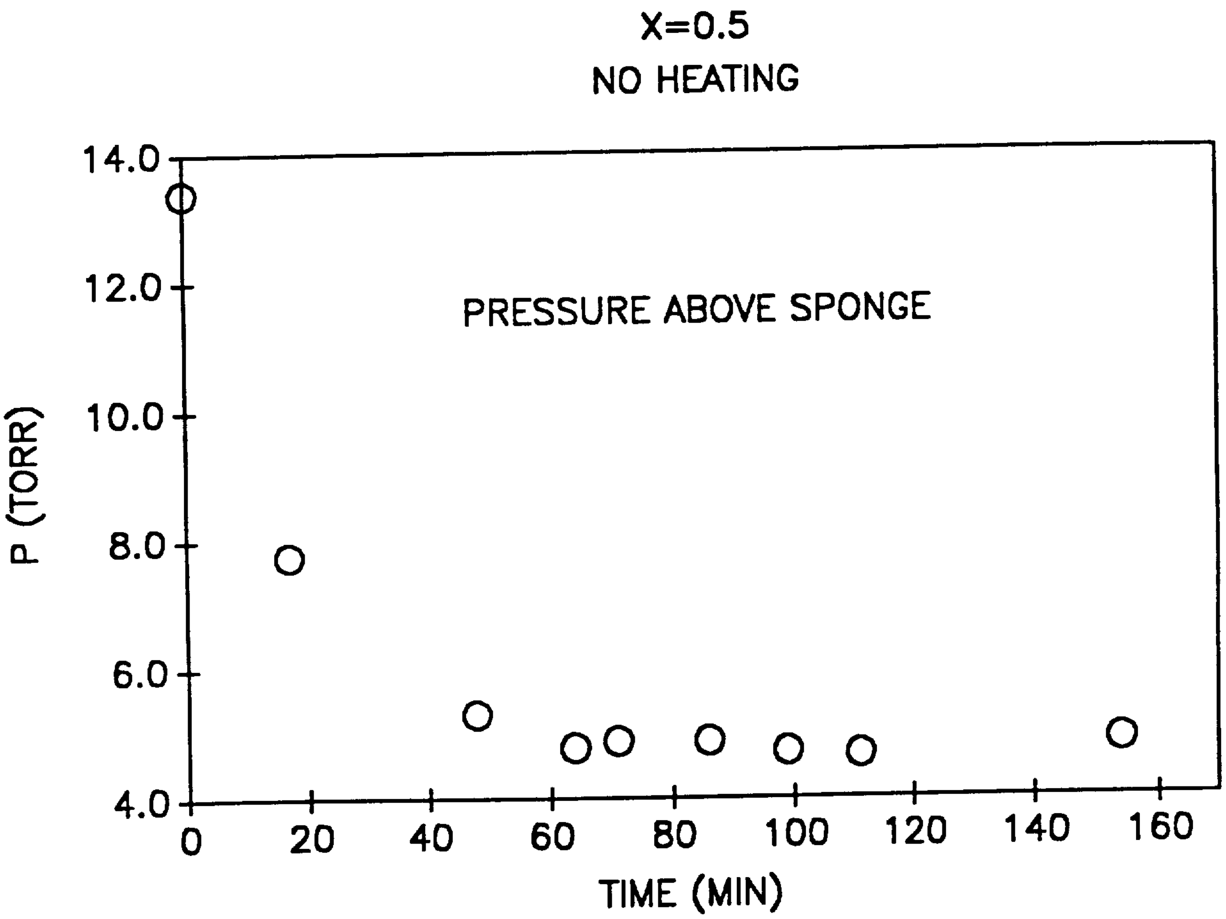

Figure A-9 Pressure above the sponge versus time for the run shown in Figure A-8

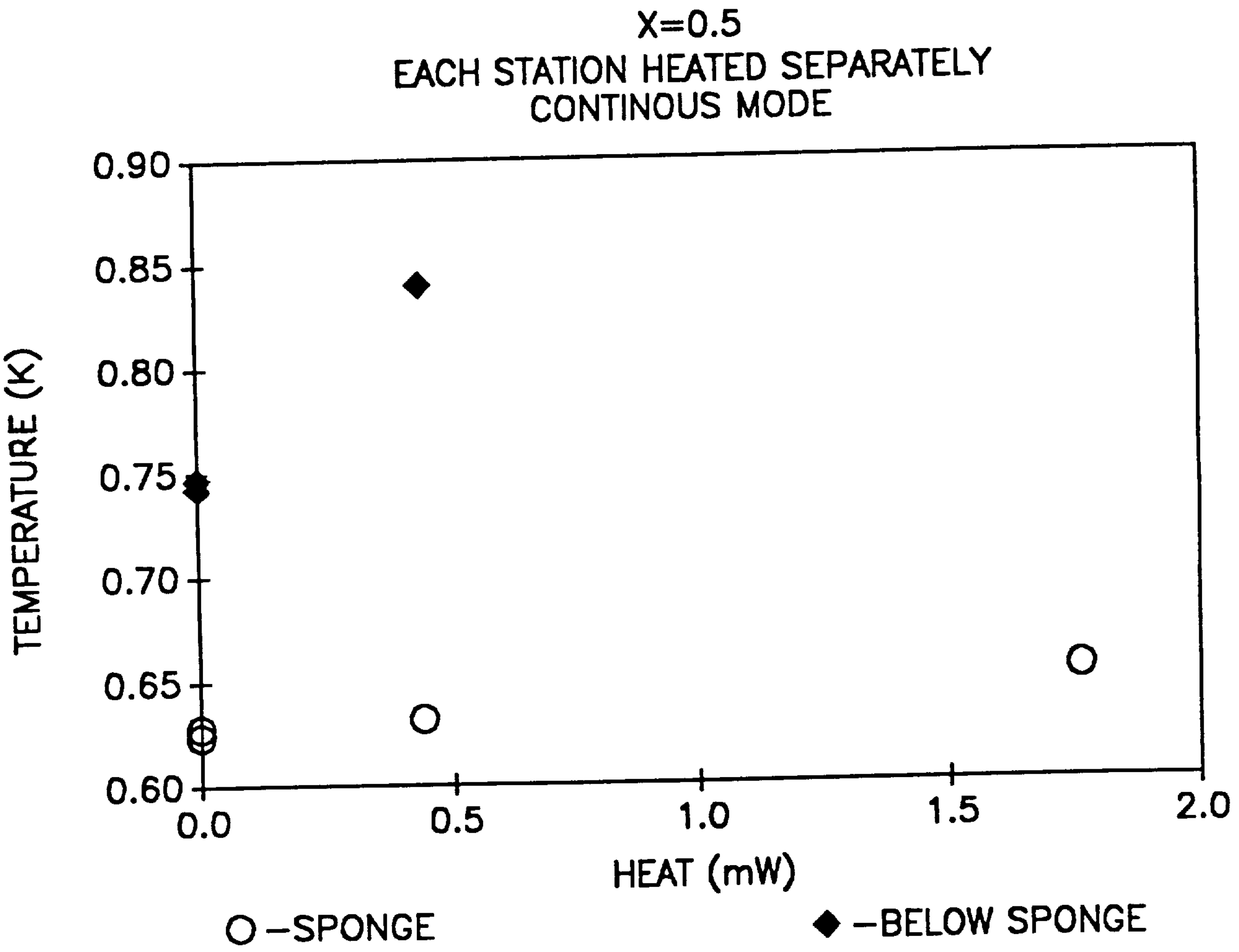

Figure A-10 Temperature versus heat input to each station when the sponge is running in continuous mode (mass flux in = mass flux out).

of the station below the sponge for a given input heat flux is proof that loss of liquid trapping has not occurred, and that the liquid mixture is being held above the sponge against gravity.

Conclusions

The following conclusions have been reached based on the results of the surface tension phase control test program for ^{3}He-^{4}He mixtures:

1. That a highly conductive, porous metallic matrix can be used to retain liquid mixtures of ^{3}He-^{4}He against the pull of gravity (-1-g) provided that the ^{3}He concentration of the mixture is not severely depressed.
2. The above result implies that the porous metallic matrix method can be used to achieve liquid-vapor phase separation in zero gravity, since zero-g operation is a less strenuous requirement than the inverted (-1-g) configuration.
3. That the above mentioned phase separation technique was successfully tested in a continuous operation mode, where ^{3}He was reintroduced as liquid above the porous sponge at the same rate ^{3}He vapor was drawn from the sponge by pumping from below.
4. The results listed above demonstrate the feasibility of operating the "still" portion of a ^{3}He circulation dilution cryocooler in zero gravity.
5. That the ^{3}He rich - ^{4}He rich phase separation that must occur in the mixing chamber of the dilution cryocooler can be accomplished using porous matrix phase separation techniques. It should be noted that direct testing of this concept was not carried out, but the differences

in the surface tension of the two phases indicates that it should be possible.

6. That a ^{3}He circulation dilution refrigerator that operates in zero gravity is a viable technology that can be successfully achieved.

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16. Abstract

The purpose of this effort has been to design, construct and test prototype dilution cryocoolers based on dilution refrigeration and adiabatic demagnetization refrigeration (ADR) cycles. Although devices we built and tested did not operate as fully functional dilution cryocoolers, important information was gathered. The porous metal phase separator was demonstrated to operate in the -1-g configuration; this phase separation is the critical element in a ^{3}He circulation dilution cryocooler. Improvements in instrumentation needed for additional tests and development have been identified.

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