A He\textsuperscript{3} CRYOSTAT FOR STEADY STATE NUCLEAR  
MAGNETIC RESONANCE MEASUREMENTS  
IN METALS

by

MANFRED PAUL PULS  
B.A.Sc., The University of British Columbia, 1964

A THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE  
REQUIREMENTS FOR THE DEGREE OF  
MASTER OF APPLIED SCIENCE

in the Department  
of  
PHYSICS

We accept this thesis as conforming to the required standard

THE UNIVERSITY OF BRITISH COLUMBIA  
May, 1966
In presenting this thesis in partial fulfilment of the requirements for an advanced degree at the University of British Columbia, I agree that the Library shall make it freely available for reference and study. I further agree that permission for extensive copying of this thesis for scholarly purposes may be granted by the Head of my Department or by his representatives. It is understood that copying or publication of this thesis for financial gain shall not be allowed without my written permission.

Department of **Physics**

The University of British Columbia
Vancouver 8, Canada

Date **April 5, 1966**
A He³ cryostat was designed for steady state nuclear magnetic resonance measurements. The cryostat required about one liter of He³ at NTP. This amount of He³ was liquified at 1.2⁰K, which was the temperature of the surrounding liquid He⁴ bath, and then pumped on by means of a four-stage mercury diffusion pump. By this procedure, a liquid He³ temperature of 0.35⁰K was achieved and maintained for four hours. The r-f coil and sample were in direct contact with the liquid He³ to ensure sufficient heat transfer between the two. The temperature of the liquid He³ was measured by means of vapour pressure measurements of the evaporating He³ and by means of resistance measurements of a carbon resistance in contact with the liquid He³. The system was non-recirculating, since a total, uninterrupted run of four hours was considered long enough for most experiments. The He³ could also be retransferred within 20 minutes after such a run, and this process could be continued until insufficient quantities of liquid He⁴ remained to cool the surroundings.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Title</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>ii</td>
</tr>
<tr>
<td>LIST OF ILLUSTRATIONS</td>
<td>iv</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENT</td>
<td>v</td>
</tr>
<tr>
<td>CHAPTER 1 INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>CHAPTER 2 EXPERIMENTAL METHODS AND APPARATUS</td>
<td>3</td>
</tr>
<tr>
<td>APPENDIX CRYOSTAT</td>
<td>23</td>
</tr>
<tr>
<td>HEAT LEAKS AND PUMPING SPEEDS</td>
<td>28</td>
</tr>
<tr>
<td>He³</td>
<td>36</td>
</tr>
<tr>
<td>BIBLIOGRAPHY</td>
<td>38</td>
</tr>
</tbody>
</table>
# LIST OF ILLUSTRATIONS

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Resistance measurement by Rubicon potentiometer</td>
<td>10</td>
</tr>
<tr>
<td>2</td>
<td>Schematic diagram of steady state spectrometer</td>
<td>16</td>
</tr>
<tr>
<td>3</td>
<td>Schematic of He$^3$ cryostat and associated vacuum system</td>
<td>18</td>
</tr>
<tr>
<td>4</td>
<td>Schematic of pumping lines and cans</td>
<td>24</td>
</tr>
<tr>
<td>5</td>
<td>Schematic of r-f coil, sample, carbon resistance and low temperature seals</td>
<td>26</td>
</tr>
</tbody>
</table>
ACKNOWLEDGEMENT

I wish to express my gratitude to Dr. D. Ll. Williams for the initial, helpful ideas and for his ready assistance throughout the course of this work, particularly in the later stages when he spent many long hours on helping with the completion of the experiment.

I also wish to thank G. A. deWitt for the many helpful discussions, and J. Lees for his advice and assistance and the construction of all of the glass equipment that went into the building of the cryostat. Further, I would like to thank all those members of the Physics Department, who, through their advice, helped me in carrying out this work.

This research was supported financially, in part, by the National Research Council through grants to Dr. D. Ll. Williams and by the Dean's Committee on Research through the award of assistantships to the writer.
CHAPTER 1
INTRODUCTION

Rapid advancements have been made in recent years in the field of nuclear magnetic resonance (NMR) in metals\(^1\). Although much of the earlier work had been done on powder samples, the size of which was much smaller than the electromagnetic skin depth, improvements in the signal to noise ratio of the electronic equipment made it possible to investigate NMR effects on metal single crystals. Some of the early work along this line was pursued by members of this laboratory on aluminum and tin at approximately \(1.2^\circ\text{K}\) liquid He\(^3\) temperature\(^2\). It was felt, that a further reduction in temperature would reveal, particularly in single crystal tin, oscillations in the Knight shift as a function of magnetic field, much the same sort of oscillations, known as de Haas–van Alphen oscillations, that are evident in the electronic susceptibility\(^3\).

It was with this main application in mind, that the He\(^3\) cryostat to be described in the subsequent pages was designed. However, any other experiment using steady state methods and requiring low temperatures could equally well be done with it. Mostly the same electronic equipment, although somewhat improved, was to be used as in the earlier experiments by Jones and Williams\(^2\), with the notable exception of the magnet. Here, a higher field magnet was thought more desirable, since it would, as well as the lower temperatures, aid in increasing the size of the oscillations.
and facilitate their detection. It would also, of course, increase the overall size of the signal. Unfortunately, an upper limit on the frequency of oscillations of the Pound - Knight - Watkins marginal oscillator to be used was about 19 Mc./sec. for optimum signal to noise operations. This frequency corresponds to a magnetic field strength for tin of only approximately 12 Kilogauss, which is only a small increase in field strength over the previously used maximum value of 10.1 Kilogauss.

The He$^3$ cryostat was designed for an uninterrupted run of about 3 - 4 hours at approximately 0.4$^0$K. The He$^3$ could also be retransferred after such a run, and this could be done within a timespan of about 20 minutes. Two such runs were then usually possible, the limitations for a third run being the liquid He$^4$ level.

In Chapter 2 the principle of the cryostat is explained and its features described to give a fairly complete overall picture. Some problems with the magnet and its use are mentioned and a brief description of the electronics is given. A typical He$^3$ run is described, and an estimate made of the ultimate temperature obtained with the liquid He$^3$ as well as the length of the run. The Appendix gives a more detailed description of the cryostat and further, detailed heat leak and pumping speed calculations. The accuracy of these calculations is also investigated.
CHAPTER 2
EXPERIMENTAL METHODS AND APPARATUS

Introduction

Liquid He$^3$, an isotope of He$^4$, has the advantage of a much higher vapour pressure at low temperatures. For example, at 1.2°K, where the vapour pressure of liquid He$^4$ is 0.63 mm. Hg, that of He$^3$ is 19.8 mm. Hg$^4$. Hence, while a reasonable limit to the temperature of liquid He$^4$ that can be obtained by pumping on its vapour would be about 1.2°K, temperatures of 0.4°K to 0.3°K can usually be expected when pumping on liquid He$^3$. Liquid He$^3$ can thus provide another factor of four lower temperatures and would be useful as a coolant in the range 0.4°K to 0.3°K. A difficulty in using He$^3$ as a coolant is that He$^3$ costs about $150 a liter at NTP and hence economy demands that only a small amount of the gas be used. Further, care must be taken that the He$^3$ will circulate in an isolated and leaktight part of the system. Since part of the He$^3$ will be immersed in liquid, superfluid He$^4$, very stringent conditions are consequently put on the vacuum tightness of this part of the cryostat.

It was thought from the beginning to design as simple a system as possible. Approximately one liter ( NTP ) of He$^3$ was stored in cans at low enough pressure that a four-stage mercury diffusion pump could alternately pump the gas into the condenser pot, or pump on the vapour when the He$^3$ had condensed. Temperature measurements of the
liquid He$^3$ were done by vapour pressure thermometry, using an oil manometer for the high pressure and a McLeod gauge for the low pressure measurements. A resistance thermometer was used as a check. The main part of the apparatus consisted of three storage cans: two of 5 liter capacity and one of 15 liter capacity in each of which He$^3$ was stored at a pressure of 35 mm. Hg. A four-stage mercury diffusion pump with a liquid nitrogen trap was used to pump the He$^3$ from the cans into the condenser pot, and alternately, to pump on the vapour above the liquid He$^3$ after it had condensed, with the cans used as the backing volume. No recirculation was used as enough He$^3$ was available to make a reasonably long run. After all the He$^3$ had been boiled off, it was recondensed and this was continued until insufficient quantities of liquid He$^4$ were available for proper cooling of the surroundings. An overall schematic diagram of the cryostat is given in Fig. 3.

Cryostat

The main features required of the cryostat were

1) Good thermal isolation of the He$^3$ condenser pot from the liquid He$^4$.

2) Sufficiently fast pumping speed along the He$^3$ pumping line to take care of the evaporating He$^3$.

3) Geometry of tubes and cans such that the cryostat would fit inside a 2.24 in. magnet pole face gap.

A detailed description of the cryostat is given in the Appendix. In the following sections only the main features will be described and some of the reasons behind
The Sample Holder (Condenser Pot)

As shown in Fig. 3, as well as in Figs. 4 and 5 in the Appendix, the sample holder was situated at the bottom of the dewar and provided a place for the He\textsuperscript{3} to condense. It contained the r-f coil and sample and a carbon resistance for temperature measurements. To ensure that the condensed He\textsuperscript{3} would flow around the sample a lucite filler was used to surround and support the sample. The sample holder was thermally isolated from the He\textsuperscript{4} bath by means of a vacuum jacket.

Pumping Lines

The sample holder and vacuum jacket were suspended by means of two pumping lines. One of them extended through the vacuum jacket into the sample holder, while the other opened into the vacuum jacket. To cut down the heat leak between the top of the vacuum jacket, which was in contact with the liquid He\textsuperscript{4} bath, and the sample holder at liquid He\textsuperscript{3} temperatures, the material used for the tubes was thin-walled stainless steel, which has an extremely low thermal conductivity and good strength. Room temperature radiation funnelling into the condenser pot was eliminated to a degree by placing radiation bends 8 in. above the top of the vacuum jacket into each of the pumping lines. The size of the tubes was chosen as large as possible, the diameter of the tubes increasing towards the high temperature part of the dewar, to ensure sufficiently fast pumping speeds along the tubes. Unfortunately, an increase
in the tube diameters increases the heat leak, which is a linear function of the cross-sectional area of the tube walls, and a reasonable compromise had to be made. The size of the tubes was further limited by the size of the lids on the vacuum jacket and condenser pot, the larger ( outer ) lid being only about one inch in diameter. Fig. 4 of the Appendix gives a schematic of the above mentioned tubes.

**Coaxial Cable**

A fairly long coaxial cable passed from the dewar cap to the lid of the vacuum jacket. The cable was 23 in. long and consisted of a \( \frac{3}{4} \) in. OD stainless steel tube which constituted the outer shield of the cable, and a \#34 copper wire spaced inside the stainless steel tube by means of teflon spacers. Holes were punched into the tube to allow the helium to flow into it. This was to eliminate the necessity for a superfluid seal and the consequent dangers of a leak, and hence, possible thermal oscillations which could have occurred otherwise. The wire was passed through the two cans by means of platinum to glass seals. No shielding of the wire was considered necessary inside the cans. At the dewar cap the coaxial cable was passed through the lid by means of a Kovar seal, the wire terminating in a housing on top of the cap that contained a BNC connector. A disadvantage of the coaxial cable was its length, which introduced additional and unwanted capacitance and inductance into the oscillator circuit, and
consequently resulted in a loss in signal. However this was not too serious for the material that was studied, since the signal was quite large at liquid He\textsuperscript{3} temperatures anyway. To reduce the amount of heat leaking into the sample holder from the liquid He\textsuperscript{4} bath, the center wire used here was made of \# 34 manganin wire, rather than the copper wire used for the rest of the cable. Manganin has about the same thermal conductivity as stainless steel at low temperatures, while its electrical conductivity is good enough to introduce a resistance of less than one ohm for a one inch length of wire.

The total amount of heat leaking into the sample holder was calculated in the Appendix to be about 110 ergs/sec. In practise, however, it was found that the true heat leak was closer to 1500 ergs/sec.

**Temperature Measurement**

Since a high degree of accuracy in measuring the final temperature of the He\textsuperscript{3} bath, and consequently of the sample, was not required, the simplest of temperature measurement methods were used. The methods used were

1) Vapour pressure measurement of the He\textsuperscript{3} vapour above the liquid.

2) Resistance measurement of a carbon resistance immersed in the liquid He\textsuperscript{3}.

These will now be described.

A most reliable way of measuring the temperature of the He\textsuperscript{3} bath was to measure the vapour pressure above
it. To simplify the construction of the apparatus, the tube coming from the manometers, however, was simply joined to the He\textsuperscript{3} pumping line at the top, i.e. at the dewar cap. Hence the He\textsuperscript{3} vapour pressure that was measured was that at the top of the pumping line, and allowances had to be made for the pressure rise down the pumping line due to the pumping action. Such a calculation was made in the Appendix.

Since the pressure to be measured ranged from about 19 mm Hg to a few microns of Hg, two gauges had to be used. One was an oil manometer used for the high pressure range, and the other a McLeod gauge that had been especially designed to measure within the range microns of Hg with reasonable accuracy. The total range of this gauge was from 3.67 mm Hg to 6.37 \times 10^{-5} mm Hg. However, the low pressure reading cannot be taken as too accurate. The vapour pressure values of the liquid He\textsuperscript{3} bath thus obtained were then used to get the corresponding temperatures by using a vapour pressure – temperature table for He\textsuperscript{3} that had been experimentally determined by Roberts and Sydoriak\textsuperscript{4}. The pressures given in this table are the pressures of the He\textsuperscript{3} vapour just above the liquid, while the pressure usually read by a manometer are at room temperature. Since the temperature difference between the liquid He\textsuperscript{3} bath and the manometer was considerable, a correction would have to be applied, if the tubing from the manometer reached all the way down to the condenser pot, to the actual manometer readings, to obtain the true equilibrium He\textsuperscript{3} vapour pressure above the bath. This is the thermomolecular
pressure correction, which applies for low vapour pressures, small connecting tubes, and a large temperature difference between the manometer and the bath. However, since the manometer tube was actually only attached to the pumping line at the dewar cap where the temperature of the He\textsuperscript{3} vapour was close to room temperature, no thermomolecular pressure correction would be necessary for the section of the tube connecting the McLeod gauge to the dewar cap. A correction would be needed for the tubing inside the dewar reaching down to the condenser pot, but this was actually already included in the pressure rise calculations mentioned previously and given in the Appendix.

The most useful temperature measuring device was the carbon resistance, since it could be used to measure temperature not only at He\textsuperscript{3} temperatures, but also of the inside of the dewar while it was being cooled with liquid nitrogen and liquid He\textsuperscript{4}, respectively. The method consisted simply of measuring the resistance of a carbon resistance inside the inner can by means of a Rubicon potentiometer, as shown in Fig. 1. In order to read the potentiometer directly in ohms, the value of the current was set for a 100 mv. drop across the standard resistance. Hence the unknown resistance was obtained by using \( R_t = R_s \frac{E_t}{E_s} \), where \( R_s \) was always picked to be the nearest multiple of ten above the unknown resistance to be measured and where \( E_t \) and \( E_s \) were the voltages, respectively, across the unknown (low temperature) resistance and across the standard resistance. Hence \( R_t \) was obtained directly from
Fig. 1 Resistance measurement by Rubicon potentiometer

$R_t$ - Resistance to be measured inside sample holder

$R_s$ - Standard resistance

$S_2$ - Reversing switch

To output labelled EMF on Rubicon potentiometer

1.5V

To potentiometer
the value of $E_t$. $R_t$ was obtained at various standard temperature points such as the liquid nitrogen point, the liquid $\text{He}^4$ point at atmospheric pressure (4.2°K) and the liquid $\text{He}^4$ point at 1.2°K. At liquid $\text{He}^3$ temperatures, only the lowest value of pressure was correlated with the resistance measurement. The resistance used was a useful one for temperature work below 1°K, since it had its greatest change with respect to temperature in that range. Curves of temperature versus resistance of some Speer carbon resistances were published by Black, Roach, and Wheatley (subsequently designated by BRW) who determined which Speer carbon resistances and grades were most useful for work below 1°K. Of those found most useful, the 1002 grade, $\frac{1}{2}$ watt, 470 ohms nominal resistance, was used in this experiment. Hence it was possible to check the resistance measurement versus temperature value obtained in this experiment against the value obtained by BRW. It was found that the two agreed within the obtainable accuracy.

BRW also published data on the decrease of the resistance when the cylindrical axis of the resistance was placed at right angles to a magnetic field. They found that at 0.3°K and at a field of 10 Kilogauss the decrease was about 5%. Since the field used in this experiment was about 12 Kilogauss with a temperature of about 0.35°K the above correction factor was considered accurate enough to be useful in making the resistance measurement correction in this experiment.
A rough check was also made, to see if there would be an appreciable Kapitza resistance between the carbon-resistance-liquid He$^3$ interface and hence, if the resistance readings were appreciably higher than the actual bath temperature. Data by Lee and Fairbanks for a Cu-He$^3$ interface in the temperature range $0.3^0K$ to $2^0K$ were used to make the calculation. It turned out that for the power input of this experiment the temperature difference was completely negligible.

The r-f Coil and Sample

The sample investigated was a single crystal of tin, the same that had been studied by Jones and Williams in a previous experiment. To simplify measurements, it was desirable to obtain two signals close together. This was done by using two single crystals glued together, but whose 001 axes were oriented at $45^0$ to each other. Both crystals were of equal size, about $\frac{3}{4}$ in. long each. They were cut by means of a jeweller saw in a holder with paraffin wax used to support the crystal. The wax was used to eliminate the possibility of strain occurring in the crystal while it was being cut. After the two crystals were cut they were etched in nitric acid and examined for strain boundaries. About 25 turns of # 34 copper wire were wrapped around the sample, making sure that equal numbers of turns were around both crystals. When the sample was attached to the coaxial cable and the condenser pot lid, which
served as the ground, the approximate position of the two 001 axes was noted, to allow optimum orientation of the magnetic field with respect to the specimen.

**Pump**

A mercury diffusion pump was used to pump on the He$^3$ in the condenser pot and in the same token to pump the He$^3$ gas into the condenser pot. The pump was bought from the Edwards Company of England (tradename 2M4). The pump consisted of four stages and its principal advantage was that it could pump against a maximum backing pressure of about 35 mm. Hg with little reduction in its speed. Hence it was possible to eliminate the use of a mechanical rotary forepump and the consequent dangers of a leak. The speed of the pump was rated at between 30 - 35 liters/sec with a liquid nitrogen trap attached at its intake.

**Magnet**

The magnet used was purchased from the Magnion company and could be used with either 6¾ in. pole faces and a 1.8 in. pole face gap up to a field of about 23 Kilogauss, or 8¾ in. pole faces, a 2.24 in. Pole face gap and a maximum field of around 18 to 19 Kilogauss. Since fields of around 12 Kilogauss were used in the experiment, and it was necessary to have the 2.24 in. pole face gap, the second pole faces were used. Unfortunately, since the high field pole faces were on the magnet at the time, these had to be dismounted and the other ones put on.
This job required three people, physical strength, and a great deal of patience. A further disadvantage of having to change pole faces was that they had to be aligned for maximum field homogeneity. The adjustment could be made by means of tightening the three screws each which held the pole faces, and by adjusting the $\mu$-shims after a good homogeneity had been obtained. The $\mu$-shims were cylindrical pieces of metal that could slide in and out of the center of the magnet pole faces, the adjustment being made by means of a vernier. The most suitable equipment that was available in the lab for the initial alignment, was a 30 Mc/sec. pulse spectrometer designed by W. Hardy. Used with a deuterium sample, the resonance of deuterium was looked for on an oscilloscope. A pulse apparatus was useful for this task, since even with very poor homogeneity a signal can usually still be seen. After the signal was found the screws were adjusted, with both $\mu$-shims flush, until the signal indicated maximum field homogeneity over the 2 cm. long sample. The $\mu$-shims were not changed at this field, since the field at which maximum homogeneity was desired was around 12 Kilogauss and the $\mu$-shim setting would be different for that field. However, the tightness of the screws was not adjusted any further when looking for maximum field homogeneity at other fields. It was found that it was usually better to align the field at a higher field if no pulse apparatus was available to this at the field desired, and then use steady-state equipment for the lower field alignment.
For the 10 to 12 Kilogauss field alignment a Pound - Knight - Watkins marginal oscillator steady-state spectrometer designed by S. Sharma was used, with protons as the sample. A fairly large field modulation (approximately 7 gauss) was used in order to facilitate the seeing of the signal, and signal was received by means of an oscilloscope. Nevertheless, the signal was seen only after many laborious and futile field sweeps, which were necessary, since no reliable current versus field curves were available. After seeing the signal (which was almost undetectably small) the $\mu$-shims were adjusted until maximum homogeneity was achieved. This homogeneity was close to 1 part in $10^6$. It was found that at the lower field the $\mu$-shims had to be moved in towards the center of the magnet to account for the decrease in pulling force that the magnetic field exerts on them.

Electronics

As shown in Fig. 2, the electronic equipment used in conjunction with the $\text{He}^3$ cryostat consisted of a standard Pound - Knight - Watkins marginal oscillator and its associated equipment. The oscillator was initially built by E. P. Jones and subsequently much improved upon by Surrendra Sharma. The modulation coils, built by Eric Enga, consisted of 100 turns of copper wire each, and when attached in series, could give an undistorted field of up to 7 gauss.
Fig. 2 Schematic diagram of steady state spectrometer
Vacuum System

As shown in Fig. 3, the $^3\text{He}$ system was connected to a vacuum pumping system which was used to evacuate the $^3\text{He}$ system before putting $^3\text{He}$ into it. The pumps used were an oil diffusion pump with a speed of approximately 20 liter/sec., and a mechanical forepump purchased from the Cenco company (Hyvac 7). The vacuum that was obtained with these pumps on the combined glass and metal system was about $10^{-6}$ mm. Hg after about a day or so of pumping, as measured by a Phillips gauge that had been calibrated for air by means of a McLeod gauge up to approximately $6 \times 10^{-5}$ mm. Hg. The entire system was repeatedly leak tested, particularly the storage cans and the low temperature tubing inside the dewar, by means of a Veeco $^4\text{He}$ leak detector. The low temperature tubing was also leak tested at liquid nitrogen temperatures. This was done with the inner liquid $^4\text{He}$ dewar filled with $^4\text{He}$ gas and liquid nitrogen in the outer dewar cooling the $^4\text{He}$ exchange gas. The leak detector was then attached to pump on the tubes inside the dewar and the $^4\text{He}$ count was checked. A further test was also made at 1.2°K to check for the existence of any superfluid leaks. The check was made by pumping on both the vacuum jacket and sample holder and reading the pressure obtained on the Phillips gauge. A satisfactory pressure was considered to be about $10^{-5}$ mm. Hg, after about half an hour of pumping.

Usually, during an actual run, the vacuum pumps were shut off from the $^3\text{He}$ system and the pumps were used only
Fig. 3 Schematic of He³ cryostat and associated vacuum system
to pump He\textsuperscript{4} exchange gas out of the vacuum jacket. It was found that it took almost half an hour to get a pressure reading of $10^{-5}$ mm. Hg with an air calibrated Phillips gauge. A discussion of the pressure that might actually be in the vacuum jacket is given in the Appendix under the Heat Leak calculations.

\textbf{He\textsuperscript{3} Run}

Before precooling the dewars with liquid nitrogen, the vacuum jacket of the He\textsuperscript{3} condenser pot was filled with about 2 cm. Hg of He\textsuperscript{4} exchange gas. It was found also, that it was necessary to have some exchange gas inside the condenser pot itself, since lucite is a poor thermal conductor and hence the cooling of the sample would take a long time. For a first run, after the cans had just been assembled and leak tested, He\textsuperscript{4} was used as the exchange gas at about the same room temperature pressure as in the vacuum jacket. For any subsequent runs the He\textsuperscript{3} that had remained in the tubing was used as the exchange gas. The reason for the above procedure was two-fold. To check that no superfluid leaks were present, the pumps were used to pump on the vacuum jacket and sample holder. Consequently, to avoid the loss of any He\textsuperscript{3} exchange gas, He\textsuperscript{4} was used instead, and it was determined whether the pressure in the sample holder could be pumped low enough in a reasonable time ($10^{-5}$ mm. Hg in $\frac{1}{2}$ an hour).

After each run a little He\textsuperscript{3} always remained in the condenser pot and tubing above it. Since the leak tightness
of the tubing had been determined in a previous run this, plus some additional He\(^3\), was now used as the exchange gas, and no pumping on the sample holder was attempted.

After a liquid He\(^4\) transfer, the liquid He\(^4\) was pumped on to a pressure of about 0.6 mm. Hg, which corresponds to \(T = 1.2^\circ\text{K}\). The He\(^3\) was then pumped into the condenser pot by means of the 2M4 and the pressure of the He\(^3\) checked using the oil manometer to see if it had condensed. This pressure was about 19.6 mm. Hg. With the 2M4 still pumping into the condenser pot the oil diffusion pump was used to pump out the vacuum jacket. This usually took about half an hour. After a room temperature pressure of about \(10^{-5}\) mm. Hg had been reached by the oil diffusion pump on the vacuum jacket, the pumping action of the 2M4 was reversed. To achieve the longest run possible, only one 5 liter can was opened for the 2M4 to exhaust the He\(^3\) vapour into. The pumping action was fairly rapid and this can was filled to about 35 mm. Hg in 5 minutes. The can was then closed and the other 5 liter can opened. This one was filled to about 30 mm. Hg when the pressure build-up became imperceptibly small. The can was then closed and the 2M4 was allowed to pump into the 15 liter can. The manometer was shut off from the cans and connected instead to the top of the pumping line. The McLeod gauge was now used to measure the pressure of the He\(^3\) vapour at the top of the pumping line. The pressure measured was about 6.37 microns of Hg. At this pressure reading the corrected value of the carbon resistance in contact with
the He³ was measured to be 5.12K. The He³ remained at this temperature for about 4 hours.

In the Appendix an estimate is made of the heat leak into the condenser pot and a value of about 110 ergs/sec. is calculated. This would correspond to a He³ run of a little less than 40 hours, for 1 cc. of liquid He³. Since the actual run was only about 4 hours for about 1.5 cc. of liquid, it must be assumed that the actual heat leak was closer to 1500 ergs/sec. The temperature of the He³ obtained, however, indicated both by the vapour pressure data and the resistance measurements, shows that the calculation of the pumping speeds was essentially correct despite various simplifying assumptions. Of course the calculations involving pumping speeds only apply at the lowest vapour pressure readings where molecular flow conditions are applicable. However, pumping speeds calculated assuming molecular flow conditions are always less than corresponding calculations assuming viscous flow. Hence, the above would correspond to a theoretical lower limit.

In the Appendix an estimate was made of the pressure rise down the pumping line. The correction obtained was added onto the vapour pressure reading gotten from the McLeod gauge. The pressure value thus calculated gave the corresponding temperature of the liquid He³ that was obtained in this experiment. It was felt that this correction to the vapour pressure data was reasonably reliable
in view of the statements made above of the reliability of the pumping speed calculations.

The value of the temperature of the liquid $\text{He}^3$ that was obtained using the corrected vapour pressure data agrees well within the accuracy of both readings with the resistance measurement data taken from the paper by BRW. From calculations in the Appendix this value was approximately $0.35^\circ\text{K}$, and this temperature could be maintained for about 4 hours.
APPENDIX

Cryostat

In these sections some of the features of the cryostat which have not been mentioned before or only touched upon will be described in detail. A schematic diagram of the dewar and its associated tubing was given in Fig. 3 in the previous section. Figs. 4 and 5 in the following section each show details of various parts of the jacket. A description of these parts is given below except for the coaxial cable and sample coil, which were described under EXPERIMENTAL METHODS AND APPARATUS.

Condenser Pot

The condenser pot was constructed of a 7/8 in. copper tube approximately 2 in. long, whose walls had been thinned down to a thickness of about 0.040 in. in order to cut down on the amount of metal that had to be cooled. Copper was used rather than brass, because the sample holder was suspended between the pole faces of an electromagnet, and it was essential that a very non-magnetic material be used, since a magnetic material would destroy the homogeneity of the magnetic field. A field homogeneity around the sample of better than one part in $10^5$ was considered to be necessary. A copper lid was hard soldered onto the bottom of the tube and the sample and carbon resistance were fitted into a lucite cylinder which sat snugly inside the copper can. The purpose of the lucite was mainly to fill out
Fig. 4 Schematic of pumping lines and cans
the large available space inside the can and to ensure that, even with a small amount of He\(^3\) left, this would be around the sample. Hence the hole in the center of the lucite was made a bit larger than was required for the sample, so that part of the liquid He\(^3\) could run around it. The carbon resistance used for temperature measurements was put directly beside the sample and in contact with it. This was to ensure that the temperature measured by the resistance was reasonably close to that of the sample.

The can was closed off at the top by means of a copper lid which was soldered to the can with Wood's Metal. This was necessary since the can had to be removed from time to time which had to be done without melting the other solder joints on the lid. Four leads coming from the carbon resistance were passed through the lid by means of a five-lead platinum to glass seal, one lead being extra in case of one of the others breaking off. Similarly, the coaxial cable lead was passed through the top using a platinum to glass seal.

The ground lead from the sample coil was soft soldered onto the lid before putting on the can. Both this lead and the can itself were soldered on using a soldering gun, although care had to be taken not to overheat the lid, as then the soft-solder would melt. Since Wood's Metal melts at around 70\(^\circ\)C this provided little problem; it was just necessary to stop heating the can and lid after the Wood's Metal had started running. It was found that the Wood's Metal solder provided a reliable low temperature
Fig. 5 Schematic of r-f coil, sample, carbon resistance and low temperature seals.
seal as did all the other seals mentioned above.

**Vacuum Jacket**

Surrounding the sample holder was another copper can, which, when evacuated, thermally isolated the sample holder from the liquid He\(^4\) bath. Both the can and lid were of identical construction and similarly mounted as the sample holder described above, the only difference being an additional pumping line starting out of the lid of this can, plus the coaxial cable. The \(\frac{3}{4}\) in. OD stainless steel pumping line was silver soldered into the lid. It should be noted that all of the stainless steel tubes consisted of type 403 or 328 stainless steel which was picked for its non-magnetic properties.

**General Construction of Cryostat**

A supporting structure had to be built for the He\(^3\) dewar cap, the various tubing coming out of it, the valve board, the diffusion pumps and the manometers. The structure was designed by E. Watchorn and constructed by the Physics Machine Shop. The structure was built out of aluminum angle iron and care was taken to make it as vibrationless as possible, since vibrations are a significant source of noise in the marginal oscillator, and since, also, possibly a significant heat input to the He\(^3\) is caused by vibrations. The structure was big enough so that it contained two stations, one of which was for He\(^3\), the other for He\(^4\). The He\(^4\) valve board served both stations.
The $^3\text{He}$ system was constructed entirely out of metal while the surrounding liquid $^4\text{He}$ and liquid nitrogen dewars were of glass, designed by the Physics Department glass blower, J. Lees.

Heat Leaks and Pumping Speeds:

Introduction

To achieve as low a temperature as possible with the liquid $^3\text{He}$, it was necessary to simultaneously optimize the pumping speed of the line above the condenser pot and the heat leak into the condenser pot.

The amount of heat leaking into the $^3\text{He}$ condenser pot determines the mass rate of flow ($Q$) of evaporating $^3\text{He}$. The temperature (pressure) obtainable by pumping on the liquid is related to the mass rate of flow and the combined pumping speed of the pump and tubing between the pump and the condenser pot by

$$Q = S_T P_1.$$  

This equation was used to arrive at an estimated value of $P_1$, the vapour pressure of $^3\text{He}$ obtainable above the liquid. $Q$ was calculated from an estimate of the amount of heat leaking into the sample holder, while $S_T$ was calculated from a knowledge of the geometry of the tubes and the temperature and pressure of the gas being pumped.

Heat Leaks

The main sources of heat input from the liquid $^4\text{He}$ bath into the condenser pot were

1) Conduction of heat by helium gas inside the
vacuum jacket.

2) Heat leak down the stainless steel tube.
3) Heat leak down the electrical leads.
4) Heat leak by radiation.
5) Heat input from the sample coil.
6) Heat input from the carbon resistance.

Another possible source of heat leak was mechanical vibrations of the liquid He\(^3\), but since no data could be obtained on this effect it was not estimated. In the calculations that follow it was assumed that the liquid He\(^3\) bath was at 0.3\(^°\)K and the liquid He\(^4\) bath at 1.2\(^°\)K. This was approximately the case in the actual experiment.

A large heat input resulted from the conduction of heat by the small amount of helium gas remaining in the vacuum jacket. Since the outer mantle of the jacket was at 1.2\(^°\)K this was about the only material which had not yet frozen out. Using the equation\(^9\)

\[ Q = 0.028 a_0 \cdot p_{mm}( T_2 - T_1 ) \text{ w.cm.}^{-2} \]

this heat leak was estimated. The above holds at low temperatures and pressures for approximately parallel surfaces ( \( p_{mm} \) measured at room temperature ) for helium, where

\[ T_1 = \text{temperature of inside}, \]
\[ T_2 = \text{temperature of outside}, \]
\[ a_0 = \text{accommodation coefficient} = \frac{a_1 a_2}{a_2 + A_2/A_1(1 - a_2)a_1}, \]

where \( A_2, A_1 \) are the areas of the outside and inside surfaces, respectively, and where the accommodation coefficient in general is defined by

\[ ( T_2' - T_1 ) = a( T_2 - T_1 ) \]
where $T_1$ is the temperature of the gas, $T_2$ the temperature of the surface and $T'_2$ an intermediate temperature that the gas molecule has acquired after bouncing off the surface at $T_2$.

For our case it was assumed that 'a' had an upper limit of approximately $\frac{1}{2}$ and that $a_1 = a_2$, since both surfaces were copper of about equal shinyness. Assuming $p_{mm} = 10^{-5}$ mm. Hg, and calculating $a_0$ from above, a value of $Q = 25$ ergs/sec. was obtained.

The pressure actually obtained after $\frac{1}{2}$ hour of pumping was about $10^{-5}$ mm. Hg, as read by an approximately air calibrated Phillips gauge. Garfunkel and Wexler estimated that an air calibrated Phillips gauge connected to a jacket containing He$_4$ exchange gas at very low pressures would read a factor of about 100 higher pressure than was actually the case; i.e. for a Phillips gauge reading a pressure a bit more than $10^{-6}$ mm. Hg the pressure of the He$_4$ exchange gas would actually be only about $10^{-8}$ mm. Hg. Hence the heat leak calculated above may be a bit too high by a factor of 10, perhaps.

The heat leak down the thin-walled stainless steel pumping tube was calculated using

$$dQ = A / l \ k(T) dT.$$  

This equation can be integrated, assuming knowledge of the functional dependence of the thermal conductivity of steel with respect to temperature in the range 1.2°K to 0.3°K. Berman has determined $k(T)$ experimentally in the range 90°K to 2°K for german silver, constatan, and stainless steel. Assuming $k(T)$ to vary in the same manner
at the lower temperature as at $2^\circ K$ and higher, a heat leak of

$$Q = \frac{A}{2} \int_{2.3}^{1.2} 3 \times 10^{-4} T^{1.45} dT = 41 \text{ ergs/sec.}$$

was calculated.

Similar calculations as the above were made for the electrical leads going into the condenser pot. Four manganin leads of # 40 gauge were used for the resistance measurement leads and a lead of # 34 gauge was used for the coaxial cable. Manganin was used, since it has a low thermal conductivity, reasonable electrical conductivity and a small thermoelectric coefficient. All leads were connected at top and bottom to platinum in glass seals. Since platinum is as good a thermal conductor at low temperatures as copper, it was assumed that the end points of the manganin wires were at $1.2^\circ K$ and $0.3^\circ K$, respectively, heat leak calculations were made using this assumption. Using an average value of $k$ in the range $1.2^\circ K$ to $0.3^\circ K$ of $3 \times 10^{-3}$ watts/cm.deg.K for manganin, a value of 0.1 erg/sec. for one # 40 gauge wire was obtained. To cut down the heat leak, the wires used were much longer than was necessary. This was not attempted with the # 34 wire for the coaxial cable although it could have been done, but with greater difficulty than for the above. The heat leak down this wire was calculated to be about 1 erg/sec., with the resistance of the wire less than one ohm for that length.

Heat leaks due to radiation were difficult to estimate. However, a reasonable upper limit was calculated
by using the equation

\[ \dot{Q} = \sigma A (T_1^4 - T_2^4) \frac{\xi}{2} \]

assuming \( \xi \ll 1 \) and making a guess at the actual value of \( \xi \).

There were two main sources for radiative heat leak

1) Radiation between the walls of the vacuum jacket.
2) Radiation down the He\(^3\) pumping line.

The first part was straightforward to calculate, since the outer wall was always at 1.2°K and the inner at 0.3°K.

Using the above equation, the heat leak was calculated to be about 10^-4 ergs/sec. for an estimated \( \xi = 0.05 \) for shiny copper.

To eliminate much of the radiation coming down the pumping line a radiation bend was put into the tube where it changed diameter from 3/8 in. OD to 1 in. OD. This bend was about 8 in. above the lid for the vacuum jacket. As long as the liquid He\(^4\) level was above the bend or around it, the amount of radiation down the tube was, at worst, assuming perfect specular reflection down the tube, less than the value for the vacuum jacket. However, after the level of the liquid He\(^4\) has dropped to about 2 cm. below the radiation bend it was assumed that the radiation entering the top of the tube was at about 30°K. For this value of the temperature, the radiative heat leak would be about 30 ergs/sec., which is a considerable increase over the previous value. The above calculations, however, are extremely crude and would only give an upper limit to the amount of radiation reaching the condenser pot.

A large amount of the heat input to the liquid He\(^3\)
was coming from the power put into the sample coil. For a high Q circuit (Q ≥ 10) this power input is given by the equation

\[ P = \frac{V^2}{R'} \]

where \( R' = Q\omega L \) and where \( L = 3 \mu \text{h}, Q = 50, f = \frac{\omega}{2\pi} = 19 \times 10^6 \text{cps.}, \) and \( V = 100 \text{mv}. \) An approximate heat input of 30 ergs/sec. was calculated. Since the power input varies as the square of the voltage level across the r-f coil, it was important that this level be kept as low as possible. Since a low level of oscillations was also most desirable for best signal to noise operations of the marginal oscillator, this provided no difficulty.

Another source of electrical heat input was the current flowing through the carbon resistance. This current was adjusted to be exactly 10 ua. by means of a potentiometer. Hence this heat input was calculated to be not more than 6 ergs/sec. The heating of the manganin leads can be considered negligible, since their resistance was a factor of 1000 less than the carbon resistance.

The total heat leak, as calculated above, came to about 130 ergs/sec., for the case when the radiative heat leak was at its worst. A value of 110 ergs/sec. would be an acceptable mean value. However, as mentioned in Chapter 2, the experimentally determined heat leak was about 1500 ergs/sec., which constitutes a considerable discrepancy between the theoretically calculated value. It is thought that a possible error in the theoretical calculations might be the assumed value of the pressure in
the vacuum jacket in the heat conduction by a gas calculations, since the Phillips gauge reading this pressure was actually far closer to the pump than to the vacuum jacket, and part of the tubing between gauge and vacuum jacket consisted of a considerable length of \( \frac{3}{4} \) in. OD copper tubing.

**Pumping Speeds:**

A general method for calculating the ultimate pressure obtainable above the liquid He\(^3\) bath is to use the equations\(^8\)

\[
Q = S_T P_1 \quad \text{and} \quad \frac{1}{S_T} = \frac{1}{S_t} + \frac{1}{S_r},
\]

where \( S_T \) = total pumping speed of the system (in l/sec.),

\( S_t \) = room temperature speed of the tubes (l/sec.),

\( S_r \) = effective room temperature speed of tubes inside dewar (l/sec.),

and where it is assumed that the pressure at the pump mouth is zero.

Garfunkel and Wexler\(^\text{10}\) have derived the effective room temperature speed of a series of tubes with differing diameters in a temperature gradient, making the following assumptions

1) Small P/T gradient.

2) Cylindrical tubes.

3) Molecular flow conditions apply (mean free path of molecules \( \gg \) radius of tubes, \( r_i \)).

4) End effects are negligible (length of tube, \( l_i \gg r_i \)).

They have obtained the equation

\[
S_r = \frac{9000}{\sqrt{M_i}} \frac{\ell}{c} \quad \text{l/sec.}
\]

for \( T_r = 295^\circ \text{K} \).
$T_1$ and $P_1$ are the temperature and pressure, respectively, at the low temperature end of the tubes. For the part of the tubing which was outside the dewar and hence at a reasonably constant temperature the equation

$$S_t = 30.5 \frac{r^3}{L \sqrt{T/M}} \text{ 1/sec.}$$

was used, which applies when assumptions 2), 3) and 4) are valid. It was assumed, when using the above equation, that the He vapour was close to room temperature, i.e. at a temperature equal to $T_r$. Hence, knowing $S_r$ and $S_t$, and thus $S_T$ and also $Q$ (in liter micron / sec.) it was possible to calculate a value for $P_1$ (in microns of Hg). The values obtained for the various pumping speeds were:

$S_r = 33.6$ liter/sec., $S_t = 15.7$ liter/sec. Hence $S_T$ was calculated to be about 10.1 liter/sec. The total heat leak calculated came to about 110 ergs/sec. as calculated in the previous section, but as was stated earlier, the actual heat leak was believed to be closer to 1500 ergs/sec. Using this value the corresponding evaporation rate of the liquid He was $Q_M = 4.2 \times 10^{-5}$ gm./sec. This value was converted to lusecs.(liter micron / sec.) by using

$$Q = Q_M \times 1/1.5 \times 10^5 \text{ T/M lusecs.}$$

Hence, $Q = 40$ lusecs. was calculated for $T = T_r$, which gave $P_1 = 3.7$ microns of Hg. This corresponds to a liquid He temperature of about $0.33^\circ\text{K}$.

For the calculation of the rise in pressure from the dewar cap to the condenser pot due to the pumping action, the equation $P_d = Q/S_r$ was used and it was assumed, as before, that the temperature of the He was close to room temperature once it had gotten past the dewar cap.
Hence, using the previously calculated values of $Q = 40$ lusecs., and $S_r = 33.6$ liter/sec., $P_d = 1.2$ microns of Hg. Hence this pressure rise was added to the McLeod gauge reading noted in Chapter 2. This gave a pressure reading of the $\text{He}^3$ vapour pressure directly above the bath of $7.5$ microns of Hg. It is believed that the error in the pressure drop might be as large as $50\%$. Also, the actual pressure reading is accurate only to $10\%$. Hence, this would give for the limits of the liquid $\text{He}^3$ vapour pressure $\pm 1$ micron of Hg. At $0.35^\circ\text{K}$ liquid $\text{He}^3$ temperature, this uncertainty in the temperature, corresponding to the above uncertainty in the pressure, is $\pm 0.005^\circ\text{K}$. Hence, even though the uncertainty in the vapour pressure determination of the $\text{He}^3$ vapour is rather large, the corresponding uncertainty in the temperature of the liquid $\text{He}^3$ is small enough to be acceptable.

$\text{He}^3$

Five liters of $\text{He}^3$ were purchased from the Monsanto company with a quoted tritium concentration of $8 \times 10^{-6}$. The $\text{He}^4$ concentration was stated as zero. Since it was felt, that this tritium concentration was too high for safe handling of equipment after it had come in contact with the $\text{He}^3$, an attempt was made to purify the $\text{He}^3$ of the tritium. This was done by a method devised by Erdman et al.\textsuperscript{13} who used $1\%$ of hydrogen as a carrier gas and passed the $\text{He}^3$ slowly through tubes immersed in liquid $\text{He}^4$ at $4.2^\circ\text{K}$. Using this method they achieved a purity of $6 \pm 2$. 
parts in $10^{11}$ from a starting concentration of tritium of $1.7 \times 10^{-8}$. The same equipment as was used by Erdman et al. was used in this experiment with the exception that no attempt was made to measure the concentration of tritium by means of a Geiger counter.

The He$^3$ was pumped into a 5 liter can that was constructed of a 3 in. OD brass tube with the ends closed by hard-soldering a brass plate on either side. Hence the pressure in the can was approximately one atmosphere. A tritium counter was used to check the concentration of tritium in the air while dismantling the sample holder to make some changes after a few runs had been made and no large tritium concentration was detected. Hence it was felt that either the He$^3$ had been purified or that most of the tritium had been pumped back into the cans and what was left was not appreciable enough to register.


