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LATENT HEAT
OF
LIQUID HELIUM

SOME PROBLEMS IN THE DETERMINATION
OF THE
LATENT HEAT OF LIQUID HELIUM

By

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SCOPE AND CONTENTS:

A cryostat has been constructed for the determination of the latent heat of evaporation of liquid helium-4 from 1.8°K to the critical point. It has not yet proved possible to stabilize the cryostat behaviour sufficiently and thermal oscillations of the type reported by Taconis are strongly suspected as being the source of the difficulty. Modifications that were carried out in the design of the equipment to improve its behaviour were limited in their success so that two conclusions were reached. Firstly, a separate investigation of the problem of Taconis resonance is necessary before reliable results can be obtained. Secondly, the existing cryostat could in the meantime readily be converted to other cryogenic uses.

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TABLE OF CONTENTS

Page

ACKNOWLEDGEMENTS iii

LIST OF FIGURES v

CHAPTER I INTRODUCTION 1

THEORY 2

DERIVATION OF EXPRESSION FOR TRUE LATENT HEAT 5

SUMMARY OF PURPOSES OF PROJECT 9

CHAPTER II EXPERIMENTAL APPARATUS 10

Cryostat 10
Insulation System 10
Lifetime of Experiment 13
Calorimeter System 14
Calorimeter Heater 18
Auxiliary Components 18

CHAPTER III A. OUTLINE OF A TYPICAL EXPERIMENTAL RUN 20

Preparation 20
Cool-Down of Cryostat to Liquid Air Temperature 20
Transfer 21
Condensing-In 21
Obtaining of Data 21
Close-Down 22

B. EXPERIMENTS 22

CHAPTER IV CONCLUSIONS 32

SUGGESTIONS FOR FUTURE WORK 34

BIBLIOGRAPHY 35

LIST OF FIGURES

Page

1. Original Cryostat 10b
2. Calorimeter Condensation and Evaporation System 14b
3. Heater Circuit 17a
4. Helium Return System 18a
5. Background Boil-Off from Calorimeter 26a
6. Some Calorimeters 29a
7. Latent Heat Determination - Typical Curve 32a

CHAPTER I

INTRODUCTION

The first reported measurement of the latent heat of evaporation of liquid helium (isotopic mass 4) at different temperatures was by Dana and Kamerlingh Onnes in 1926 (Ref. 1). Their method involved noting the rate of fall of the liquid-vapour interface in a glass calorimeter when power was supplied to a heater in the body of the liquid. Despite the relative simplicity of the method they obtained a well-defined curve showing a maximum at 3°K and tending to zero at the critical point (~5.2°K). In addition they noted anomalous behaviour at the temperature of the previously discovered liquid density maximum. Their suggestion that there might be a discontinuous behaviour associated with the liquid at 2.2°K was not further pursued at the time with the result that the existence of the lambda-transition remained unappreciated.

In 1952 Berman and Poulter (Ref. 2) outlined a method they had used to carry out latent heat determinations with much improved accuracy. It consisted of thermally isolating a small calorimeter containing liquid helium, and finding the latent heat from the mass of gas evolved when a known amount of heat was supplied.

Using a slightly modified apparatus Berman and Mate (Ref. 3) carried out a more extensive investigation of the problem and obtained results from 2.2 to 5.18°K. Again considerable scatter showed up in the region of the lambda-transition; also their values exhibited deviations of the order of 1% about the smoothed curve through them, a value much in excess of the predicted experimental error.

It was with the intention of reducing this uncertainty in latent heat values and clarifying the nature of the "latent heat versus temperature" curve in the vicinity of, and below, 2.2°K that the present programme was undertaken.

A cryostat closely resembling that of the latter two groups was constructed. The experimental procedure involved condensing a few cubic centimetres of liquid helium into a previously cooled calorimeter, anchored at helium temperatures by a bath of liquid, and then monitoring the amount of gas evolved from the calorimeter for known heat inputs on a constant-pressure burette. Corrections for background heat-leaks must, of course, be applied in such circumstances.

A theoretical discussion of the usefulness of latent heat values in clarification of some of the problems of liquid helium is given next, followed by a description of the apparatus and experiments carried out.

THEORY

The quantity measured in these experiments is the apparent latent heat of evaporation, L_a, which is related to the true latent heat, L, by the relation (Ref. 4):

$$L = L_a \left(1 - \frac{\rho_v}{\rho_\ell}\right) \tag{1}$$

where ρ_v and ρ_ℓ are the densities of the saturated vapour and liquid respectively. The correction factor ρ_v/ρ_ℓ arises because some of the vapour replaces the liquid evaporated and therefore the apparent boil-off of gas is correspondingly reduced.

Since evaporation is an example of a first order phase transition the Clausius-Clapeyron equation may be applied, i.e.

$$\frac{dp}{dT} = \frac{L}{T\left(\frac{1}{\rho_v} - \frac{1}{\rho_\ell}\right)} \tag{2}$$

where T is the temperature at which vaporization occurs under pressure, p.

Now either L or ρ_v may be eliminated from equations (1) and (2) to give the relations (Ref. 5):

$$\rho_v = \frac{1}{L_a} T \frac{dp}{dT} \qquad (3)$$

$$L = L_a - \frac{T}{\rho_\ell} \frac{dp}{dT} \qquad (4)$$

Consideration of equation (3) shows that if L_a be measured accurately then using the values of T and the slope of the vapour pressure curve, both of which are well known, it is possible to determine ρ_v, the saturated vapour density. On the other hand if ρ_v is known accurately, better delineation of the vapour pressure curve would be feasible. Recent work of Edwards (Ref. 6) is pertinent to this argument. Using an optical cryostat he has determined the molar polarisability of saturated helium vapour over the range 1.8 to 4.2°K and has found it is constant within experimental error ($<1.0\%$) and the weighted mean agrees with the room temperature value to within 0.1%. This involved using the known values of ρ_v in the Lorentz-Lorenz equation:

$$\frac{n^2 - 1}{n^2 + 2} = \frac{4\pi}{3M} (N_0\alpha)\rho_v \qquad (5)$$

where n is the refractive index of the vapour

M is the molecular weight of helium

$N_0\alpha$ is the molar polarisability of the vapour

If it is concluded that the polarisability is indeed accurately constant then by reversal of the argument, ρ_v values may be found to greater accuracy than before. With accurate values of ρ_v at hand it is possible to examine the "vapour pressure versus temperature" curve for small discrepancies and so to calculate any small corrections necessary to the helium vapour-pressure temperature scale.

Also it would be possible to correlate the experimentally determined values of ρ_v with the virial expansion form of the equation of state for saturated vapour, viz:

$$pV_v = RT\left(1 + \frac{B}{V_v} + \frac{C}{V_v^2} + \ldots..\right) \qquad (6)$$

where V_v $(= \frac{1}{\rho_v})$ is the specific volume of the saturated vapour, and thereby determine the coefficients B, C, ... etc. Knowledge of these enables a check to be made on the interatomic potential for which various forms have been postulated (Ref. 7, 8, 9, 10).

A further matter of interest that can be settled immediately by a latent heat determination is the behaviour of the quantity itself at the lambda point. If helium in the liquid state shows a temporary sharp increase in its specific heat at about 2.2°K and if it is assumed that there is no such anomalous behaviour associated with the vapour, then a corresponding decrease can be expected in the latent heat for transitions between the two states in this region. This is implicit in the relation (Ref. 11):

$$\frac{dL}{dT} - \frac{L}{T} = C_{LIQ} - C_{VAP} \qquad (7)$$

where C_{LIQ} and C_{VAP} are the specific heats of the liquid and vapour phases at temperature T under the saturation vapour pressure. The very first determination (Ref. 1) of the curve "L versus T" showed scatter indicative of an angular point at 2.2°K but as yet there are no results available to define the behaviour unambiguously (Ref. 12, 13, 14).

DERIVATION OF EXPRESSION FOR TRUE LATENT HEAT

An expression for the latent heat, L, may be derived theoretically as follows.

Vaporization takes place isothermally and isobarically and can be conceived of as occurring reversibly. Hence during such a process the Gibbs function of the system remains constant. We have:

$$G_{\ell} = G_{v} \tag{8}$$

where G_{ℓ} and G_{v} are the Gibbs free energies of the liquid and vapour respectively.

But, $G = U + pV - TS = F + pV$ (9)

where U is the internal energy, p pressure, V volume, T absolute temperature, S entropy and F Helmholtz function of the system.

So $dG = -SdT + Vdp$ (10)

$dF = -SdT - pdV$ (11)

Integrating (10) along the saturation curve we obtain for the liquid phase,

$$G_{\ell} = G_{0\ell} - \int_0^T S_{\ell}dT + \int_0^p V_{\ell}dp \tag{12}$$

At $T = 0$, $pV_{\ell} = 0$ and $TS_{\ell} = 0$ so

$$G_{\ell} = U_{0\ell} - \int_0^T S_{\ell}dT + \int_0^p V_{\ell}dp \tag{13}$$

For co-existing phases S_{ℓ}, V_{ℓ} and p are functions of T only, and since

$$\int_0^p V_{\ell}dp = \int_0^T V_{\ell}\left(\frac{dp}{dT}\right)_{SAT} dT \tag{14}$$

then G_{ℓ} is also a function of T only.

Since G = F + pV it is convenient in the case of the vapour to calculate F and then add on pV_v where

$$pV_v = RT\left\{1 + \frac{B}{V_v} + \frac{C}{V_v^2}\right\} \qquad (15)$$

Integrating (11) from p = 1 dyne cm^{-2}, T = 1°K, ensures that the corresponding gas volume R cm^3 is large enough for the behaviour to be ideal in the range from 1°K to 5.2°K. So the integration is carried out at constant volume, R, from 1°K to T°K and at constant temperature from $V_v = R$ to V_v on the saturation curve:

$$F_v = F_{1v} - \int_1^T S_v dT - \int_R^{V_v} p dV_v \qquad (16)$$

i.e.

$$F_v = U_{1v} - S_{1v} - TS_v + S_{1v} + \int_1^T C_{V = R} dT$$

$$- RT \int_R^{V_v} \left(\frac{1}{V_v} + \frac{B}{V_v^2} + \frac{C}{V_v^3}\right) dV_v \qquad (17)$$

For an ideal monatomic gas $C_V = \frac{3}{2} R$ and $U_{1v} = U_{0v} + \frac{3}{2} R$ so we have:

$$F_v = U_{0v} + \frac{3}{2} R - S_{1v} - TS_{1v} - \frac{3}{2} RT \ln T + S_{1v} + \frac{3}{2} RT - \frac{3}{2} R - RT \ln \frac{V_v}{R}$$

$$+ RT\left(\frac{B}{V_v} + \frac{C}{2V_v^2} - \frac{B}{R} - \frac{C}{2R^2}\right) \qquad (18)$$

The last two terms are negligible since R is large and they will be omitted in subsequent development.

Now, using this expression for the Helmholtz free energy, the Gibbs energy of the vapour phase is seen to be

$$G_v = F_v + pV_v = F_v + RT\left\{1 + \frac{B}{V_v} + \frac{C}{V_v^2}\right\}$$

$$= U_{0v} + \frac{5}{2} RT - TS_{1v} - \frac{3}{2} RT \ln T - RT \ln \frac{V_v}{R} + RT\left\{\frac{2B}{V_v} + \frac{3C}{2V_v^2}\right\} \qquad (19)$$

But, $$RT \ln T - RT \ln \frac{V_v}{R} = RT \ln p - RT \ln \frac{pV_v}{RT} \qquad (20)$$

Therefore,

$$G_v = U_{Ov} + \frac{5}{2} RT - TS_{1v} - \frac{5}{2} RT \ln T + RT \ln p - RT \ln \frac{pV_v}{RT} + RT \left\{ \frac{2B}{V_v} + \frac{3C}{2V_v^2} \right\} \qquad (21)$$

This may be rewritten in a more concise notation as

$$G_v = U_{Ov} - RT \left(\ln \frac{T^{5/2}}{p} + \ln \frac{pV_v}{RT} - \frac{2B}{V_v} - \frac{3C}{2V_v^2} + i \right) \qquad (22)$$

where $$i = -\frac{5}{2} + \frac{S_{1v}}{R} \qquad (23)$$

If the vapour were an ideal gas obeying the law $pV_v = RT$ then development as above would yield

$$F_v = U_{1v} - S_{1v} - TS_v + S_{1v} + \int_1^T C_{V = R} dT - RT \int_R^{V_v} \frac{1}{V_v} dV_v \qquad (24)$$

$$= U_{Ov} + \frac{3}{2} R - S_{1v} - TS_{1v} - \frac{3}{2} RT \ln T + S_{1v} + \frac{3}{2} RT - \frac{3}{2} R - RT \ln \frac{V_v}{R}$$

$$G_v = U_{Ov} - TS_{1v} - \frac{3}{2} RT \ln T + \frac{5}{2} RT - RT \ln \frac{V_v}{R} \qquad (25)$$

Using relation (20):

$$G_v = U_{Ov} - TS_{1v} - \frac{5}{2} RT \ln T + RT \ln p - RT \ln \frac{pV_v}{RT} + \frac{5}{2} RT \qquad (26)$$

and here $\ln \frac{pV_v}{RT} = 0$ since $pV_v = RT$. Comparison of equations (21) and (26) shows that the non-ideality correction which we shall denote by ϵ, is

$$\epsilon = \ln \frac{pV_v}{RT} - \frac{2B}{V_v} - \frac{3C}{2V_v^2} \qquad (27)$$

Now from equations (8), (13) and (22) we have

$$L_O = U_{Ov} - U_{O\ell} = RT \left\{ \ln \left(\frac{T^{5/2}}{p} \right) + \epsilon + i \right\} - \int_0^T S_\ell dT + \int_0^p V_\ell dp \qquad (28)$$

This is an expression for the latent heat of evaporation at absolute zero in terms of other thermodynamic quantities. It has been predicted that the latent heat curve as a function of temperature should become linear below about 1.5°K so that if points are obtained, say, down to 1.0°K then extrapolation to 0°K would permit a check on the above expression. With a larger pumping system such a procedure would in principle be possible with the present apparatus. In the limit as the temperature tends to zero the value of the apparent latent heat tends to l_0 as may be seen by reference to equation (4).

The general expression for the latent heat at different temperatures is given by:

$$L = T\,(S_v - S_\ell) = T\left\{-\left(\frac{\partial F_v}{\partial T}\right)_{V_v} - S_\ell\right\} \qquad (29)$$

But from (18):

$$F_v = U_{0v} + \frac{3}{2}RT - TS_{1v} - \frac{3}{2}RT\ln T - RT\ln\frac{V_v}{R} + RT\left\{\frac{B}{V_v} + \frac{C}{2V_v^2} - \frac{B}{R} - \frac{C}{2R^2}\right\}$$

$$= U_{0v} - RT\left(\ln T^{3/2} + \ln\frac{V_v}{R} - \frac{B}{V_v} - \frac{C}{2V_v^2} + i + 1\right) \qquad (30)$$

Hence,

$$-\left(\frac{\partial F_v}{\partial T}\right)_{V_v} = R\Big(\ln T^{3/2} + \ln\frac{V_v}{R} - \frac{B}{V_v} - \frac{C}{2V_v^2} + i + 1 + T.T^{-3/2}.\frac{3}{2}T^{1/2}$$

$$- \frac{T}{V_v}\frac{dB}{dT} - \frac{T}{2V_v^2}\frac{dC}{dT}\Big) \qquad (31)$$

However, $\ln T^{3/2} + \ln\frac{V_v}{R} = \ln\frac{T^{5/2}}{p} + \ln\frac{pV_v}{RT}$ (32)

Therefore

$$L = RT\left\{\ln\frac{T^{5/2}}{p} + \ln\frac{pV_v}{RT} - \frac{B}{V_v} - \frac{C}{2V_v^2} - \frac{T}{V_v}\frac{dB}{dT} - \frac{T}{2V_v^2}\frac{dC}{dT} + i + \frac{5}{2}\right\} - TS_\ell \qquad (33)$$

Subtracting equation (28):

$$L = L_0 + \frac{5}{2} RT - \int_0^T C_\ell dT + \frac{RT}{V_v}\left\{B - T\frac{dB}{dT}\right\} + \frac{RT}{V_v^2}\left\{C - \frac{T}{2}\frac{dC}{dT}\right\} - \int_0^p V_\ell dp \quad (34)$$

This is now a general expression for the latent heat of evaporation at different temperatures.

As well as in this context, the value of L_0 is important in the expression that may be derived for vapour pressure by reorganising equation (28), viz.

$$\ln p = i - \frac{L_0}{RT} + \frac{5}{2}\ln T + \epsilon - \frac{1}{RT}\left(\int_0^T S_\ell dT - \int_0^p V_\ell dp\right) \quad (35)$$

SUMMARY

To summarise then, a determination of the latent heat of evaporation of liquid helium is important for the following purposes:

a) Accurate delineation of the vapour pressure curve with special regard to fluctuations in its slope over small temperature intervals, say of the order of a millidegree, to be used for correcting any small discrepancies found in the vapour-pressure temperature scale.

b) Determination of the vapour density and the consequent check on the virial coefficients of the equation of state.

c) Investigation of the behaviour of the latent heat in the region of the lambda point ($\sim 2.2^\circ$K) as a guide to elucidation of the lambda transition mechanism.

d) It would constitute a source of experimental information on the value of L_0, the latent heat at absolute zero, a ubiquitous quantity in the thermodynamics of liquid helium.

CHAPTER II

EXPERIMENTAL APPARATUS

Proposals for a latent heat cryostat were made by R. C. Roeder (Ref. 15) in an earlier thesis. These were critically examined and some alterations, the most drastic of which was aimed at making the calorimeter more accessible, were thought advisable. Since several modifications were made to the apparatus initially constructed, the description that follows is not accurate in detail except in reference to the early experiments. Later, the modifications and the underlying motives for them are dealt with in the order in which they arose.

Cryostat

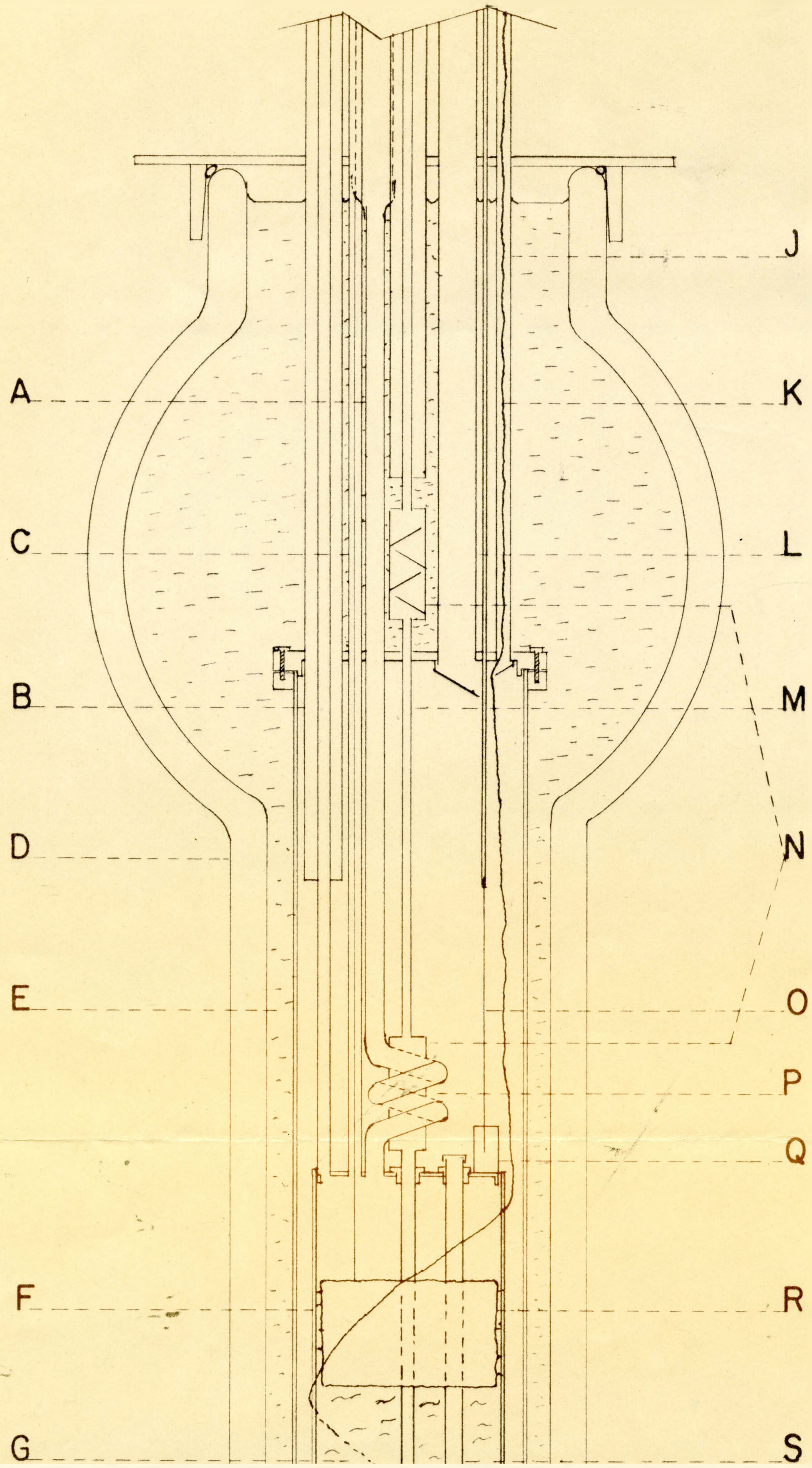
The main components of the cryostat as shown in Figure 1 were a liquid air bath, vacuum jacket, helium bath and calorimeter. The helium under investigation was put in the calorimeter and the remainder of the cryostat provided thermal insulation from room temperatures.

Insulation System

Dealing first with this insulation system it will be seen that the glass dewar, which was slit-silvered, had a bulbous reservoir to minimise the rate of level fall as the liquid air boiled off. It was held in position by two clamped "O"-braces so that it could readily be removed when desired. The brass plate near the top of the apparatus as well as acting as a cover for the dewar also constituted the support for the remainder of the cryostat. Three bolts held the plate to a dexion frame, an arrangement giving suspension that was both rigid and adjustable.

KEY TO FIGURE 1

A	Helium Bath Return Line
B	Transfer Siphon
C	Float Indicator Housing
D	Slit-Silvered Dewar
E	Liquid Air Bath
F	Helium Can
G	Helium Bath
H	Radiation Shield
J	Electric Leads Tube
K	Electric Leads
L	Vacuum Pumping Line
M	Calorimeter Evaporation Line
N	Radiation Baffles
O	Simon Thermometer Capillary
P	Heat Exchanger
Q	Thermometer Bulb
R	Styrofoam Float
S	"Spare" Tube
T	Calorimeter
U	Heater



ORIGINAL CRYOSTAT

FIGURE I

A second smaller brass plate acted similarly as a top for the main vacuum jacket which was made from 3" I.D. copper pipe with a machined brass bottom. The vacuum seal was accomplished with an indium wire "O"-ring between brass flanges tightened together with six screws. This proved to be a neat joint from the point of view of dismantlement and reassembly as well as giving very little vacuum leak trouble.

For the helium can, 2 1/2" O.D. copper pipe was used and the base was made small enough that the cylinder forming the walls could be put in place or removed by sliding it over the base. Both base and top of the can were machined copper. A cylindrical copper radiation shield for protecting the calorimeter from liquid air temperature radiation was secured to the can with Wood's metal. One part of the shield remained permanently attached to the can while the lower part could be removed during inspection of the calorimeter. Electrical leads to the calorimeter entered through the permanent upper part where a short bent length of copper tube was soldered to the inlet aperture to prevent radiation entering: the aperture also ensured evacuation of the space inside the radiation shield together with the vacuum jacket.

Since the various pipes in the cryostat are shown in Figure 1, only additional remarks on them will be given here with the sole exception of the calorimeter evaporation line which is treated in detail later. Firstly, it was decided that the vacuum-jacketed transfer siphon should be permanently fitted and have facility for re-evacuating provided by direct connection to the high vacuum system. A separate bath return line was then necessary. The advantage of a permanent transfer siphon is that the process of transfer is simplified since the helium storage dewar need only be raised up into position rather than to some considerable distance above its final location and then lowered again, an encumbrance if a heavy vessel is used. From the float indicator tube a branch tube was connected to a manometer so that the pressure reading was that of a static column in preference to monitoring the pressure via the return line in which there are inevitably some pressure gradients.

Radiation shields consisting of blackened sheet copper were fastened over the apertures of the leads tube and evacuation pipe at the top of the vacuum jacket. Helium gas filled the Simon thermometer bulb to a pressure of 1 atmosphere when at room temperature and a vacuum gauge connected to it by a capillary was used to observe the cool-down of the helium can during transfer of liquid to the cryostat.

As a general rule german silver, a low thermal conductivity alloy of copper,zinc and nickel, was used for tubes reaching into the interior of the cryostat to minimise heat leaks by conduction. Stainless steel has an even lower conductivity but it was not used because it often creates vacuum leak problems at solder joints which do not contract as much as the tubes on cooling. In regions where bends were needed in the tubes or intimate thermal contact was required, copper proved more suitable as the working material.

Some of the permanent joints were made with hard solder (m.p. ~700°C), most however were done with soft solder (300°C) and for those that were to be frequently undone and reassembled Wood's metal was used. This latter is a low-melting-point (70°C) alloy (Bi 50%, Pb 25%, Sn 12.5%, Cd 12.5%) which was considered especially suitable for securing the helium can walls in place without heat injury to the styrofoam float inside.

In deciding on the various longitudinal dimensions calculations showed that the optimum arrangement was to have the length of the pipes between liquid air temperature and the helium bath the same as the length of the bath itself. With this arrangement the conflicting requirements of low tube conduction and high reservoir volume were satisfactorily compromised. Also to minimise the heat leaks the tubes were chosen to be as narrow as was compatible with their respective functions yet consistent with requirements of structural rigidity. The evaporation line from the calorimeter was placed in a central position and the other tubes spatially arranged around in such a way that the tendency of the assembly to undergo large amplitude oscillations if accidentally disturbed from its equilibrium vertical position was effectively overcome.

Lifetime of Experiment

The period of experimentation without refill of the helium can is governed by the capacity of the helium bath and the magnitude of the heat leak. Allowing for the volume occupied by the float (30 cc) the net can capacity was 685 cc: the sources of heat leak may be listed as follows:

SOURCE OF LEAK	MAGNITUDE (WATTS)
1. Radiation from vacuum jacket to helium can walls	8×10^{-3}
2. Radiation down tubes into vacuum space	5×10^{-4}
3. Radiation down tubes into helium bath	1×10^{-4}
4. Tube conduction to helium bath	1.0×10^{-1}
5. Conduction down electrical leads	2×10^{-4}
6. Joule heating in leads and accompanying conduction	1×10^{-5}
7. Gas conduction across vacuum space (at 0.1 μ Hg pressure)	1×10^{-3}

As is general in such helium systems the significant leaks are those due to tube conduction and radiation from surfaces at higher temperatures. Heat exchange between the effluent cold gas and the pipes and the consequent reduction in apparent thermal conductivity were neglected so that the estimated lifetime for the bath of helium was expected to be rather low. The total calculated heat leak was 0.11 watts. Assuming the latent heat of evaporation to be 85 Joules/mole (Ref. 1), a leak of 1 watt gives a boil-off of 1370 cc of liquid per hour so the boil-off from the bath should have been 150 cc per hour and time available between transfers 4 1/2 hours.

Calorimeter System

The calorimeter system as shown in Figure 2 consisted of the calorimeter itself, the evaporation line within the cryostat proper, a pressure-condensation system and a constant-pressure gas burette.

To minimise heat flow up the calorimeter walls into the gas rather than across a Kapitza temperature jump into the liquid, half-inch diameter german silver tubing was used for the walls of the calorimeter; the base was of copper and the exit tube fitted into a brass bushing at the top. Calculation showed that for a temperature difference, ΔT, of 0.1°K between bath and calorimeter the heat leak down 4 cm of 0.15" german silver tubing would be $\sim 6 \times 10^{-6}$ watts while other leaks such as gas conduction and radiation would be comparatively negligible. A boil-off from the calorimeter of 0.10 cc of gas per min at N.T.P. could therefore be anticipated: with electrical leads attached an additional 0.05 cc per min due to conduction and Joule heating would result.

KEY TO FIGURE 2

a	Helium Bath
bb	Radiation Baffles
c	Vacuum Jacket
d	Asbestos-Insulated Copper Pipe
e	Pressure Gauge
f	Constant-Pressure Burette
g	Mercury Reservoir
h	Needle Valve
i	Solenoid Valve
j	Differential Manometer
k	Isothermal Pressure-Reference Bulb

CALORIMETER CONDENSATION AND EVAPORATION SYSTEM

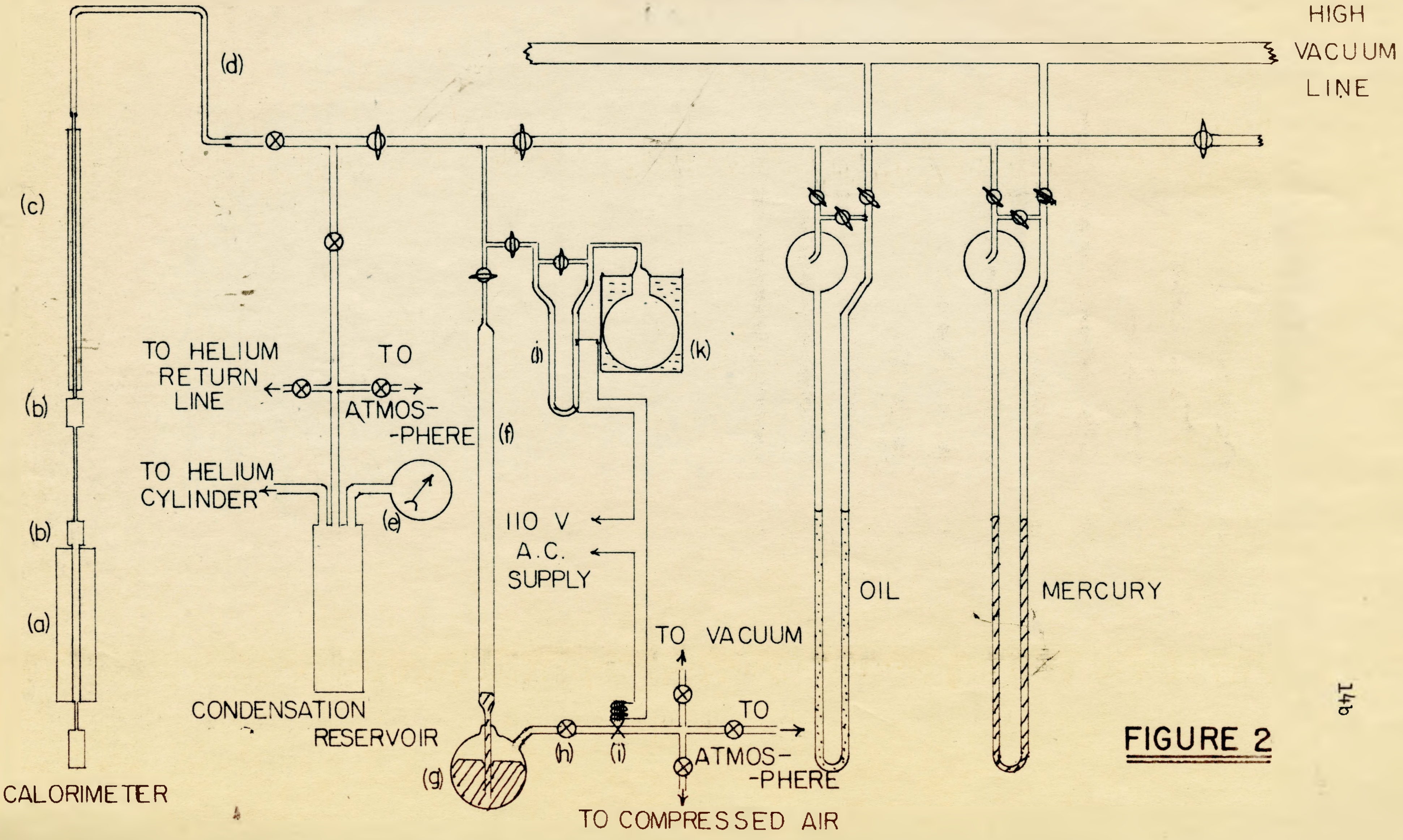


FIGURE 2

The evaporation pipe was of 1/4" copper through the helium can to ensure good thermal anchoring. Just above the helium can there was a radiation baffle assembly (at 4°K) consisting of four copper vanes embedded in a copper housing. From here, german silver tubing formed the linkage to the outside of the vacuum jacket where a second baffle arrangement (at 80°K) was located as shown in Figure 1. There was a fundamental difference in the structure of the two baffle systems. Ideally it would seem best to have the vanes sloping "upwards" so that they might have maximum efficiency in trapping radiation funnelling down the pipe. This procedure was followed for the construction of the upper baffle but was not permissible for the lower one. Such a structure in the latter case would have allowed pockets of helium liquid to collect in the 4°K baffle and these could subsequently have boiled off because of heat leaks, such as radiation, to the baffle and as a result produce a spurious calorimeter boil-off. To counteract this possibility the vanes were installed in a "downwards-sloping" fashion so that some efficiency was apparently sacrificed. Early experiments however showed that the baffles were adequate.

German silver tubing was used also from the upper radiation shield, at liquid air temperature, to the room temperature region. Here it was vacuum jacketed so that along the whole extent of the boil-off line from helium temperatures to room temperature there was a temperature distribution that did not vary with liquid air level or other transient influences. If a variation of temperature distribution along the evaporation pipe were to occur during the course of an experiment there would be consequent expansions or contractions of the gas, leading to inaccuracies in the monitored boil-off and hence errors in the latent heat determinations.

The pressure-condensation reservoir (1400 cc) was joined to the calorimeter evaporation line through a valve; there were alternative connections from the reservoir, one to the main bath return line for release of overpressures and another to provide a source of pressurized gas suitable for use during transfer.

Two manometers, one containing mercury and the other butyl phthallate, were incorporated into the system. To monitor low temperatures where pressures of less than 1 cm of mercury are measured it was convenient to have the less dense oil for amplifying the liquid level differences. On each occasion the oil must be standardised against mercury since oil density varies considerably with temperature.

The constant-pressure burette arrangement consisted of a differential manometer with a reference pressure enclosed in an isothermal bulb (immersed in a water bath) and a precision bore tube with variable mercury level. Two terminals in the differential manometer "made" and "broke" a solenoid valve circuit according as the pressure in the burette side of the manometer was higher or lower than that in the reference bulb respectively. The solenoid valve permitted air-pressure release from the mercury reservoir at the bottom of the burette and thus allowed the mercury level in the burette to fall. Pressure constancy to within 7 mm of oil was obtained in the burette. That is, the pressure in the calorimeter varied by ± 0.2 mm Hg which at 4.2°K corresponds to temperature constancy within ± 0.0003°K. Even smaller variations could have been achieved if needed, by locking the solenoid in the open position and manually regulating the needle valve in series with it so as to give a steady rate of mercury fall in the burette. Pipelines to 20 lb/sq in compressed air, to atmospheric pressure, and to the vacuum fore pump provided the necessary sources of pressure for regulating the mercury level under all conditions of calorimeter temperature.

Another feature of the evaporation system was the heat exchanger between itself and the return line of the main helium bath. During condensation into the calorimeter at the beginning of an experiment the bath boil-off rate increases considerably. From pressure-temperature-enthalpy plots (Ref. 16) the following table may be compiled:

	Pressure (P) (atmospheres)	Temperature (T) (°K)	Enthalpy (H) (calories/gram)
Initially	2	293	360
	2	90	140
Finally	1	4	7

So if it is assumed that the incoming gas is cooled to liquid air temperatures before reaching the helium bath then the enthalphy change provided by the bath, ΔH, is

$$\Delta H = 133 \text{ cal/gm} = 2230 \text{ Joules/mole}$$

whereas if all the cooling is done by the bath we have

$$\Delta H = 353 \text{ cal/gm} = 5916 \text{ Joules/mole}$$

Assuming that the latent heat of evaporation is 85 Joules/mole the ratio of liquid boiled off from the bath to that condensed in the calorimeter would be 30:1 and 70:1 in the two circumstances respectively. It is here that the advantage of the heat exchanger becomes evident since it allows transfer of heat to the vaporized helium in addition to the latent heat of evaporation and so conserves the liquid in the bath. Hence the conversion ratio was expected to be better than suggested above.

HEATER CIRCUIT

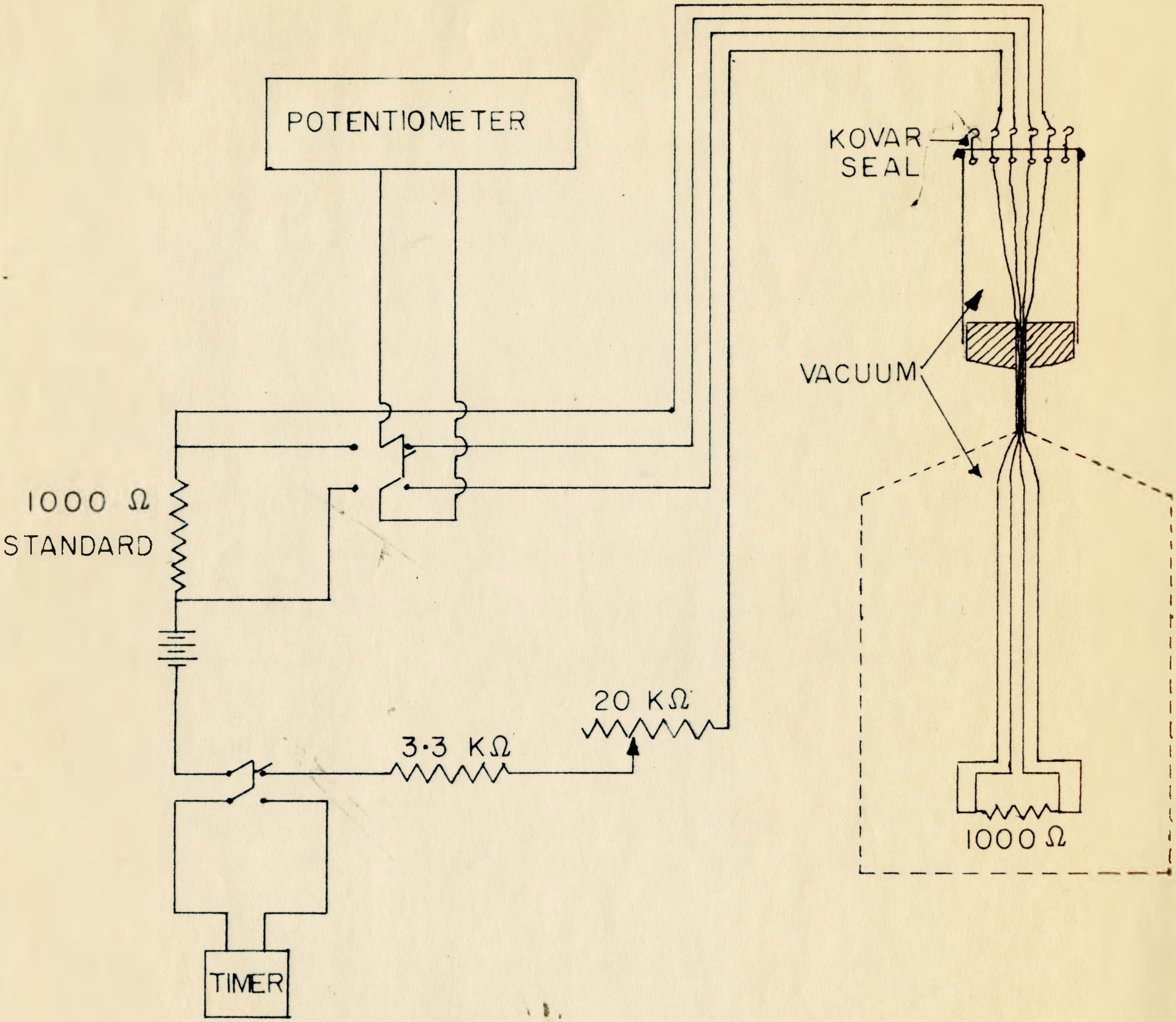


FIGURE 3

Calorimeter Heater

The heater circuit is shown in Figure 3. The heater itself was of 140 Ω/ft copel wire designed to a total value of (1000 ± 100)Ω wound on a copper former in the base of the calorimeter and secured with an epoxy resin (STYCAST 2651). The surface of the copper exposed to the helium was roughened to provide more intimate contact with, and therefore better heat transfer to, the liquid in the calorimeter. Various methods of introducing the leads to the vacuum jacket were tried until the best was found to be with a Kovar multiple feed-through terminal housed as shown in Figure 3. Wood's metal was used for the final seal to minimise the chances of heat damage to the teflon insulation of the leads. German silver wires 0.01" in diameter were used for the voltage leads and 0.005" diameter copper for the current leads, from the vacuum seal at room temperature right to the calorimeter.

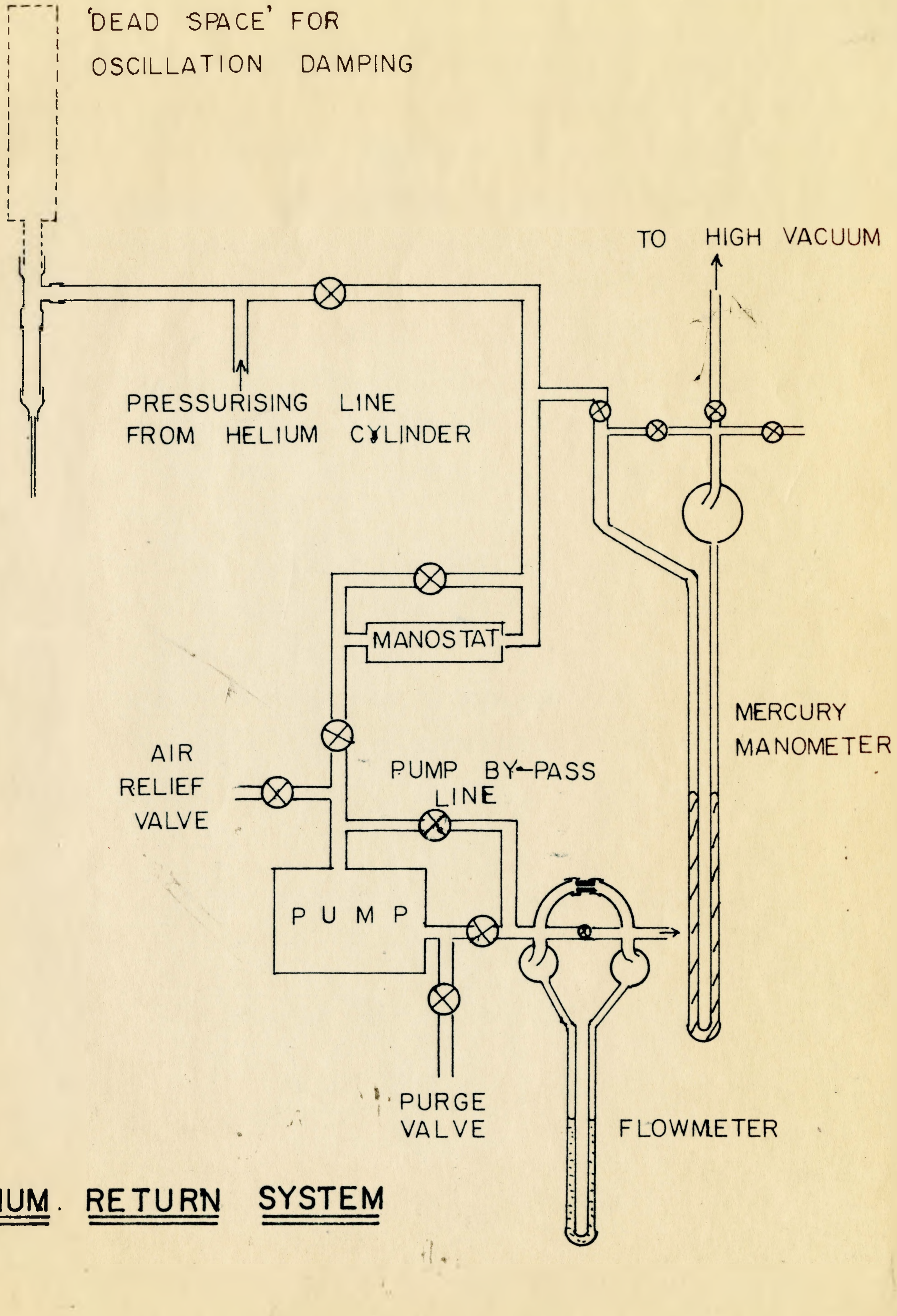
Potentiometer readings to ± 1 μV were available and the timer, (an A. W. Haydon clock, model No. K 15120), was accurate to 0.01 sec so for a total heating time of 100 sec and voltages of 1 volt, 0.01% accuracy was obtainable.

Auxiliary Components

Brief remarks on the high-vacuum system, helium bath return line, and transfer dewar, will complete the description of the experimental set-up.

A mercury diffusion pump in series with a mechanical pump consistently gave a pressure of 0.01 μ Hg or less in the main vacuum jacket. This was the reading recorded on a McLeod gauge. When corrected for thermomolecular pressure effects in the pumping tube the pressure was

FIGURE 4



HELIUM RETURN SYSTEM

therefore 0.005 μ Hg or better in the space between the vacuum jacket walls and the helium bath and about 0.001 μ Hg within the radiation shield round the calorimeter. Evacuation of the thermal insulation vacuum jacket round the central evaporation pipe and the vacuum jacket on the transfer siphon was also effected through the main system. Both were permanently connected to it with stopcock cut-offs for isolation.

The helium return line was of 1/2" copper pipe with a glass Cartesian Diver for manostatting at the desired pressure and a flowmeter to monitor the boil-off rate. A 50 litre/min rotary pump produced helium temperatures down to 1.8°K; this pump could be bypassed when not in use as shown in Figure 4.

The transport dewar was a twin double-walled slit-silvered glass assembly, the outer vessel being filled with liquid air and the inner one holding 2 litres of liquid helium. Styrofoam was used both here and in the transfer siphon vacuum jacket as spacer since it has a low thermal conductivity and is easy to work yet withstands thermal cycling without structural injury.

CHAPTER III

A. OUTLINE OF A TYPICAL EXPERIMENTAL RUN

Preparation

As a preliminary to every run the main vacuum space was pumped on for at least 24 hours to remove vapours from the system. Sometimes the vacuum jackets round the transfer siphon and central evaporation pipe were checked and re-evacuated as necessary.

Immediately before initial cool-down the helium system was evacuated and flushed a few times with cylinder helium and then filled to atmospheric pressure. Often the calorimeter section was evacuated through the high-vacuum line to remove blockage-forming impurities or check its leak-tightness.

Cool-Down of Cryostat to Liquid Air Temperature

To accomplish this, helium exchange gas to a pressure of about 50 μ Hg was introduced into the vacuum jacket and the outer dewar was filled with liquid air. Generally about 1 hour was required to reach equilibrium, during which time helium gas has to be supplied to the system from a cylinder to maintain the pressure at 1 atmosphere. It was considered inadvisable to allow the helium pressure to fall below atmospheric pressure unnecessarily as this would result in air getting into the system through any small leaks that might have remained undetected.

At the same time as the main system cooled the transport dewar was prepared. This involved filling the outer dewar with liquid air and then putting helium gas into the inner one.

Before transfer the exchange gas was pumped out of the vacuum jacket and the calorimeter system closed off to prevent condensation there.

Transfer

No real difficulties were encountered here though it was necessary to transfer 4 litres of liquid to be perfectly sure that the bath had an adequate amount in it for a 5 hour run. There did seem to be some tendency for oscillations of the liquid helium levels in the coupled vessels, i.e. transport dewar and cryostat bath, and it was found difficult to fill the bath beyond about the 3/4-full level. Different overpressures applied to the liquid in the transport dewar did not appreciably alter these effects.

Condensing-In

After transfer, helium gas at elevated pressures was fed to the calorimeter where it condensed to form liquid. Decrease in pressure in the 1400 cc condensation reservoir and increased bath boil-off acted as complementary monitoring devices.

Obtaining of Data

To obtain data, the main bath pressure was fixed with the Cartesian manostat and the calorimeter pressure was set in the reference bulb of the differential manometer at a lower value corresponding to a temperature difference of about 0.01°K. Background boil-off was then recorded with the readings of burette level versus time. Heating periods were timed and current and voltage measured. To recharge the burette, the calorimeter was closed off and some helium gas from the burette system was removed through the high-vacuum line. When sufficient had been withdrawn the calorimeter was again opened to the burette, ready for another run. In this way there was no contamination of the calorimeter helium.

Close-Down

If the experiment was terminated before the helium had all boiled off, special care had to be taken to ensure that an escape existed for the evaporating liquid. Warm-up of the apparatus could be expedited by removal of the liquid air bath and introduction of a few microns exchange gas into the vacuum jacket.

B. EXPERIMENTS

Under this title will be discussed the experiments that were carried out, the results of these, and consequent apparatus modification.

The early runs were devoted to elimination of structural defects and other deficiencies in the apparatus until the performance of the equipment approximated the anticipated behaviour. It soon became clear that the background boil-off from the calorimeter with no heater input was much in excess of the 0.1 cc of gas per minute at N.T.P. that had been calculated (vide p. 14). Different possible sources of the thermal leak to the calorimeter were investigated in turn and will now be discussed.

First the possibility of residual exchange gas within the radiation shield which might carry heat from the warmer shield to the calorimeter was inspected. Calculations showed that pumping for a few seconds should reduce the pressure to the value required for insulation but in order to check this another pumping pipe was installed in the shield and a bent tube fitted to cut off entrant radiation. After pumping through this for one hour before transfer it was found that there was no significant change in the background.

Both lacquer (DUCO No. 1,234 LACQUER) and Wood's metal were used in turn to anchor the radiation shield in thermal contact with the main bath without apparent difference in the heat leak. If heat radiated from the 80°K vacuum jacket to the 4°K shield were not conducted away quickly enough through the shield to the helium bath the temperature of the shield would rise and result in perhaps an appreciable heat leak to the calorimeter by re-radiation. That there was no sensible difference in the heat leak when different fixatives were used to secure the shield to the bath suggested radiation was not a major contributory factor to the unexplained heat leak.

Vacuum troubles occurred frequently at this stage in development with the result that Wood's metal was replaced by soft solder where possible. This proved to be a considerably more workable arrangement. The helium can walls were soft soldered on so it was no longer possible to retain the low-melting-point styrofoam float but by this time sufficient experience had been gained in the transfer idiosyncrasies of the apparatus to render the float unnecessary.

A second cylindrical copper radiation shield co-axial with the first was then installed and both were secured to the can with soft solder which was also used in the assembly of the two parts of the inner shield. At this stage, furthermore, it was found convenient to introduce the electrical heater leads to the calorimeter space through a different aperture. In the initial construction of the apparatus a second spare tube had been inserted through the helium can from the main vacuum jacket to the calorimeter space within the radiation shield, parallel to the central evaporation pipe. This was lined with teflon "spaghetti" to provide electrical insulation and the leads were then threaded through. The leads were thermally anchored at 4°K by wrapping them round the main helium can. Lacquer was used to fasten them to fine tissue paper which in turn was lacquered to the can. In this way the leads were electrically insulated yet in good thermal contact with the 4°K bath. Compliance with the conflicting electrical and thermal requirements provided a considerable problem in the early experiments.

After these modifications experiments were carried out to investigate the background heat leak. With a temperature difference of $\sim 0.02^{\circ}$K between the bath and calorimeter it was found that the boil-off rate without using the heater was 12 cc/min initially, decreasing steadily during the evolution of the first 100 cc to 9 cc/min, both rates measured at approximately atmospheric pressure. About one hour later the calorimeter was set at a temperature just below that of the bath ($\Delta T \sim 0.0004^{\circ}$K) and the boil-off was again monitored. Surprisingly it was found to be much better than the desired value of 0.1 cc/min. An attempt was made to make a latent heat determination by switching on the heater (at this stage an Allen-Bradley carbon composition resistor as shown in Figure 6 (c)) for a fixed time and then watching the subsequent return of the boil-off to the original background conditions. After heating there was a sudden increase in the "no-heater" value of boil-off before it had significantly re-approached its final asymptotic value. It was noteworthy that the new high value was 9 cc/min which was similar to that encountered in the earlier observations. So although the latent heat determination was not successful, some clue was given as to the source of trouble. It was variable in character and therefore leaks by radiation and solid conduction which would be expected to be constant could not easily be invoked in explanation. Another possibility, variation in the quality of the vacuum, could be eliminated on the grounds that no indication of any change was detected on the McLeod or Pirani gauges: in any case an air leak would be unlikely to reach the calorimeter space in gaseous form. More plausible was the theory that the variable heat leak arose through some dynamic mechanism in the helium itself in the calorimeter.

Had superheating of the helium liquid occurred there would have been a resultant high boil-off rate when the instability was disturbed but the effect would have been temporary. So this mechanism can be eliminated since no such transience was noted. Sudden ebullition especially if accompanied by "bumping" might, on the other hand, act as a trigger for the mechanism that is discussed below.

Taconis (Ref. 17) in 1949 made mention of thermal oscillations in low temperature equipment. He pointed out that in an apparatus such as the one described here it would be possible for the gas travelling up the evaporation pipe to create compressions at the top of the tube. Also, in this region the gas undergoes considerable thermal expansion. If, then, the two compression effects were out of phase, oscillations could be sustained. The present writer is of the opinion that the presence of a liquid reservoir at the lower end may lead to a further sustaining influence, viz. periodically enhanced evaporation of gas from the liquid surface. Such evaporation increases do occur and have been widely reported (vide Ref. 3, 17, 18) but whether or not they actually contribute to the oscillation mechanism is uncertain. Edwards and Woodbury (Ref. 19) have detected oscillations in helium systems even in the absence of liquid and at pressures of one-tenth the saturation vapour pressure so liquid is by no means essential to the propagation of Taconis resonances.

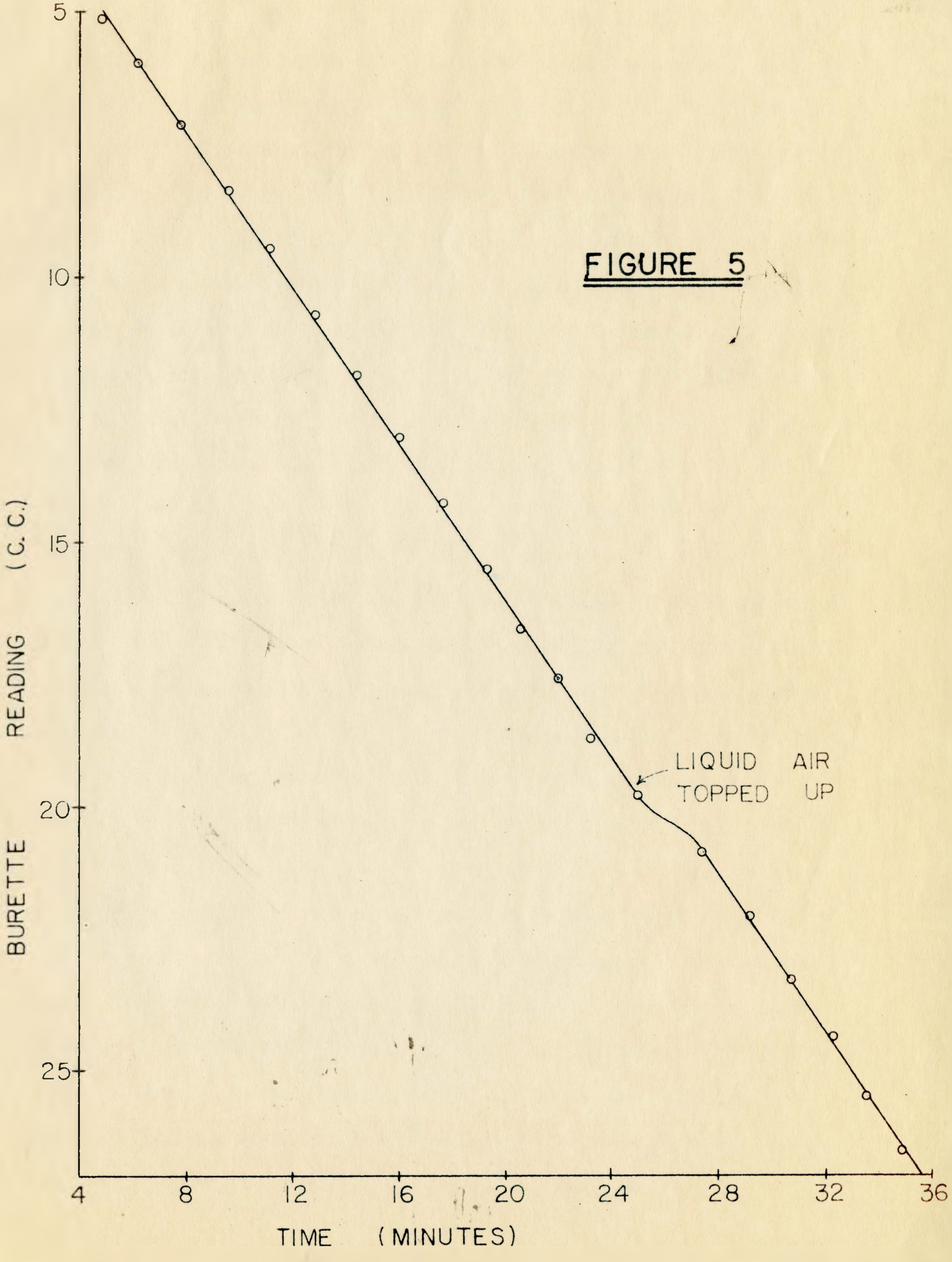
H. A. Kramers in 1949 decided from a theoretical investigation of the topic (Ref. 20) that

> 'Although ..... owing to the temperature variation there may be a marked tendency towards a negative damping of the vibrations it appears that in many cases this effect does not suffice to account for the observed instability. Its explanation has perhaps to be sought in the non-linear effects occurring in vibrations with finite amplitudes.'

These conclusions are valid for oscillations in "wide" tubes. For "narrow" tubes D. ter Haar (Ref. 21) developed a theoretical argument giving results in qualitative agreement with some experimentally determined quantities. In this context "wide " and "narrow" refer to diameters of more than about 0.6 cm and less than about 0.1 cm respectively. Unfortunately the transition region between these limiting values is rather complicated and seemingly has not yet yielded to theoretical analysis. Equally unfortunate is the requirement that the evaporation pipe from the calorimeter of the latent heat cryostat should have diametric measurements within this difficult region so that attempts at extinction of the resonance must be along empirical lines.

It should be pointed out here that no direct experimental evidence of the oscillations was obtained - they could not be seen on the butyl phthallate manometer but since this contained some 100 cc of liquid the inertia of the column was too great to reveal them in any case. Neither was there any audible signal from the apparatus.

BACKGROUND BOIL—OFF FROM CALORIMETER



As a step towards eliminating the suspected oscillations the evaporation pipe was narrowed in the helium bath region before the next experiment. Capillary copper tubing 20 cm long and 0.05 cm in internal diameter was substituted for the 1/4" copper tubing previously used. A calculation showed that the pressure drop along this capillary would not lead to errors significant within the accuracy required. The results of this change were encouraging: background boil-off was reduced to 2 cc of gas per minute. In this experiment copper wires had been used for both current and voltage leads so to see if they were effective thermal conductors another series of runs was carried out after they had been disconnected between the liquid air anchor and the 4^{o}K region. The boil-off was now 0.7 cc/min. Some improvement had therefore been effected. Turning off the rotary pump and water supply to the diffusion pump (after the main vacuum jacket had been sealed by closing the stopcock) did not influence the value so that heat leaks from mechanical vibration encountered by some experimenters in the millidegree region were not contributory. Topping up the liquid air level in the outside dewar showed up as a barely perceptible lateral displacement in the "burette reading versus time" plot as shown in Figure 5. This is to be expected from gas contraction. If the slope of the curve had been permanently changed then radiation down the evaporation pipe itself would have been suspected but this was not so. When the pump and water were off, a solid aluminum rod was held against the evaporation pipe. By listening at the other end it was hoped that audiofrequency vibrations might be heard. The bubbling of liquid air drowned out anything else and when the bath was temporarily removed leaving only the vacuum jacket as insulation nothing whatever could be detected aurally.

As a further measure to damp possible oscillations two modifications were made to the cryostat. Since it was conceivable that Taconis oscillations were originating in the tubes to the main bath and were being transduced to the calorimeter system across the thermal coupling afforded by the walls of the evaporation pipe itself, it seemed advisable to fit a damper on the helium reservoir. This took the form of a length of 3" copper pipe of about 1100 cc volume fastened to the return line at the top of the cryostat. Such "dead spaces" have been used in other cryostats with effect (Ref. 22). In addition, a spiral of german silver capillary tubing, 0.04" O.D. and 28 cm long, was substituted in the evaporation pipe between the calorimeter and bath to act as a direct inhibitor of resonance in the line. For this experiment both radiation shields were fitted round the calorimeter and the electrical leads were severed at the top of the vacuum jacket. The results obtained appeared at first most satisfactory. The boil-off from the helium bath was at a very low level and the calorimeter boil-off was in the region of 0.06cc/min to 0.08 cc/min for $\Delta T = 0.03^{\circ}K$. So the desired thermal insulation had apparently been achieved.

Leads of german silver were then installed. The thermal conductivity of 0.012" diameter wires (28 gauge) was low enough and Joule heating sufficiently small for the insulation requirements. In general these two conditions lead to conflict because of the Wiedemann-Franz-Lorenz Law.

No background boil-off or response from the heater was obtained after apparently condensing in about 1 cc of liquid. This was attributed to blockage in the spiral. A complete check on the apparatus was carried out. The leads were found to be intact and by drilling a small hole in the wall of the calorimeter and applying an overpressure from the top of the evaporation pipe it was established that no solder or other mechanical blockages had occurred. The hole in the calorimeter was useful subsequently for occasional checks on the evaporation system and could readily be sealed with a small patch of german silver sheeting 0.01" thick, soft soldered in place.

Another experiment again resulted in no heater response. At the time it was thought that the difficulty arose because of the spiral capillary so it was replaced by a straight length of 0.15" tubing as in the original apparatus to see if the dead space alone was responsible for the good background achieved in the earlier run. Boil-off's of from 2 cc/min to 16 cc/min were witnessed. The helium bath lasted for 5 1/2 hours which was comparable to other runs so it then seemed that nothing untoward was happening in the bath and that the oscillations were apparently being propagated in the calorimeter system itself. An attempt at a latent heat determination resulted in a value some 60% above the generally accepted one. On disassembly it was found that the heater which had been embedded in epoxy resin within the copper base was no longer securely held; hence the high value. A new heater winding technique was developed with a manganin wire heater wound on the outside of a solid copper base, as illustrated in Figure 6 (e). A fifty-fifty mixture of glyptal-acetone was used as fixative and the new heater was put in a glass test tube and evacuated on the high vacuum system. No separation of the wire from the copper former occurred in this test.

Two alternative evaporation pipes were installed through the helium can, a central 0.15" tube and an off-set 0.04" tube, both of german silver. To reduce thermal coupling between the bath and calorimeter

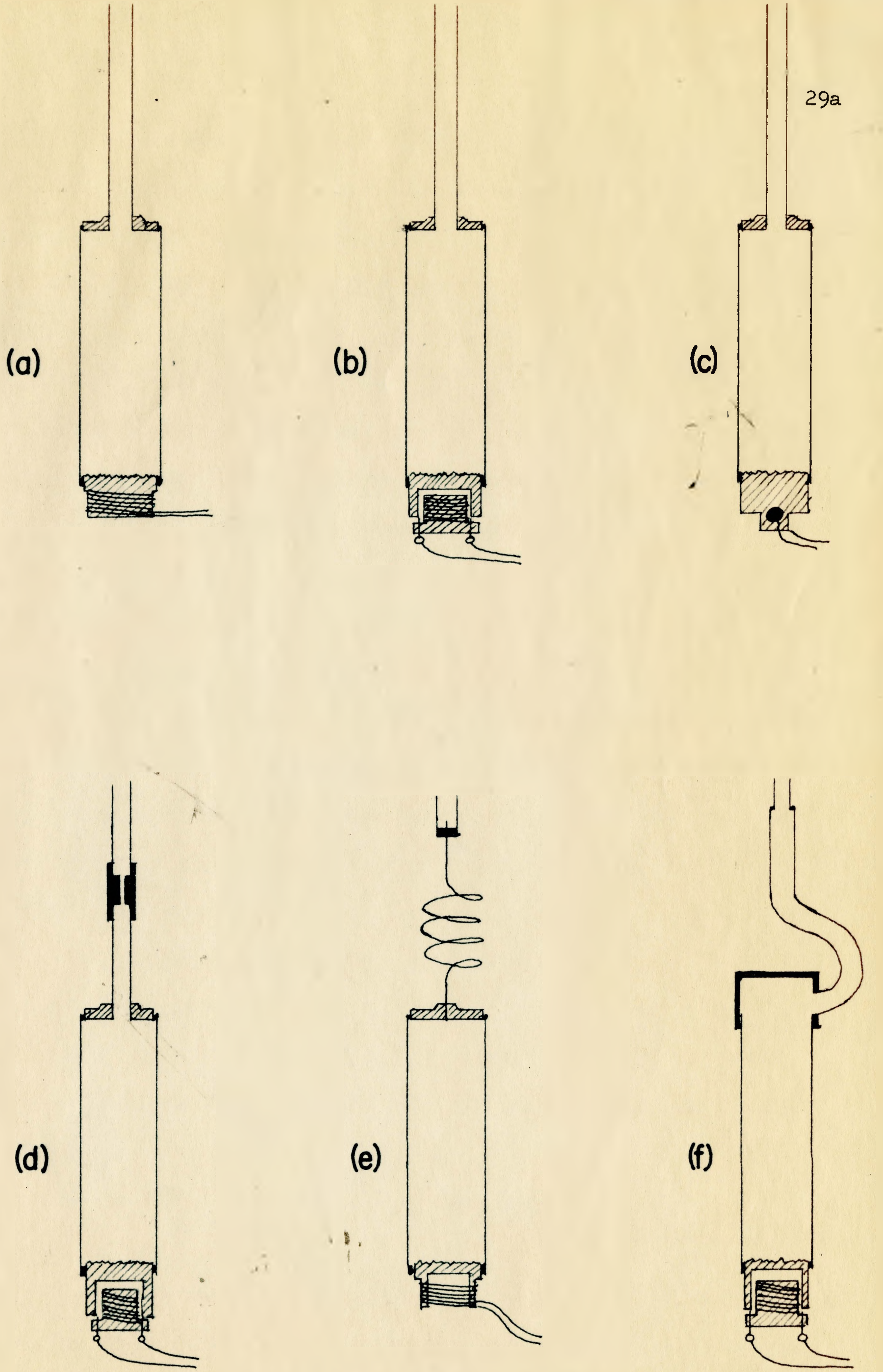


FIGURE 6 ——— SOME CALORIMETERS

systems the heat exchanger was removed and the pipes from the two systems were joined up separately. The lower radiation baffle had to be removed for structural reasons.

An experiment using the capillary centrally-off-set evaporation tube failed to give condensation while a later attempt with the 0.15" tube gave similar troubles. To prevent radiation travelling down the wider tube a lateral stagger of copper tubing was installed inside the can and to damp oscillations a brass bushing with a constriction was used just above the calorimeter. Impurity blockage was suspected as the reason for non-condensation but calculations based on the known composition of the helium gas used showed this to be virtually impossible.

Reference was made at this point to the apparatus used by Berman, Poulter and Mate in their work on latent heat and specific heat problems. There is was noted the evaporation pipe did not leave the calorimeter at the top but at the side. This idea was copied, as in Figure 6 (f), and condensation was achieved; a boil-off of ~6 cc/min decreasing after 3 hours to 2 cc/min resulted.

A suggestion to use a cotton batten plug (Ref. 23, 19) to damp the oscillations was tried. The calorimeter pipe used was a simple 0.15" straight tube from the top central position and a boil-off of 5 cc/min steady over a period of 3 hours occurred. So in this apparatus it did not prove to be a solution of the problem.

At this stage experimental investigation of the problem terminated.

It might be noted that in comparable apparati at Oxford and Leiden no such formidable difficulties were encountered. In both these centres it is customary to use liquid hydrogen rather than liquid air shielding round the insulating vacuum jacket. Perhaps a 20°K bath imposes a better temperature distribution on the evaporation pipe for damping possible oscillations than is the case with a 77°K bath.

CHAPTER IV

CONCLUSIONS

Analysis of the individual experiments just discussed will show that no satisfactory determination of the latent heat of evaporation was performed. Much ado has been made of the unexpectedly high background boil-off of gas. Calculations had set an upper value on this of 0.1 cc at N.T.P. without electrical leads connected and 0.15 cc with leads installed whereas in most experiments values 10 to 100 times these were encountered. The inability to account for such behaviour in itself constitutes justification for concern in the mind of an experimenter but it would not be possible to conduct latent heat determinations in any case with such large background even if the cause were well understood. In the first place the volume available in a precision bore tube is limited since the diameter must be small enough, say 1 cm, to give visible mercury level changes for small volume changes and yet it must be long enough to accommodate the background plus heater-induced gas boil-off summed over the period of a complete run. Technical difficulties limit the length of accurate bore tubes to about 200 cm so a small background is imperative. Secondly, the calculations of latent heats rely on the initial and final background rates being identical to give accurately parallel slopes on a graph as otherwise mathematical ambiguity arises. The smaller the slope therefore, the less possibility there is of error. A typical curve shown in Figure 7 illustrates these points. To get final results of 0.1% accuracy boil-off in excess of 0.15 cc per minute at N.T.P. is not tolerable.

LATENT HEAT DETERMINATION — TYPICAL CURVE

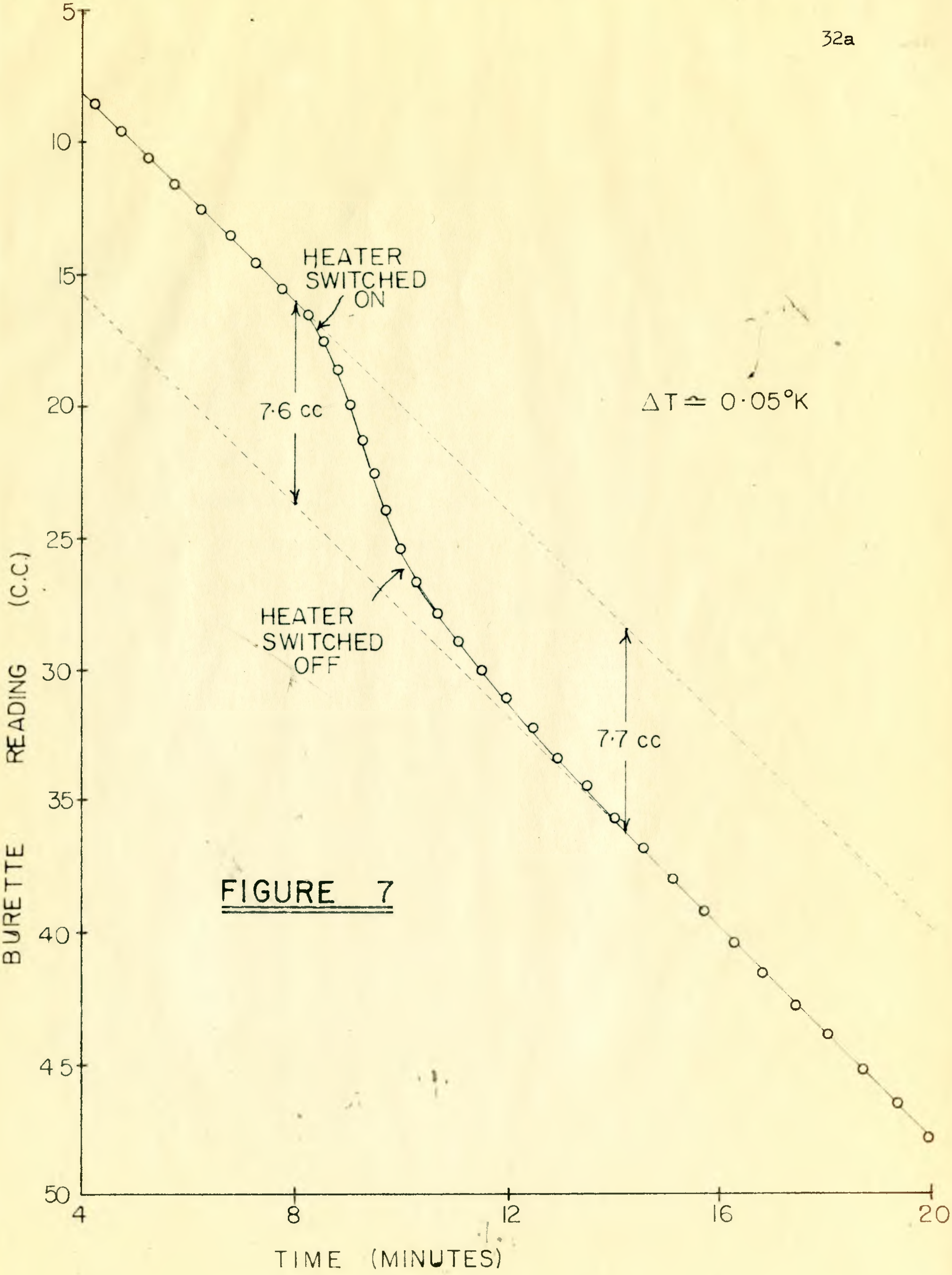


FIGURE 7

Why was this value not achieved as predicted? Much evidence for elimination of radiation, conduction and convection as the mechanisms of the heat leak was obtained from the experiments carried out. Variation of the parameters that control these leaks could not be correlated with the large background in question. Particularly noteworthy was the randomness of the heat leak and it was this aspect which suggested a dynamic mechanism within the helium of the calorimeter itself. Since Taconis (thermal) resonance has been known to cause such behaviour it was suspected as the source of the trouble. Many structural modifications were carried out in an attempt to damp the oscillations but only once did complete success seem to have been achieved. In this case the evaporation pipe contained a length of fine copper capillary tubing which must in retrospect be adjudged as having been the most effective form of damping. However, it seems that since all the evidence for oscillations was circumstantial rather than direct, the difficulty would command much attention before a satisfactory solution is found. For example, an investigation of the conditions under which oscillations may be sustained would be invaluable. Parameters such as temperature distribution, size shape and dimensions of cavity, and pressure would all be expected to have some influence. It has been the practice in cryogenic laboratories in the past to neglect the general problem raised and settle for a solution by trial of each case when and if it occurred. Now that cryogenics is of such widespread application any progress in this direction would be widely appreciated.

Some experimental runs were later realised to have been useless in so far as condensation had apparently not occurred in the calorimeter. Despite overpressures of 2 atmospheres the helium seems resolutely to have remained in a supersaturated state. It was felt at the time that the initial decrease in pressure within the condensation reservoir denoted condensation into the calorimeter: a sudden refusal to condense further was not treated as a serious impediment to progress in the experiment. However it now seems that the initial decrease was merely gas contraction to a supersaturated state. The extent of the supersaturation was much greater here than the critical values measured by Edwards and Woodbury (Ref. 19) in an investigation of the problem in glass containers. Differences in the shape and material of supersaturated vapour containers have long been recognised as dominant features in determining the critical values so it is felt that no real conflict of results arises.

SUGGESTIONS FOR FUTURE WORK

It has already been suggested that further investigation of the latent heat problem with this cryostat should be delayed until a separate analysis has been made of oscillation phenomena. However, the apparatus is quite versatile and could conveniently be converted to other researches at helium temperatures. Many experiments that do not involve gaseous coupling to room temperatures would be feasible. There is available in the cryostat within the radiation shield, a volume of some 400 cc to which the heat leak is less than 10^{-7} watts. To introduce electrical leads and exchange gas facility only relatively minor structural modifications would be necessary and two types of experiment that seem possible are specific heat determinations on solids and an analysis of the thermal conductivity of helium gas especially in the interesting Knudsen region.

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