

A CRYOSTAT  
FOR  
LATENT HEAT DETERMINATIONS

A CRYOSTAT FOR DETERMINATIONS OF THE  
LATENT HEAT  
OF  
LIQUID HELIUM

By

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SCOPE AND CONTENTS:

A cryostat has been constructed for the determination of latent heat of vaporization of liquid helium at temperatures from the critical temperature ( $5.2^{\circ}\text{K}$ ) to below the  $\lambda$  point ( $2.2^{\circ}\text{K}$ ). Some details of the apparatus, hints on low temperature technique and suggestions for future equipment are presented, together with results of some experiments on the apparatus.

## PREFACE

The cryostat described in this thesis was built during the first year of low temperature research at McMaster. For this reason ample space has been devoted to details of construction and cryogenic technique. If the reader is interested only in a general description of the project and results he may omit sections II 2b, II 3b, II 7b, and III 2a-f with no loss of coherence.

The author wishes to acknowledge the assistance and encouragement given by his supervisor, Dr. C.F. Mate, under whose direction the project was carried out. Thanks are also due to the author's colleague in the Low Temperature Laboratory, Mr. D.E. Brodie, M.Sc., for hints and help in construction, and to the author's brother, Mr. G.A. Roeder who helped with the drawings in this thesis. Finally, it should be noted that the author is indebted to the National Research Council of Canada for a graduate bursary.

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## INTRODUCTION

The apparatus discussed in this thesis was built for the purpose of measuring the latent heat of vaporization of liquid helium at various temperatures, in particular near the critical point ( $T_c \sim 5.2^\circ\text{K}$ ) and the lambda point, ( $T_\lambda \sim 2.2^\circ\text{K}$ ). As will be indicated, measurements of latent heat can be used to determine the density of the saturated vapour. Values of this quantity near  $T_c$  are valuable for two reasons:

- (a) comments can be made on the consistency of any temperature scale in this region, (ref. 1)
- (b) direct comparison with vapour densities calculated from theory can be made and the number of virial coefficients necessary to give adequate representation may be examined, (refs. 1, 2, 3, 4, 5.).

Careful measurements near  $T_\lambda$  are necessary to show whether any evidence as to the nature of the  $\lambda$  transition appears in the latent heats. Finally, it is desirable to know the latent heat as a function of the temperature in order to be able to extrapolate to  $L_0$ , the latent heat at absolute zero which is a quantity of some thermodynamic interest (ref. 5). For these purposes it was deemed necessary to have an accuracy of at least 0.1% in the measured values.

The actual quantity to be measured in the experiments is the apparent latent heat,  $L_a$ , defined as the heat required to expel one mole of helium vapour from a calorimeter containing liquid. It is related to



the true latent heat,  $L$ , by the expression

$$L = L_a \left( 1 - \rho_v / \rho_l \right) \quad (1)$$

where  $L$  and  $L_a$  are defined as above,  $\rho_v$  and  $\rho_l$  the densities of saturated vapour and liquid. The fraction  $\rho_v / \rho_l$  represents a correction for the vapour that occupies space formerly filled by liquid. The expression is derived in the appendix.

Since the change of phase from vapour to liquid is of the first order, the Clausius - Clapeyron equation can be applied along the liquid-vapour transition curve on a pressure-temperature diagram. But this curve is just the helium vapour pressure temperature scale. Thus the temperature scale enters the picture.

The Clausius - Clapeyron equation is

$$\frac{dp}{dT} \Big|_T = \frac{\Delta s}{\Delta v} \quad (2)$$

where  $\frac{dp}{dT} \Big|_T$  is the slope of the demarcation curve at  $T$ , the temperature at which the transition occurs,  $\Delta s$  and  $\Delta v$  being respectively the changes in specific entropy and volume upon completion of the phase change.

(2) may be rewritten as

$$\frac{dp}{dT} = \frac{L}{T \Delta v} = \frac{L}{T \left( \frac{1}{\rho_v} - \frac{1}{\rho_l} \right)} \quad (3)$$

upon substituting for  $L$  in (1) we see that

$$\rho_v = \frac{T}{L_a} \frac{dp}{dT} \Big|_T \quad (4)$$

One may also eliminate  $\rho_v$  between (3) and (1) to get

$$L = L_a - \frac{T}{\rho_l} \frac{dp}{dT} \Big|_T \quad (5)$$

(4) shows that if  $L_a$  is accurately known and a vapour pressure temperature scale is provided  $\rho_v$  may be calculated. According to (5),

if our values of  $L_a$  show very small scatter we can extrapolate the curve to absolute zero and obtain  $L_0$  quite accurately without calculating  $L$ .

The measurement of the vapour pressure over a helium bath is the standard laboratory method of determining temperature in the liquid helium region. The vapour pressure scale of temperature recommended by the "Comité consultatif de Thermometrie" of the "Comité international des poids et mesures" is known as  $T_{58}$ , (ref. 6.) It was first published at the end of 1958 jointly by the Kamerlingh Onnes Laboratory of Leyden and the U.S. Naval Research Laboratory, Washington, and represents an amalgamation and recalculation of the two former scales  $T_{L55}$  and  $T_{55E}$  which were respectively promulgated by the above laboratories in 1955. It is hoped to use this new and more accurate scale in conjunction with the measurements of  $L_a$  for the purposes outlined above.

The values of  $p_v$  thus calculated may be compared with those obtained from the equation of state of the vapour, in virial form, i.e.,

$$p v_s = R T \left( 1 + \frac{B}{v_s} + \frac{C}{v_s^2} + \dots \right) \quad (6)$$

since the density and specific volume of the saturated vapour are reciprocals:  $p_v = \frac{1}{v_s}$ .

Values of the second virial coefficient have been obtained by Keller (ref. 2, 3) who also concluded that in his range of measurement it is not necessary to carry the third virial coefficient,  $C$ . Since Keller did not make his measurements at saturation pressure, any difference between calculations of the vapour density from (4) and (6) can be expected to clarify the magnitude and number of higher order virial coefficients necessary for adequate representation of the saturated vapour.

The reason for the desired precision of 0.1% should now be

apparent. A glance at equations (4) and (5) will show that if  $L_v$  is accurately measured, the major uncertainty in the calculation of  $\rho_v$  and  $L$  will come from the quantity  $\left. \frac{dp}{dT} \right|_T$ , since  $T$  itself and  $\rho$  are now known quite precisely. The quantity  $\left. \frac{dp}{dT} \right|_T$  was different for  $T_{L55}$  and  $T_{55E}$ , the percentage difference being variable and usually much greater than the percentage temperature difference on the two scales. It may be noted that the slopes  $\left( \frac{dp}{dT} \right)$  of nearly all helium vapour pressure temperature scales so far published diverge widely as  $T_c$  is approached. Plots of  $L$  and  $\rho_v$  will help to show how smooth  $\left. \frac{dp}{dT} \right|_T$  is for  $T_{58}$ , and this in the experimentally difficult region near  $T_c$ .

Values of latent heat published by Berman and Mate (ref. 1) showed differences from other values of  $\frac{1}{2}\%$  to  $1\frac{3}{4}\%$  over the common temperature range. Their comparison of saturated vapour densities (viz.  $\rho_{\text{virial}} \approx \rho_{L_v}$ ) suffers from the admitted possibility of inaccuracy in  $L_v$  as well as from the fact that the previous temperature scale  $T_{55E}$  was used. Indeed, their comparison, pointed out some defects in this scale. It is therefore expected that, using  $T_{58}$  and values of  $L_v$  accurate to 0.1% or better, a worthwhile estimate of the effects of higher order virial coefficients can be obtained.

The following pages contain an account of the author's contribution to the program of latent heat measurements suggested by the above discussion, namely the development of a suitable cryostat design.

## II

### APPARATUS

#### 1. General:

Precise measurement of latent heat by the direct method obviously means precise measurement of the heat input to, and the amount of gas evolved from, a calorimeter. That this is no small task may be seen by glancing at the following tabulation of factors affecting the determination of the heat input and of the mass of gas evolved:

##### (a) Heat input;

- (1) precise wattage measurements at the calorimeter heater
- (2) unwanted joule heat generated in the heater leads
- (3) heat influx by thermal conduction and by radiation
- (4) thermal oscillations in the helium vapour above the calorimeter.

##### (b) Gas evolved;

- (1) precise buretting
- (2) the  $\frac{R}{\rho}$  correction factor
- (3) temperature expansion effects in the burette system
- (4) changes in temperature gradients within the cryostat
- (5) a leak-proof system.

The control of these factors, with the exception of a .4, will be discussed with the various parts of the apparatus that they affect. It

is not possible to make a correction for heat influxes by thermal oscillations (i.e. the well-known Taconis oscillations) as it cannot be determined beforehand whether or not they will arise in any given system.

With due attention to these points an apparatus was designed to carry out the following experimental sequence: a small calorimeter was to hold a few cubic centimeters of liquid helium evaporating slowly because of unavoidable heat leaks. By monitoring the vapour pressure above the calorimeter, the temperature of the liquid could be obtained. When it had reached the desired value, the vapour pressure was to be held steady and the background evaporation rate measured by trapping the gas evolved in a burette tube. A heat pulse would then be supplied to the calorimeter. After the background rate had reached its former value, the amount of gas evaporated could be measured from a plot of burette readings versus time (as in fig. 6).

The design and construction of the first cryostat and its auxiliary equipment can now be discussed.

## 2. The Cryostat:

(a) The cryostat (literally: "cold regulator") is the equipment which actually holds the liquid helium, a fact which made careful design imperative. The first necessity was to get a small calorimeter as completely isolated as possible, with a minimum of connections to the outside world which is, in this case, an excellent example of the thermodynamic "infinite reservoir" from which any quantity of heat may be extracted. This suggested first of all a vacuum jacket, and secondly a main helium bath to absorb most of the incident heat. Since a Collins Helium Cryostat was available for the production of liquid helium at

rates up to  $8 \text{ l/h}$ , it was felt that the main bath could be quite large. To keep the equipment as simple as possible, it was decided to use the standard doubledewar arrangement - an inner dewar containing liquid helium enclosed by an outer dewar containing liquid air, both dewars being slit silvered to enable the experimenter to observe the main bath level.

The inner dewar had to be sealed at the top since air will condense into an open dewar of liquid helium, causing it to be rapidly vaporized. This top seal was accomplished by using a graded-glass to Kovar seal above the dewar proper, the Kovar being soldered to a brass flange which in turn could be bolted to a fixed brass plate to support the dewar from the top (see fig. 1). The outer dewar could then be supported from below.

The vacuum can was placed at the bottom of the helium bath where it would be kept at the main bath temperature. This temperature was to be controlled so that it was approximately  $0.1^\circ\text{K}$  above the temperature of the calorimeter liquid, thus keeping the heat leak to the calorimeter by conduction to a minimum while preventing recondensation of boiled off vapour in the evaporation tube. Calculation using the heat transfer equation

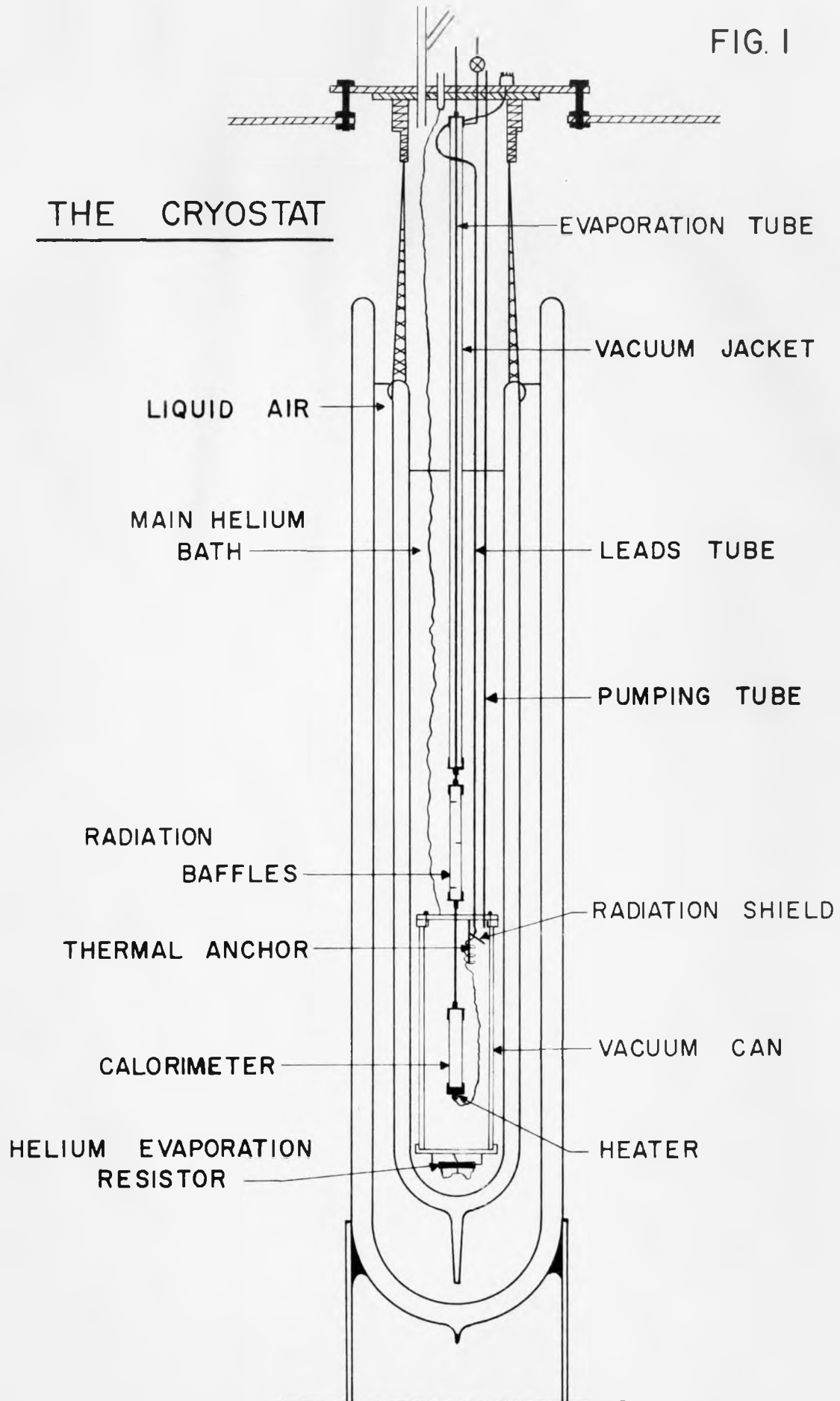
$$\dot{Q} = K(T) A \frac{dT}{dx} \quad (7)$$

showed  $\dot{Q} \sim 3.1 \times 10^{-6}$  watts

was the magnitude of the heat leak by conduction down 7 cm. of  $\frac{1}{4}$ " german silver tube with a .005" wall. Since 1 milliwatt causes a boil-off of about 12 cc/min. of gas (N T P), the contribution to the evaporation rate by heat conduction down the tube was calculated as .05 cc/min.

There was also the possibility of radiation from the room

FIG. 1



temperature end funnelling down the evaporation tube and making an added contribution. This was kept to a minimum by putting a copper spiral into the tube just above the vacuum can. Later experiments caused this to be changed to a set of baffles. (See section IV, 2, p. 21)

The calorimeter itself had to be constructed to allow maximum heat transfer from the heater to the helium while preventing loss of heat by conduction up the calorimeter walls. The bottom of the calorimeter was therefore made of the copper former onto which the heating coil was wound. (see fig. 3). The walls were made of a section of  $\frac{1}{2}$ " german silver tube with a .010" wall and of such a length that the volume enclosed would be  $5\text{cc} < V < 10\text{cc}$ . The actual calorimeter volume is 7.2 cc.

(b) Assembly of the vacuum can caused some trouble as it had to be detachable, yet withstand the shock of sudden cooling to low temperatures. It was eventually decided to bolt the can to its top flange using an indium wire O-ring to ensure a vacuum seal. The method of taking the heater leads out of the can then presented a problem.

A cold-setting epoxy-type resin was tried for a vacuum seal through which to take the leads at the top of the can, but was found unsatisfactory. The epoxy resins must evidently be cooled very slowly and treated as extremely fragile even at liquid air temperatures. Exceptions to the above statement are the epoxy resins marketed under the trade name "Araldite". These have been used successfully at liquid helium temperatures but unfortunately were not available to this laboratory.

It was finally decided to take the leads through a german silver tube to the room-temperature end of the dewar and make the vacuum seal there. This necessitated a thermal anchor and radiation shield being placed at the



cold end of the leads tube. More will be said about this in a discussion of the electrical system.

All joints, tubes, bushings, etc. were soft-soldered. During the leak testing stage when and/or where it was not possible to stop a leak with soft solder, Woods metal was used. This is a low melting-point ( $50^{\circ}\text{C}$ ) solder which will successfully hold at liquid helium temperatures provided that no great or sudden strain is placed upon it. Used with  $\text{ZnCl}$  flux and a copper iron it is ideal for the purpose mentioned.

The top flange of the cryostat was made of  $\frac{1}{4}$ " brass plate. It was fitted with leveling screws and drilled to receive the helium dewar as well as the various tubes from the vacuum can and calorimeter. Also provided was a  $\frac{1}{2}$ " hole for the vapour pumping line and transfer siphon.

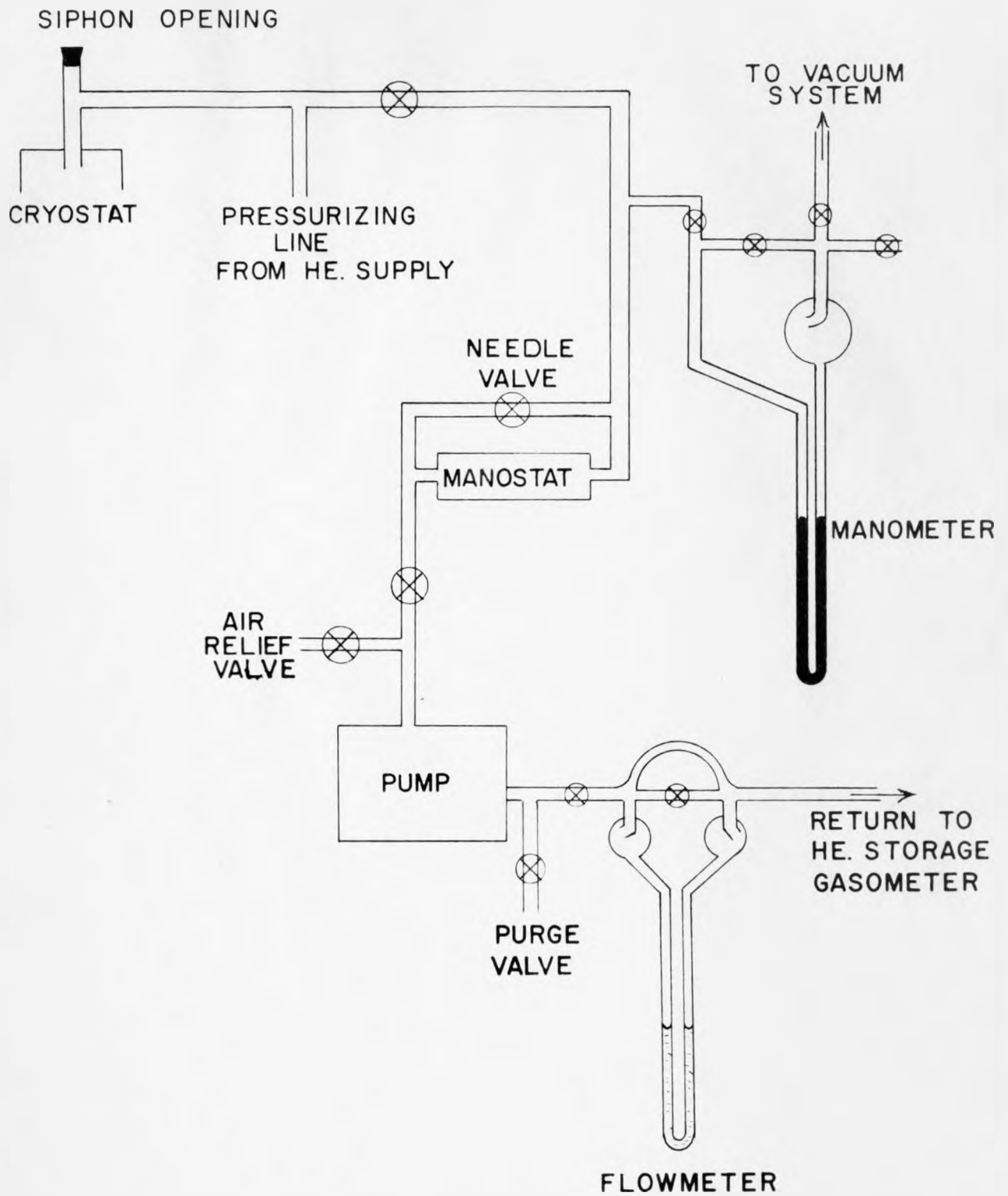
### 3. The Vapour System:

(a) In order to maintain the vapour pressure (and hence the temperature) of the main bath at any desired value the vapour pumping system depicted schematically in fig. 2 is used. The main line is made of  $\frac{1}{2}$ " copper pipe with rubber hose connections where necessary. Soft solder joints are used throughout. The manometer, manostat and flowmeter are of glass.

Since liquid helium cannot be allowed to come into contact with air, it is necessary to have a means of putting He gas into the cryostat before the liquid is syphoned in. Hence a pressurizing line from the He supply is included.

(b) In the course of an experiment the cryostat pressure is monitored on the manometer and allowed to reach a desired value. This may be done by allowing the pressure to build up or by pumping. When the

FIG. 2



THE VAPOUR PUMPING SYSTEM

desired pressure is reached, the needle valve by-passing the manostat is closed and the manostat set to retain the selected value. Many types of manostat are available, the most common being the Cartesian diver (see ref. 7). When cryostat pressure rises above that selected, the diver is forced down, thus opening a pumping orifice. When the pressure in the cryostat falls below the set level, the diver rises and closes the orifice.

Gas from the manostat is pumped through a flowmeter and back into a storage gasometer. From the gasometer it may pass into the liquefaction cycle or be stored in a high pressure tank by means of a special recovery system built into the liquefier. Since the supply of helium is somewhat uncertain the use of return lines and a recovery system is good laboratory practice.

#### 4. The Condensation System:

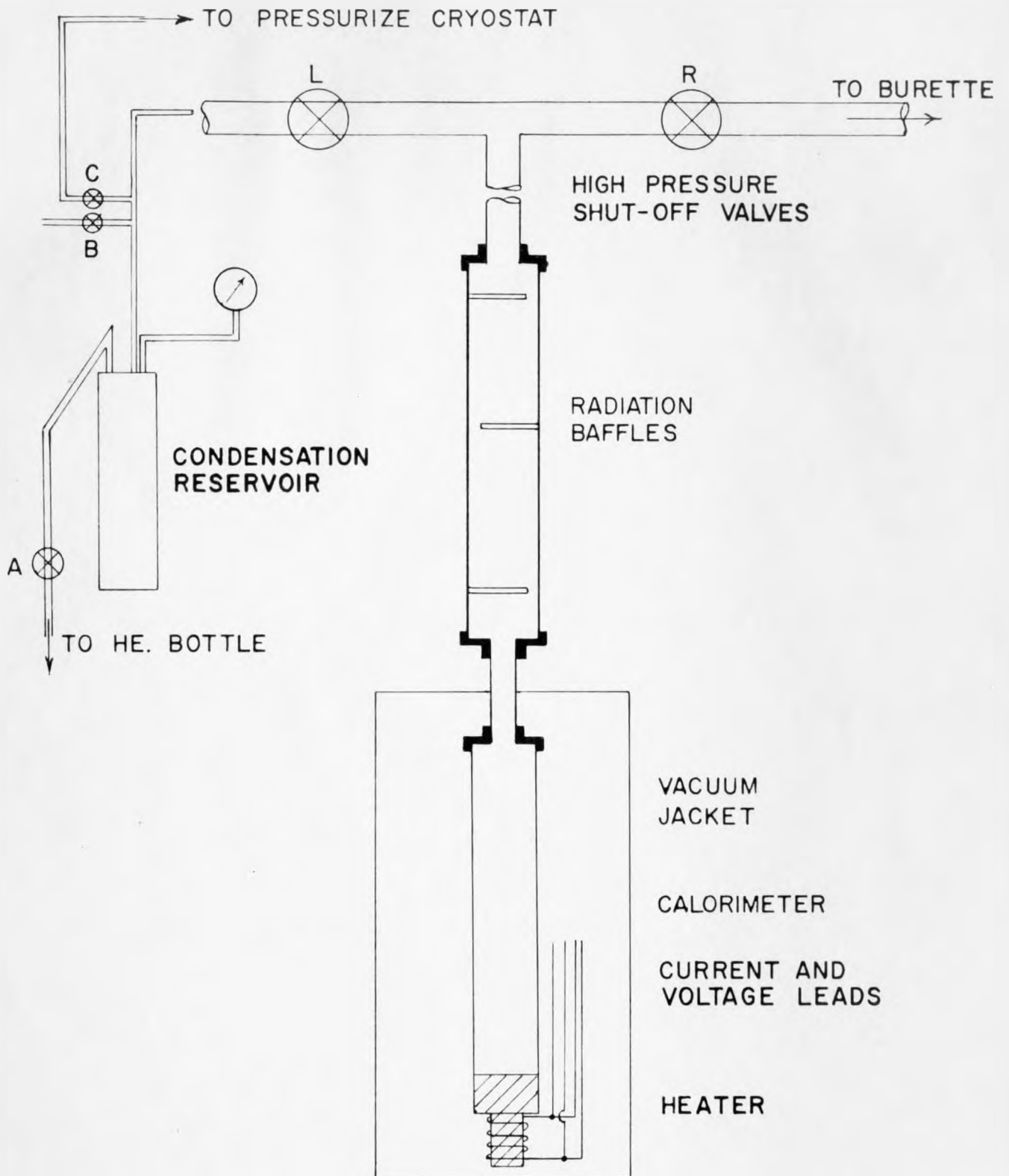
In order to fill the calorimeter with liquid, a condensation process is used. The apparatus necessary for this is shown schematically in fig. 3. The volume inside the calorimeter is 7.2 cc. The volume of the condensation reservoir is 930 cc. Now 700 cc. of helium gas at N T P are equivalent to 1 cc of liquid at  $4.2^{\circ}\text{K}$ , 1 atmosphere pressure. Hence if the condensation reservoir were filled to 1 atm. and all the gas then pushed into the calorimeter, being cooled by the main bath, on the way in, ~ 1.3 cc of liquid would be formed.

From the temperature-enthalpy-pressure diagram of helium (ref. 8) it is found that in the above process

$$\Delta h = 5880 \text{ j/mole}$$

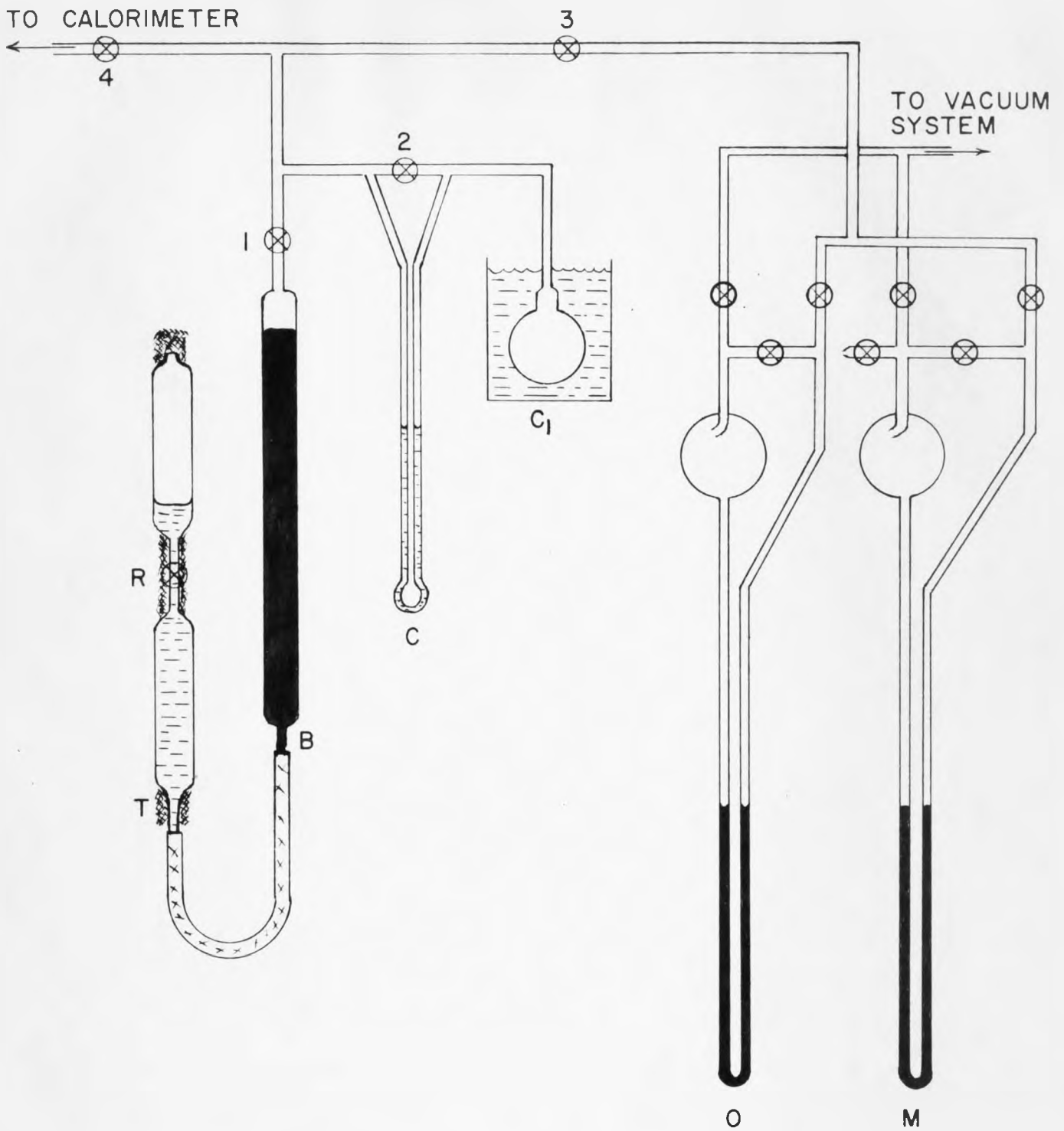
This must be transferred to the main bath as heat. As a result, since the latent heat of helium is 83 j/mole at  $4.2^{\circ}\text{K}$ , it is expected that

FIG. 3



THE CONDENSATION SYSTEM

FIG. 4



THE BURETTE AND MANOMETERS

~ 70 cc of the main bath will be vaporized for every cc of liquid produced in the calorimeter.

In practice, the valves B, C and R are closed, the reservoir and calorimeter filled with gas to 1 atm. and the valve L then closed. The pressure in the reservoir is increased to 2 atm and the valve A is closed. Valve L is then opened. The pressure in the reservoir drops rapidly to 1 atm. and approximately 70 cc of main bath liquid are vaporized. The sudden influx of heat is indicated by a sudden change in cryostat pressure which blows out of the siphon opening a cotton plug placed there for this purpose. Valve L is then closed, A opened and the condensation reservoir refilled to 2 atm. The whole process is then repeated not more than four times after which the calorimeter will be full.

#### 5. The Burette and Manometer:

To measure the amount and monitor the pressure of the gas evaporated from the calorimeter during an experiment, the burette and manometer system illustrated in fig. 4 is used. This is made entirely of glass, with the exception of the valve R which is a Hoke regulator valve. O and M are manometers, filled with butyl phthalate and mercury respectively. Since the density of the oil is  $\sim \frac{1}{15}$  that of mercury, pressures near the  $\lambda$  point (37.80 mm of Hg) and below will be measured more accurately by O while pressures near and above the normal boiling point can be measured on M.

C is a comparison manometer filled with Cenco high vacuum oil and C, a 1-l flask in a constant temperature bath which will maintain a "room" temperature without the small fluctuations of the latter. The

burette tube B contains 150 cc of mercury. It is connected to a movable trolley T by means of a rubber hose. The trolley carries 2 bulbs each of volume 150 cc; the lower bulb contains oil which passes through R into the upper as the mercury level falls in the burette. This is to provide control over the rate at which mercury in the burette drops.

During the course of an experiment when it is desired to monitor the background evaporation from the calorimeter stopcocks 2, 3 and 4 are open, 1 is closed. The appropriate stopcocks on the manometers are also open. When the desired pressure is reached, stopcocks 2 and 3 are closed, and at the same time 1 is opened. A sample of gas at the appropriate pressure has thus been caught in the comparison bottle C, . The regulator valve R is then manipulated so that the mercury in the burette drops at a rate such that the oil levels in the two arms of the comparison manometer remain coincident, or as nearly the same as possible. With a little practice the oil levels can be maintained within  $\pm 1$  mm of each other, corresponding to temperature fluctuations of less than  $\pm 10^{-4}$  °K for calorimeter temperatures of about 4°K. When the burette has been filled, stopcock 4 is closed and the helium is sucked out via the vacuum system, allowing the mercury to refill the burette tube.

The inside diameter of the present tube is  $3/4$ " precision bore, its length 50 cm. This pattern of burette is not sufficiently accurate for the latent heat determination. If the tube is narrow enough for good background evaporation plots, it will not accommodate the comparatively large amount of gas evolved by the heater, unless the burette is very long ( $>1$  metre) in which case it is difficult to meet the requirement of uniform bore. It was planned, therefore, to use the present

tube only for testing and to replace it later by a calibrated bulb with narrow arms at top and bottom. Unfortunately, the background evaporation rate has proved very troublesome as will be discussed in Chapter IV.

#### 6. The Electrical Circuit:

The electrical circuitry necessary is quite simple, as shown by fig. 5. Since the value of heat input is desired as accurately as possible, voltage and current are measured potentiometrically. It is also necessary (for the leads to the calorimeter) to strike a balance between conflicting electrical and thermal requirements, i.e. (1) high electrical but (2) low thermal conductance. The Wiedemann-Franz-Lorenz law:

$$\frac{\kappa}{\sigma T} = \mathcal{L} \text{ (a constant)} \quad (8)$$

where  $\kappa$  is the coefficient of thermal conductivity

$\sigma$  is the coefficient of electrical conductivity

and  $T$  the absolute temperature,

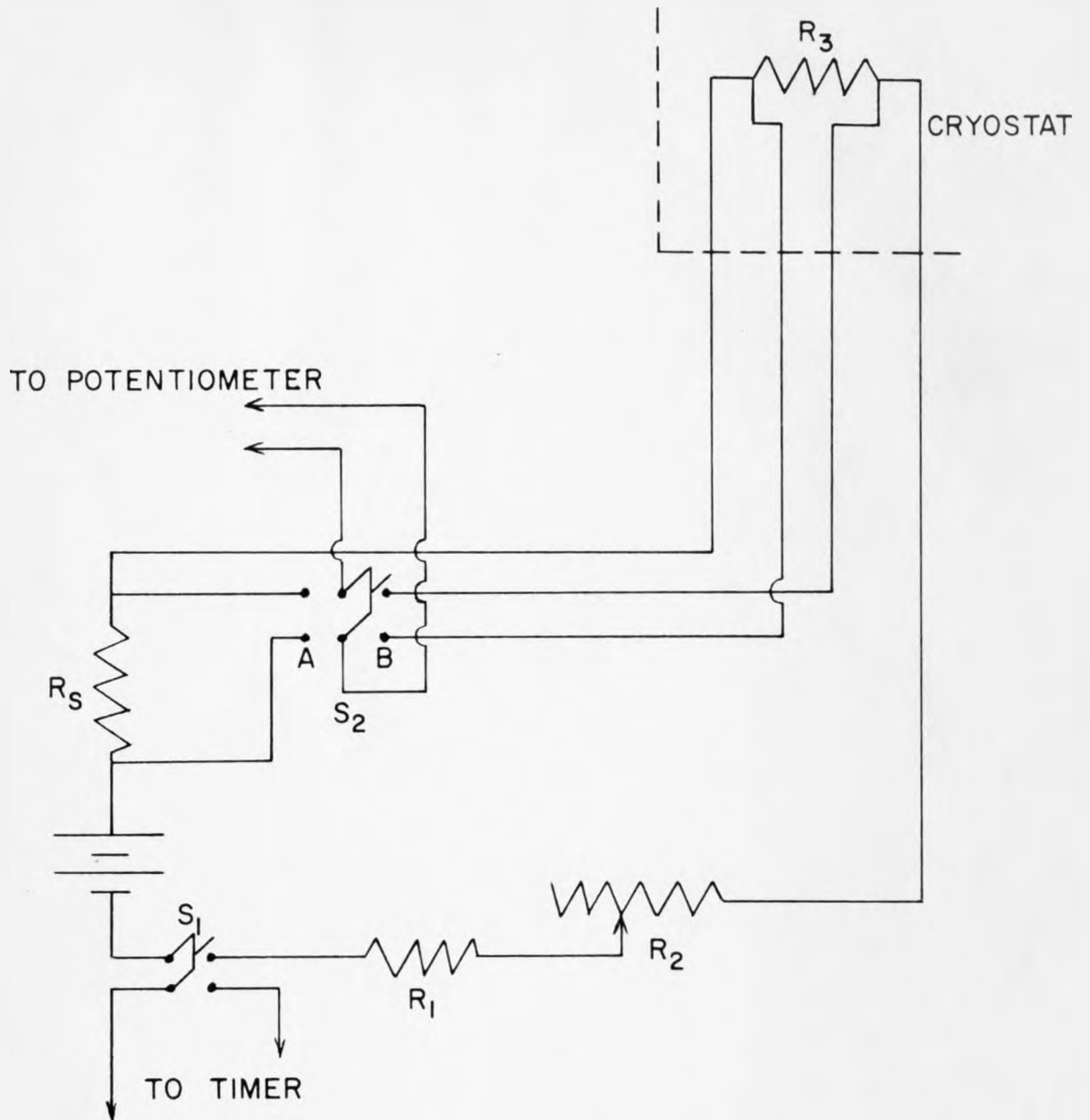
shows that this cannot be entirely achieved. The best that can be done is to choose an optimum diameter for the heater leads. Condition (1) implies short, thick leads, whereas (2) indicates the exact opposite.

It was decided to anchor the leads thermally to the temperature of the main bath. This was done by winding the leads around a small vertical brass rod which was soldered to the inside of the vacuum can (see fig. 1) and cementing them there firmly using nail polish. It was felt that this would remove any heat conducted down the leads.

The heater,  $R_3$ , was to be run at a power of  $\sim 0.4$  milliwatts with a current of  $\sim 2$  m.a. The electrical power produced in the current



FIG. 5



THE ELECTRICAL CIRCUIT

leads between the thermal anchor and calorimeter was thus restricted to  $\leq 0.0004$  milliwatts. To keep the back-ground boil-off rate down, the thermal heat conducted in from the vacuum can (which is  $0.1^{\circ}\text{K}$  above the calorimeter) had to be of the same order as that conducted down the evaporation tube. Calculations showed that copper leads,  $.005''$  in diameter (36 gauge B & S) were suitable for the current. The voltage leads, which carry no current, were made of 30 gauge nickel silver wire which has a low thermal conductivity.

$R_3$  was wound on a copper former with enameled,  $.0015''$  diameter capel wire ( $142 \sim/\text{ft}$ ) to a value of  $140 \sim$  at room temperature.  $R_5$  is a  $100 \sim$  standard resistor.  $R_1$  is a current limiting resistor and  $R_2$  allows the current to be varied.

By flipping switch  $S_1$  to close the heating circuit, an auxiliary circuit to a Beckman model 5230 Universal E.P.U.T. meter and Timer is also closed. While  $S_1$  is closed,  $S_2$  is flipped, first to A and then to B to obtain the current and voltage readings on a Tinsley Vernier Potentiometer type 4363-A. After these readings have been taken, and when enough heat has been generated to fill the desired section of the burette,  $S_1$  is opened and the heating time read off the timer.

#### 7. The Vacuum System:

(a) A good vacuum system is a necessity in any low temperature experiment for the obvious reason of insulation. In this apparatus the calorimeter was to be isolated as much as possible from the main helium bath by a hard vacuum. However, since the vacuum can reaches a temperature in the neighbourhood of  $4^{\circ}\text{K}$  during an experiment, good insulation can be produced by having the system pumped down only to

$0.1 \mu$  at room temperature. Any residual air or vapour will be frozen out by the drop in temperature and the pressure of any helium gas will drop to  $< 0.002 \mu$ . In fact, the pumps used - a Cenco "Hy-vac" two-stage rotary pump and a two-stage mercury diffusion pump-can produce a vacuum of  $\sim 0.02 \mu$  at room temperature thus ensuring adequate insulation.

(b) As is usual with vacuum systems, it is important to have no leaks. The reason in this case is somewhat unusual, however. If there is a small air leak at the top of the cryostat into either the pumping tube or the leads tube, the vacuum does not immediately go bad. The air begins to freeze at the first "cold" spot in the tubes. Besides vaporizing extra helium from the main bath, this results in choked tubes which may necessitate ending the experiment prematurely.

### III

#### EXPERIMENTS

##### 1. Nitrogen Test:

Before attempting the first run with liquid helium, the apparatus was tested with liquid air in the main bath and nitrogen condensed into the calorimeter. These experiments showed the need for a condensation reservoir as well as indicating that the apparatus was, generally speaking, satisfactory. Of course, the magnitude of the heat leaks could not be determined because of the large latent heat of nitrogen. Nevertheless, the apparatus was deemed to be working in principle.

##### 2. Experimental Procedure:

General: The experimental procedure when helium is involved may be divided into six phases, the first three of which are applicable to any experiment using helium not liquified within the apparatus, the last three peculiar to this one. The phases are: clean-up, cool down, transfer, condensation, evaporation, shut-down.

(a) Clean-up: Prior to starting, the siphon, with its outer end plugged, is inserted at the top of the cryostat and the inner dewar evacuated by means of the vapour pump. The seals at the top must be checked to ensure that they can hold a vacuum of better than  $500\mu$ . If there is any reason to suspect air contamination in the condensation reservoir, it too must be evacuated. The calorimeter, burette, and

manometer system are also evacuated, along with the vacuum jacket, by the vacuum system. The vacuum jacket of the siphon may be checked at this time by connecting to the main vacuum system. After this is done, the main vacuum system is set to pump the calorimeter vacuum space (and the siphon jacket, if necessary) to less than  $0.1 \mu$ .

(b) Cool-down: Once the inner dewar has been evacuated to  $< 500 \mu$ , the outer dewar is filled with liquid air. Because the inner dewar is made of pyrex, which is porous to helium at room temperature, the liquid air level must be kept topped up above the level of the neck joint on the inner dewar where penetration takes place most easily. The inner dewar can now be filled with helium gas maintained at a pressure slightly greater than atmospheric. The cryostat may then be left to cool slowly toward liquid air temperature ( $\sim 80^\circ\text{K}$ ).

(c) Transfer: This stage is the trickiest part of any experiment for it is here that the unforeseen is most liable to happen. After filling the transfer dewar from the liquefier, the now unplugged outer end of the siphon is inserted into the transfer dewar to which an overpressure of helium gas is then applied. It may be noted here that if the cryostat does not contain the overpressure mentioned in (b), air will enter the siphon when it is unplugged and freeze there when transfer is first attempted.

The first litre of helium transferred will be vaporized as the dewar and contents are cooled to  $4^\circ\text{K}$ . As soon as liquid begins to collect in the cryostat a rapid level drop in the transfer dewar is noted. The overpressure is then increased to hasten the transfer and keep liquid wastage to a minimum. Care must be taken in the final

moments of the operation to avoid a reversal of the pressure gradient in the siphon.

(d) Condensation: After the siphon has been withdrawn, the opening is filled with a cotton plug to allow free evaporation of large amounts of liquid from the main bath during the condensation process. The calorimeter and manometer system, which had previously been evacuated, are now slowly filled with helium gas to a pressure of 1 atm. and condensation is carried out as described in chapter II, 4. After this has been completed, the next step, the actual experiment, may be carried out.

(e) Evaporation: When the desired temperature difference has been produced between the bath and calorimeter (by control of the vapour pressure over each), the background evaporation rate is monitored as outlined in chapter II, 5. After the background has been obtained the heater is switched on for a measured time and then off. When the comparison manometer and burette drop rate indicate a return to stable conditions, the background is monitored again. By plotting burette readings against time, the amount of gas expelled from the calorimeter by the heater is obtained from the difference in vertical displacement of the two (parallel) background times. Having obtained current, voltage and time readings as indicated in II, 6, the data necessary for a calculation of  $L_a$  are at hand.

(f) Shut-down: It is important in low-temperature work to pay particular attention to the state in which the apparatus is left following a run. For instance, if the calorimeter is left full of gas at 1 atm., 4°K, it will be filled with gas at several atm. when the

apparatus has warmed up.

To prevent this, calorimeter and manometer are evacuated while the main bath is left untouched. The remainder of the main bath is then rapidly boiled away by a 5 watt heater, the gas being returned to the storage gasometer for re-use. The inner dewar is then loosened, flushed with air (to remove any helium which could penetrate the pyrex and spoil the vacuum) and left to warm up.

### 3. Scope of Experiments:

The first helium experiment attempted was a failure due to inability to transfer the liquid helium into the apparatus. Transferring is an art learned only by practice.

The second run was successful in this respect but the third was not. Thermal shock succeeded in cracking the stainless steel-brass soft soldered joint on the outer end of the siphon causing the siphon vacuum jacket to go soft.

The succeeding runs were devoted to tracking down the cause of the high and variable back-ground evaporation rate from the calorimeter which was revealed in the second and fourth runs. The observations and conclusions drawn from these experiments, together with consequent modifications to the apparatus, are presented in the next chapter.

## IV

### RESULTS

#### 1. Experimental Accuracy:

The reason that the high background evaporation rates obtained on the first runs are not desired may easily be seen from a discussion of experimental accuracy.

The potentiometer used will give measurements to  $10^{-6}$  volt over the range 0-8 v. Using a current of  $\sim 2$  m.a. this means the percentage errors in the current and voltage readings will be negligible. The timer measures to 1 part in  $10^4$  or 0.01%. These errors are, in any event, random, and may be effectively averaged out by drawing a smooth curve through the experimental points.

Heat generated in the current leads and flowing along them into the calorimeter will not exceed  $3 \times 10^{-9}$  watts at  $4^\circ$  K. This is  $6 \times 10^{-3}$  % of the heater power at the same temperature and is a systematic error for which allowance can be made during calculation.

Thus, to maintain the desired accuracy of  $\geq 0.1\%$  in  $L_a$ , the error in the volume of gas evaporated must be less than this value. This means first of all that the lines giving the background rates must have a very small slope because of the limit set on the amount of gas which can be measured in the burette during evaporation by heater, as mentioned in II, 5. Secondly the background lines must be accurately parallel because of the mathematical ambiguity in determining



the separation if they are not, and also because it is desired that heat leaks to the calorimeter should not change during the heating period. If they do, the true heat input cannot be obtained as the correction for systematic heat error is no longer valid. Thus a high or variable background rate cannot be tolerated.

2. Background determinations:

Since the fourth run indicated a high background rate of evaporation, it was decided to make a new heat anchor for the electrical leads and to replace the radiation shield in the vacuum can at the bottom of the pumping tube with one slightly bigger. The experiment was then repeated and it was found that the background was more stable and reduced to half of its previous value (see fig. 8, run 5).

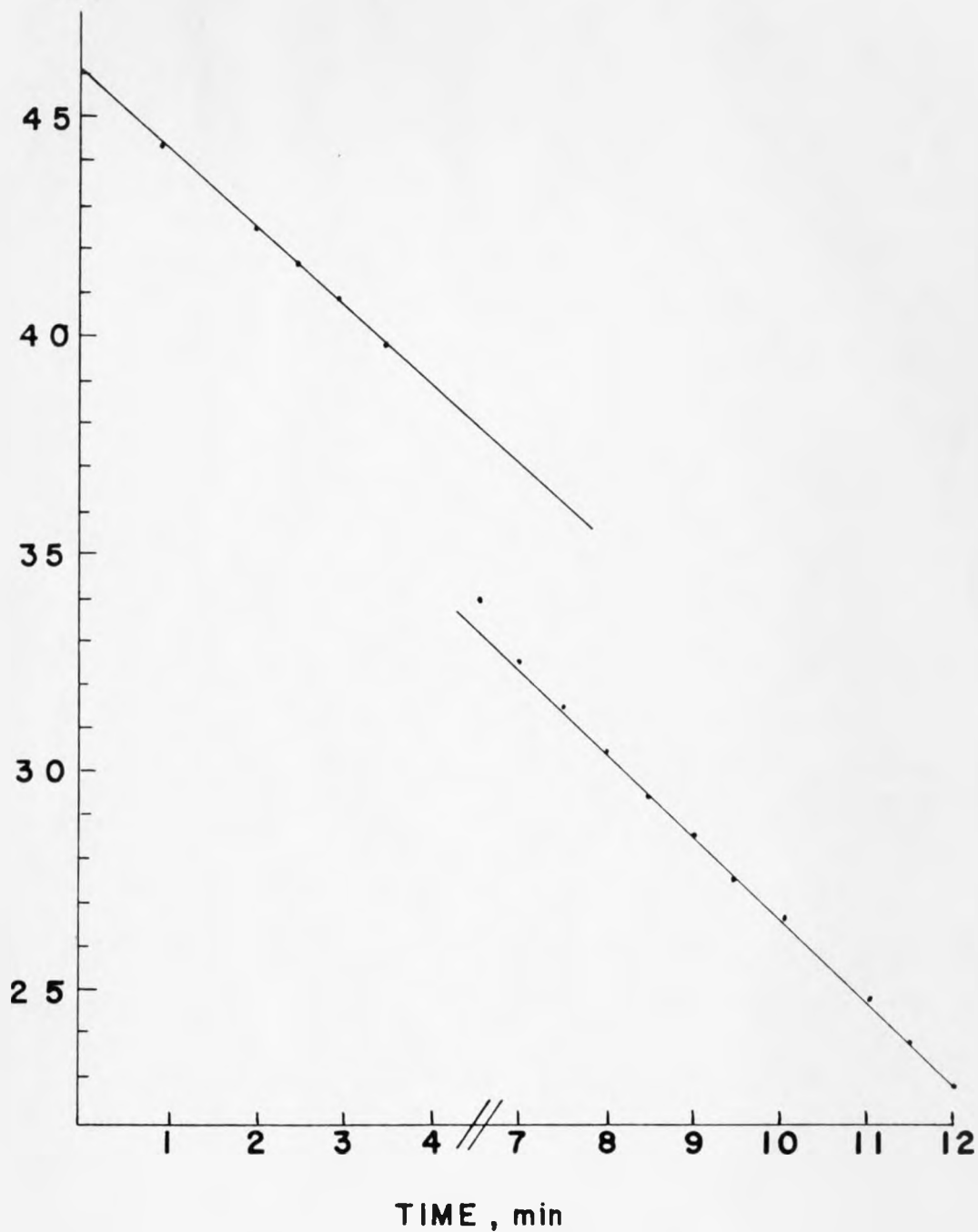
It was noted, however, that the heat pulse appeared attenuated on the evaporation plot and that because of the large background rate, there was some uncertainty as to its true value. (see fig. 6).

It was felt that the thermal anchor might still not be functioning properly. In order to see how much heat was by-passing the anchor, the leads were cut near the heater, the short ends being wrapped around it and the longer ones varnished to the vacuum can lid. Upon repeating the experiment, the evaporation rate was found to be still too high, although reduced by a further factor of 3; i.e. the rate was now  $1/6$  of that in run 4, as shown in fig. 8 (run 6).

After examining the apparatus, it was concluded that part of the remaining high background might be caused by radiation funnelling down the evaporation tube, passing by reflection around the copper spiral, and then entering the calorimeter. The spiral was therefore replaced

BURETTE READING  
cm

FIG. 6



AN EARLY EXPERIMENT, RUN 5

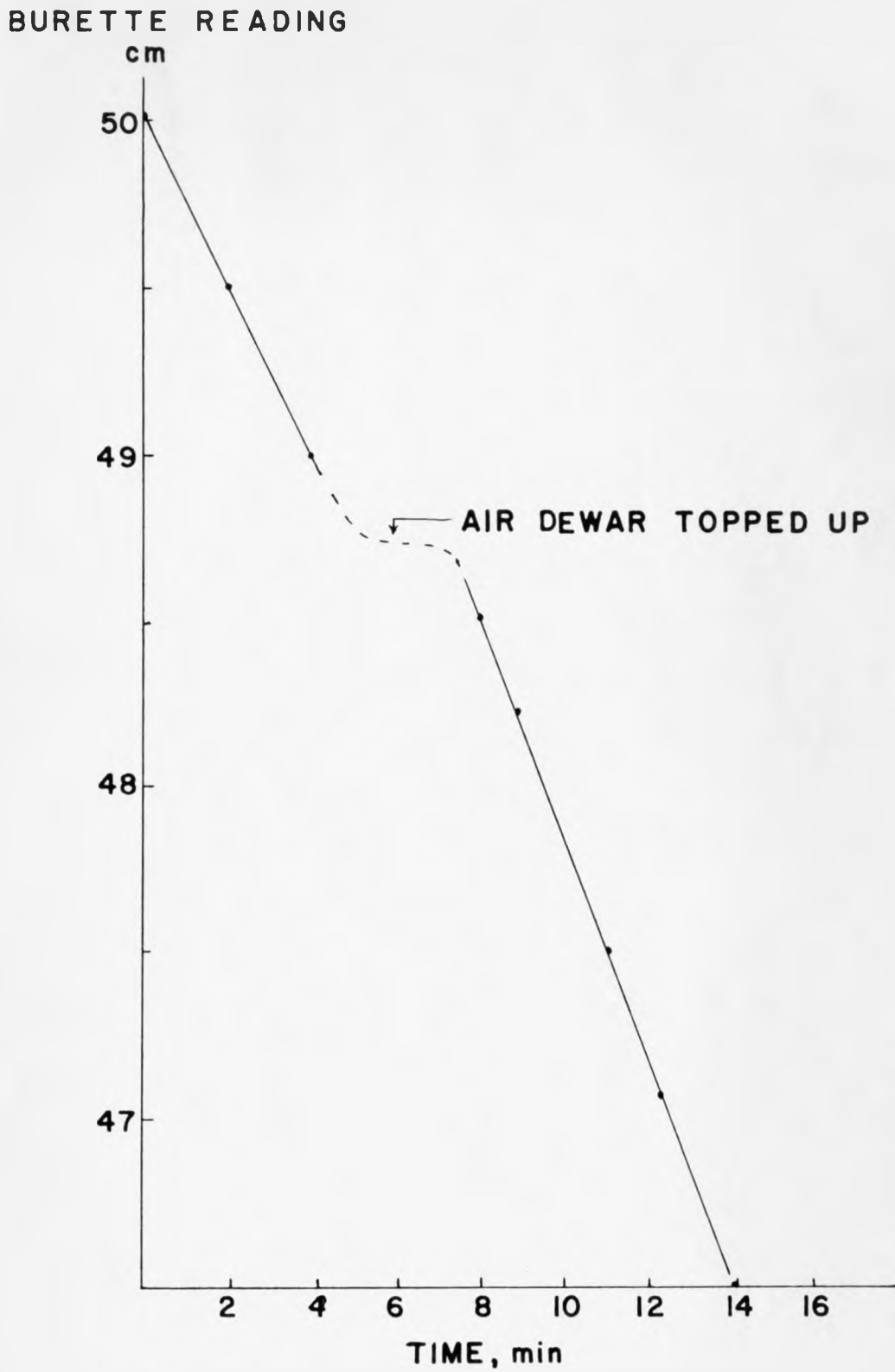
by the set of baffles indicated in fig. 3 and the evaporation rate as measured in the next run was further reduced by 50%. (see fig. 8, run 7).

During this run (no. 7) close attention was paid to a number of other factors which could conceivably be affecting the evaporation rate, which by now was 1/9 of that first observed but still too high (approx. 1cc of gas per minute). It was noted during this run that whenever the outer liquid air dewar was topped up, evaporation almost ceased for a few minutes and then resumed, but not at the previous rate (see fig. 7). Also noted was a yet unexplained trend to higher calorimeter evaporation rates as the main bath level dropped, although, as was expected because of lengthening thermal paths, the boil-off from the bath itself decreased as the level fell. Various values of  $\Delta T$ , the difference in temperature between calorimeter and main bath, were tried; when the results were plotted, little correlation was found between the evaporation rate and  $\Delta T$ , apparently indicating that heat leaks from sources other than the main bath were still too large.

Purely by accident an effect of external radiation was noted. A flashlight was shone into the inner dewar to observe the main bath level. Coincidentally a change in the evaporation rate was observed: there was a marked increase while the light was on, the increase disappearing when the light was shut off. This change was most noticeable when the light was just above the main bath level, and decreased in size as the light was moved toward the top of the dewar.

This radiation effect, completely unexpected, supported a tentative explanation for the high evaporation rate: viz. that as the bath level dropped, the temperature distribution along the evaporation

FIG. 7



INDICATION OF BACKGROUND UNCERTAINTY

tube shifted accordingly and part of the cold gas contained in the tube was warmed and consequently expanded giving rise to a spurious evaporation rate. Now the cross-sectional area of  $\frac{1}{8}$ " tube is  $0.317 \text{ cm}^2$ , hence the volume per millimeter is  $3.17 \times 10^{-2} \text{ cc}$ . The main bath dropped at a rate of  $45 \text{ mm/hr}$ . On the basis of the above "expansion effect" this would cause an apparent evaporation rate of  $1.67 \text{ cc/min}$  if the gas went from  $4^\circ$  to  $280^\circ\text{K}$ . It was probably not correct to assume the tube warming to as high a temperature as  $280^\circ\text{K}$  since cool vapour would still be present close to the liquid level; nevertheless, this calculated rate was near the observed rate.

To try to prevent this "expansion effect" and eliminate sensitivity to the helium bath level it was decided to install a length of thin copper tube immediately above the baffles in the region normally traversed by the bath level. Then, since the thermal conductivity of copper is much greater than that of german silver ( $\frac{K_{\text{Cu}}}{K_{\text{Gs}}} \sim 300$  at  $4^\circ\text{K}$ ), the temperature gradient along the copper section of the evaporation tube should be much less than that along the german silver section. That is, the temperature drop from room temperature to  $4^\circ\text{K}$  should occur almost entirely along the german silver section, the copper tube remaining at approximately  $4^\circ\text{K}$  along its whole length. Calculation of the conductance of each section of tube showed that:

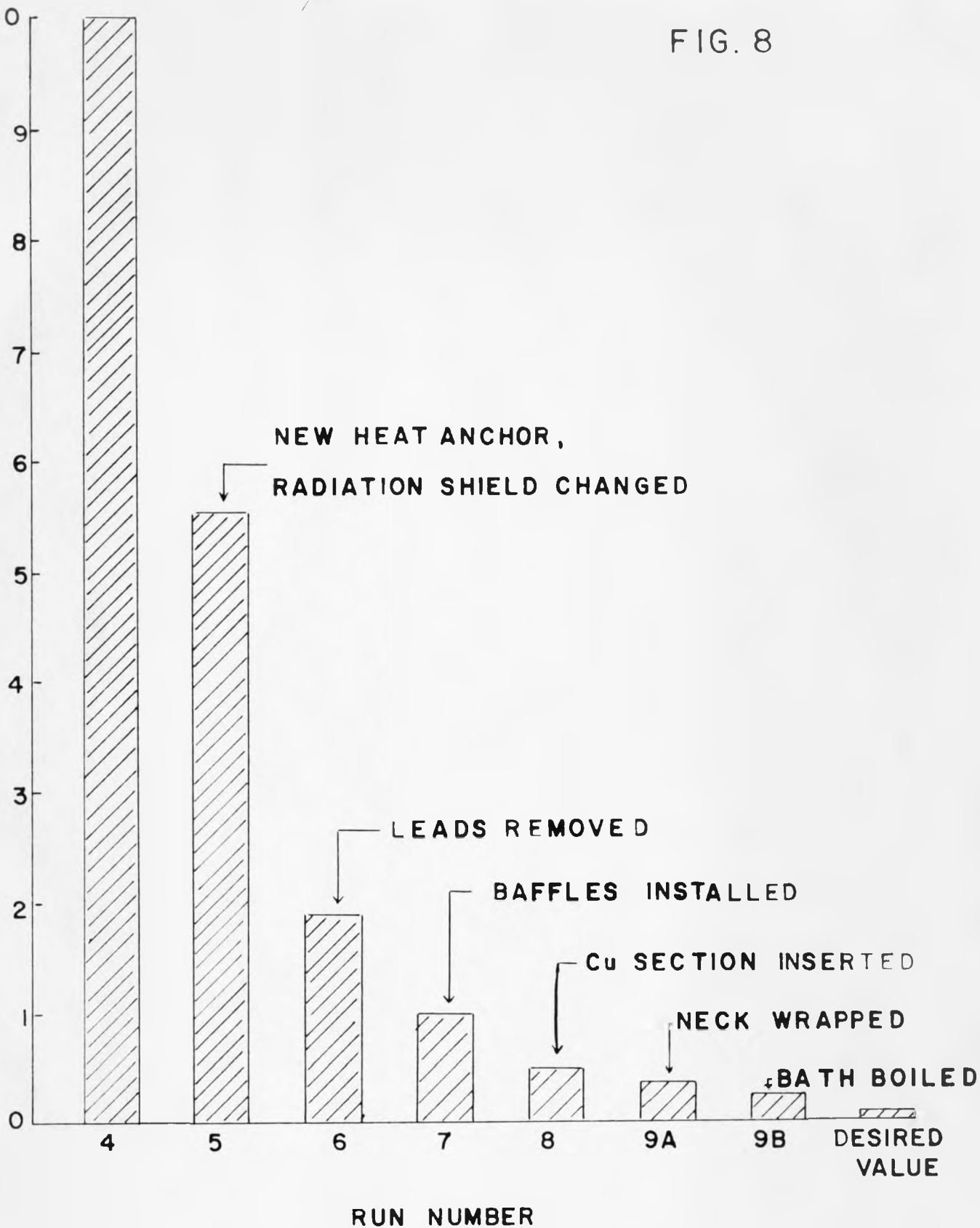
$$\frac{\Delta T_{\text{Gs}}}{\Delta T_{\text{Cu}}} = \frac{\text{Cond Cu}}{\text{Cond Gs}} \sim 3 \times 10^3$$

Movement of the bath level along the copper section should thus have no appreciable effect on the temperature distribution along the evaporation tube.

When the experiment was repeated with the copper section installed,

EVAPORATION RATE, cc/min

FIG. 8



RESULTS OF SUCCESSIVE EXPERIMENTS

(see fig. 8, run 8), the evaporation rate was reduced by a further factor of 2. The new value of approximately .55 cc of gas/minute was still five times greater than the maximum acceptable background rate.

Another run (no. 9) was then done to look for further radiation and level drop effects. To test the effect of radiation passing into the helium vapour through the glass neck of the inner dewar, the neck was first wrapped in aluminum foil and later unwrapped. It was noted that the background evaporation rate was considerably reduced while the neck was foil-covered (see fig. 8, run 9A). With the bath level on the narrow copper section, the main bath boil-off resistor was run at various power levels. It was found that by operating this resistor at sufficiently high power levels the background evaporation could be still further reduced. The lowest background recorded was 0.34 cc/min. for  $\Delta T = 0.21^{\circ}\text{K}$  at 25 milliwatts. It was still noted during this run that replenishing the air dewar had an effect on the background rate, although the effect was now much smaller.

The distinct radiation effect suggested that it might be wise to isolate the evaporation tube from the bath and so keep its temperature gradient fixed. A calculation showed that the tube could be effectively decoupled from the bath by means of a vacuum jacket from the baffles to the top of the dewar. During construction of this jacket the copper section of evaporation tube was replaced by german silver, as no variation in temperature gradient was now expected. Background evaporation rates determined with this arrangement were, however, higher than those of the two previous runs.

It was also noted that the bath level dropped more rapidly and

that the process of condensation required more helium from the bath than previously. Both of these latter effects were anticipated, the first because the vacuum jacket increased the heat leak to the bath, the second because cold vapour from the bath could no longer be used to cool gas entering the evaporation tube for condensation. Such an interpretation of these effects, however, leaves the increase in background rate unexplained. It may be that the readjustment of the temperature gradient along the evaporation tube, caused by isolating it from the cold helium gas above the main bath, has increased the amount of radiation reaching the baffles, and if these are not 100% efficient the radiative heat input to the calorimeter itself will be increased proportionately.

### 3. Conclusions:

It thus seems apparent that to achieve the low background evaporation rate desired ( $< 0.1$  cc/min. with leads disconnected,  $\leq 0.15$  cc/min. with leads reconnected), the evaporation tube must be freed from thermal disturbances and the calorimeter itself must be protected from stray heat influxes, e.g. by radiation, or by inadequate thermal anchoring of leads. To this end, a design for a new cryostat is submitted herewith (fig. 9).

The calorimeter is still surrounded by its vacuum jacket which in turn rests in the main helium bath. This bath, however, is now contained in a copper vessel enclosed by a vacuum jacket which is immersed in a liquid air bath. The evaporation tube has been given a vacuum jacket from the liquid air level to a level well above the top of the cryostat.

In this new cryostat the two points referred to in the first



paragraph of this section have received careful attention. Temperature fluctuation in the evaporation tube should be eliminated by:

- (1) fixed temperature points. The evaporation tube is soldered to a brass flange at the top of the copper He bath container ( $4^{\circ}\text{K}$ ), to a brass flange at liquid air temperature ( $80^{\circ}\text{K}$ ) and to the top of its vacuum jacket ( $300^{\circ}\text{K}$ ) which may be thermostatted if necessary.
- (2) the vacuum jacket between these points. The tube is in a vacuum between the air and helium baths and has its own vacuum jacket from the air bath to room temperature. This keeps the temperature gradient fixed.
- (3) immersion of the helium bath vacuum jacket in liquid air. This means that the only radiation striking the evaporation tube at its cold end comes from a surface at  $80^{\circ}\text{K}$  instead of room temperature.

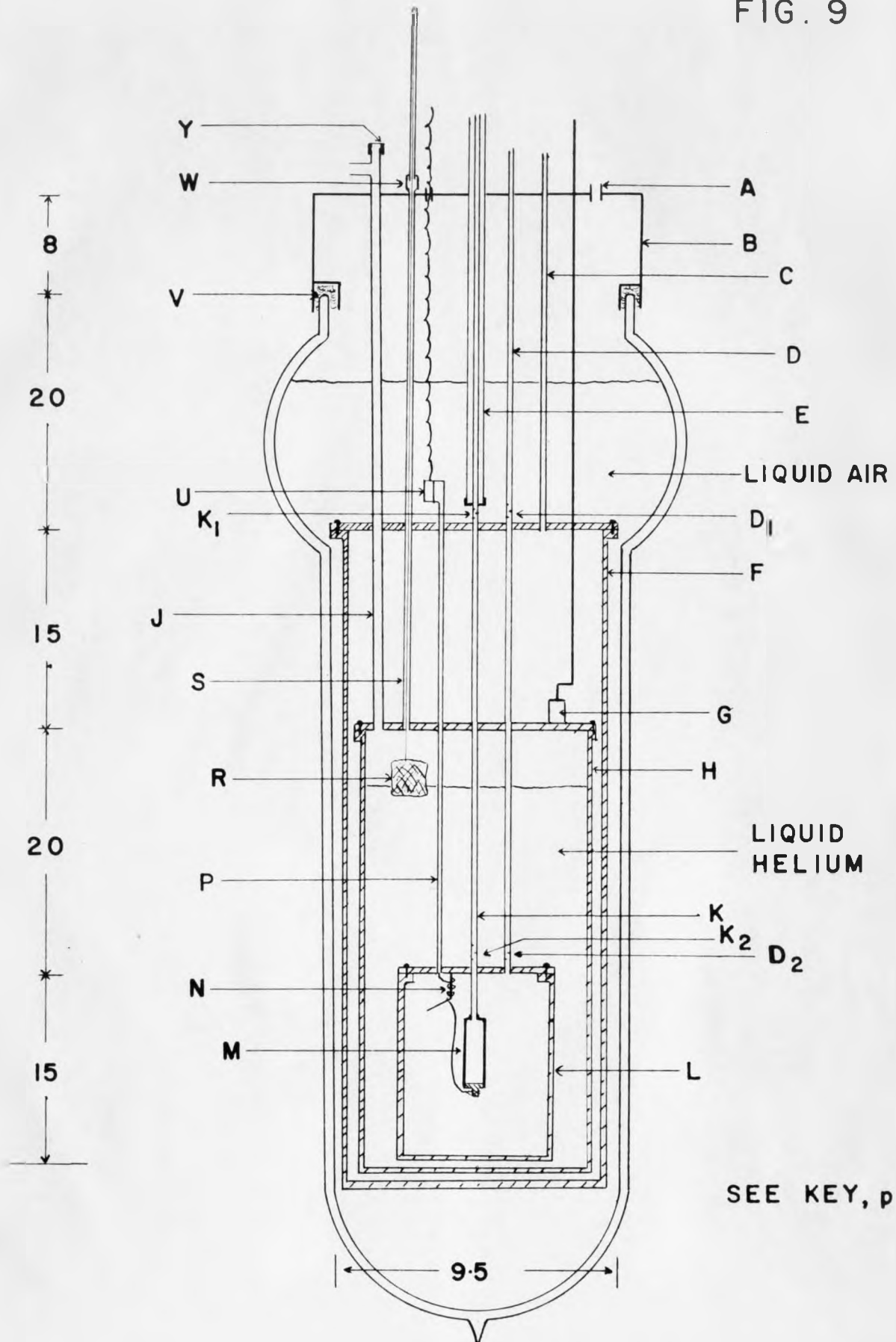
The calorimeter is protected from radiation by the installation of two sets of baffles (one at  $\sim 80^{\circ}\text{K}$ , the other at  $4^{\circ}\text{K}$ ) in both the evaporation tube and the calorimeter vacuum can pumping tube. The vacuum seal for the leads is now made at liquid air temperature, ensuring that they are at  $\sim 80^{\circ}\text{K}$  before being anchored again at  $4^{\circ}\text{K}$ .

A gas thermometer is included to allow the experimenter to follow the cool-down process; for convenience, a helium level indicator consisting of a styrofoam float with a long wire attached is also provided. The siphon inlet and pumping tube are combined as in the present apparatus.

A comparison of the cryostats depicted in figs. 1 and 9 suggests that the latter will be considerably more difficult to construct than was

the former. It is the contention of this thesis, however, that the additional labour of construction will be rewarded with experimental results of the required precision.

FIG. 9



SEE KEY, p 28

PROPOSED NEW CRYOSTAT

KEY TO FIG. 9.

- A - liquid air inlet
- B - copper spinning
- C - main vacuum can pumping tube,  $\frac{1}{4}$ " german silver
- D - calorimeter vacuum can pumping tube,  $\frac{1}{4}$ " german silver with baffles  $D_1, D_2$
- E - evaporation tube vacuum jacket,  $\frac{1}{2}$ " german silver
- F - main vacuum can
- G - He gas thermometer bulb, with copper capillary to pressure gauge
- H - liquid He container, copper
- K - evaporation tube,  $\frac{1}{4}$ " german silver with baffles  $K_1, K_2$
- L - calorimeter vacuum can
- M - calorimeter and heater,  $\frac{1}{2}$ " german silver tube and copper former, as at present
- N - thermal anchor and radiation shield for leads tube
- P - leads tube,  $\frac{1}{4}$ " german silver
- R - styrofoam float for He level indication
- S - guide tube for float wire
- J - pumping and siphon inlet tube,  $\frac{1}{2}$ " german silver
- U - vacuum seal for leads
- V - cotton batten filler inside flange of spinning
- W - metal-glass tube connection
- Y - cap in place for pumping

All dimensions are in centimetres and approximate.

APPENDIX

Derivation of  $L = L_a \left(1 - \frac{\rho_v}{\rho_l}\right)$

By definition the latent heat of vaporization is that amount of heat required to cause 1 mole of liquid to change to the vapour phase.

Suppose we have some liquid in the calorimeter and that the evaporation tube be filled with vapour. Let us vaporize X moles of liquid. We will not see X moles of vapour appear in the burette because some of the vapour will occupy space formerly occupied by liquid.

Let the heat input be Q

$$\text{Then } L_a = \frac{Q}{M_a} = \frac{Q}{V_a \rho_v}$$

where  $M_a$  and  $V_a$  are respectively the mass and volume of vapour expelled

$$\text{But } L = \frac{Q}{M} = \frac{Q}{V \rho_v}$$

where M and V are respectively the true mass and volume of vapour liberated.

But since we have vaporized X moles, we have (mass being constant)

$$M = V \rho_v = (V - V_a) \rho_l$$

$$\text{i.e. } V_a = \frac{1}{\rho_l} (\rho_l - \rho_v) V$$

$$\text{but } \frac{L}{L_a} = \frac{V_a}{V} = \left(1 - \frac{\rho_v}{\rho_l}\right)$$

$$\therefore L = L_a \left(1 - \frac{\rho_v}{\rho_l}\right)$$

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