Pertanika 8(1), 33 – 42 (1985)

Heat Inleak along the Neck of Liquid Nitrogen Containers

ABDUL HALIM SHAARI
Physics Department,
Faculty of Science and Environment Studies,
Universiti Pertanian Malaysia,
Serdang, Selangor, Malaysia.

Key words: Heat inleak; neck; liquid nitrogen containers.

ABSTRAK

Pengukuran profil suhu telah dibuat di sepanjang leher keluli nirkarat sebuah dewa kommersial 25 liter untuk penyimpanan nitrogen cecair. Pengukuran juga telah dibuat untuk profil suhu lapisan sempadan gas efluen. Kebocoran haba ke dalam bekas nitrogen cecair, terutamanya adalah disebabkan oleh pengkonduksian haba melalui dinding leher dan proses edaran semula wap. Kedua-kedua sumber ini menyumbangkan sebanyak 80% daripada jumlah kebocoran haba. Pengkonduksian haba melalui pepejal adalah kerana ukuran leher pendek dan dengan demikian nilai pendinginan dari gas efluen yang sejuk tidak digunakan dengan sepenuhnya. Proses edaran semula wap adalah disebabkan oleh fluks jisim balikan daripada gas panas ke dalam bekas cecair.

ABSTRACT

Measurements have been made of the temperature profile along the stainless steel neck of a 25 litre commercial dewar for the storage of liquid nitrogen. The temperature profile of the boundary layer of the effluent gas was also measured. The major heat inleak into the liquid nitrogen bath is due to the solid heat conduction down the neck wall and to the vapour recirculation process. These two sources contributed about 80% of the total heat inleak. The solid heat conduction was due to the fact that the neck was short and as such the refrigerative value of the cold effluent gas was not fully utilised. The vapour recirculation process is due to the reverse mass flux of the warmer gas into the bath.

INTRODUCTION

A minimum rate of heat transfers into the cryogenic liquid is the principal criterion for the design of a cryogenic vessel. The following causes of heat flow into the liquid should be considered (Hoare *et al.*, 1961). These are:

a. Heat inleak via insulating materials and residual gas in the vacuum space;

b. Conduction along the neck of the dewar;

c. Radiation through the neck aperture;

d. Conduction through supporting materials; if any, crossing the vacuum space;

e. Thermosyphon phenomena (convection) in the interior ullage of the dewar.

In the 25-litre commercial dewar, "multilayer insulations" are used in combination with a vacuum housing. The use of multilayer insulations, so called because of its alternating layers of highly reflective material and low thermal conductivity spacer, has very much reduced the total heat inleak into the cryogen (Scurlock and Saull, 1976).

However, the other main share of heat admitted to the cryogen in the dewar is through the neck. This heat conduction is called a heat short and is the subject of this paper (Shaari, 1977). In order to estimate the amount of heat short-circuited through the neck, measurements of temperature profiles along the neck wall and boil-off rates were made.

THEORETICAL MODEL

An analysis to calculate the dewar depth necessary to reduce the liquid boil-off rate to a given value can also be used to calculate the temperature profile in the dewar (Henry, 1951; Crooks, 1969). The model is illustrated in *Fig. 1.* It assumes that the inner spherical shell of the dewar may be regarded as an isothermal body at temperature T_o, the temperature at which nitrogen boils at the existing pressure. Heat exchange between the effluent gas and the neck wall is perfect. Convection, conduction and absorption of radiation are considered negligible in the gas phase of the neck column.

An amount of heat per unit time, $\dot{Q}$, flowing down the neck wall would pass a contour, where the temperature is T, into a region where the temperature is T-dT. As it does so, an amount of heat $d\dot{Q}$ is relinquished into the rising gas. The heat lost is equal to the heat required to warm the gas through the temperature interval dT. If the specific heat of the effluent gas is denoted by C_p and if m is the mass flow rate of the gas, then at any height of the wall at temperature T.

$$\dot{Q}(T) = \dot{Q}_o + \dot{m}\int_{T_o}^{T} C_p dT \qquad (1)$$

where $\dot{Q}_o$ is the power at the base of the neck, i.e. at $x = 0$.

Within the wall of the neck, the rate of heat transfer is given by a typical Fourier heat conduction equation,

$$K(T)\,A\,\frac{dT}{dx} = \dot{Q}_o + \dot{m}\int_{T_o}^{T} C_p dT \qquad (2)$$

where K(T) is the thermal conductivity of the dewar material and A is the cross-sectional area of the wall. The practical solution of the problem can be simplified by assuming that (Scott, 1959),

$$K(T) = K_o + a(T - T_o) \qquad (3)$$

where K_o is the thermal conductivity at the cold end of the neck and a is the average rate of increase of thermal conductivity of the wall material between 77K and 300K. Substituting equation (3) into (2) and assuming that the specific heat of gas is constant at constant pressure, then the position coordinate is given by

$$x(T) = A\int_{T_o}^{T} \frac{K_o + a(T - T_o)}{\dot{Q}_o + \dot{m}C_p(T - T_o)}\, dT \qquad (4)$$

Thus at $x = L$, where L is the neck length and $T = T_1$ we have,

$$\frac{L}{A} = \left(\frac{1}{\dot{m}C_p}\right)\left[a(T_1 - T_o) + \left(K_o - \frac{a\dot{Q}_o}{\dot{m}C_p}\right) \ln\left[1 + \frac{\dot{m}C_p(T_1 - T_o)}{\dot{Q}_o}\right]\right] \qquad (5)$$

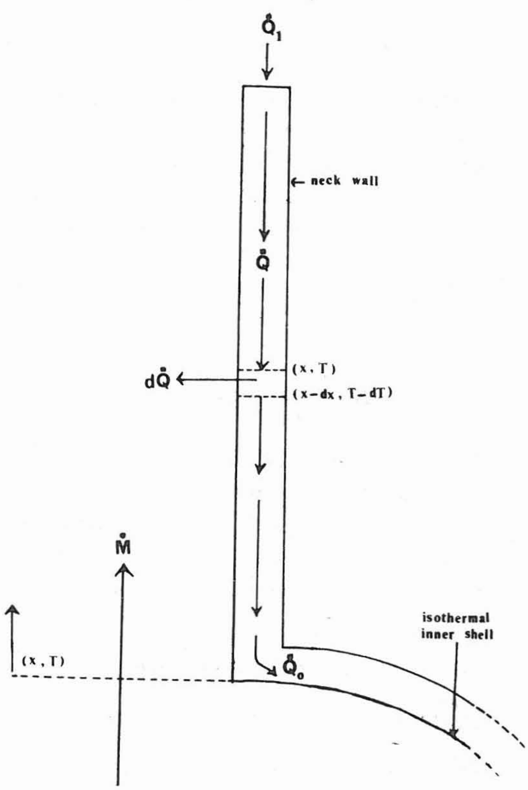


Fig. 1. Model for analysing heat inleak along the neck.

Since the thermal conductivities of most alloys used for the neck tube are rather complex functions of temperature, equation (5) is valid for a smaller interval of length in which the condition of linearity in K(T) holds.

However, if the heat exchange between the effluent gas and the wall of the neck is zero, then it can be shown that,

$$\frac{L}{A} = \left(\frac{1}{\dot{Q}_o}\right)\left[(K_o - aT_o)(T_1 - T_o) + \frac{1}{2}a\,(T_1^2 - T_o^2)\right] \qquad (6)$$

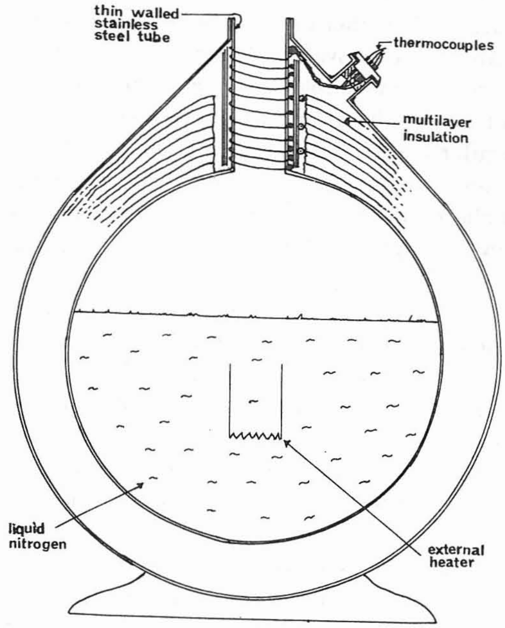
Both equations (5) and (6) were obtained at two extreme conditions. However, in practice, perfect heat transfer between the wall and the gas is never achieved. But one should not underestimate the cooling effect of the gas evolved. As such, the heat current reaching the liquid bath will have a value somewhere in between the two values calculated for the two extreme conditions.

EXPERIMENTAL

The apparatus used is shown schematically in *Fig. 2*. The dewar used was a 25 litre commercial dewar whose outer vessel is made from mild steel while the inner vessel is a 25-litre copper vessel, with the snubber tube at the neck removed so as to enable the thermocouples to be fixed along the outer diameter of the neck (length 13 cm; thickness 0.046 cm).

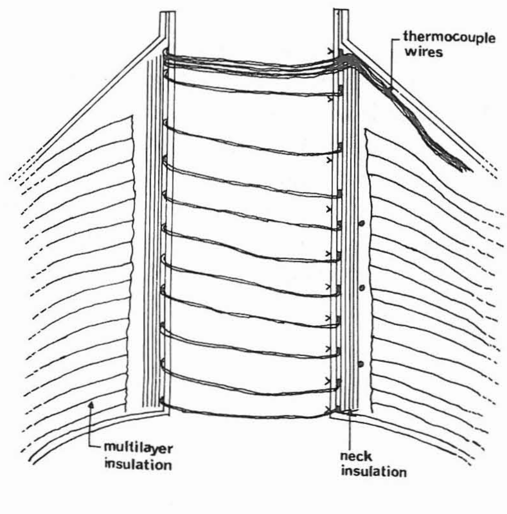
Eleven thermocouples made from manganin-constantan wires were stationed at various points along the wall of the neck, facing the vacuum space as shown in *Fig. 3*. To ensure good thermal contact with the wall, the thermocouples junctions were all covered with a piece of aluminium foil and glued to the wall. All the thermocouple wires were each wound around the neck several times and anchored at the top of the neck.

In order to obtain an estimate of the radiation heat flux entering the neck through the vacuum space, the wall of the neck was wrapped with six layers of aluminium foil, 12.0 m in



> Thermocouples along vapour boundary layer.
● Thermocouples along neck wall.
® Thermocouples around insulation.

Fig. 2. Schematic drawing of temperature



● Thermocouples along neck wall.
® Thermocouples around neck insulation.

Fig. 3. Installation of thermocouples along neck wall.

thickness, as a reflective medium. Each layer was separated by a ten per cent carbon loaded glass fibre of low thermal conductivity as a spacer material. The overall thickness was 0.102 cm. Three thermocouples were then attached to the outside of the insulation as shown in *Fig. 3*. Similarly, each thermocouple was wound several times around the insulation and was finally anchored at the top of the neck as the previous eleven thermocouples.

All the fourteen thermocouples were then glued together using varnish so as to keep the electrical insulation intact and thus avoiding short circuiting the wires during bake-out process. These wires were then taken out of the vacuum space through a lead connector fixed at the outer vessel of the dewar, as shown in *Fig. 2*.

In order to measure the temperature profiles of the boundary layer of the vapour in the neck column, ten thermocouples were stationed at various points along the wall of the neck facing the rising vapour with the junctions jutting out perpendicularly, about 1.5 mm into the vapour column (See *Fig. 3*).

Having installed all the thermouples, the test dewar was assembled under commercial conditions as shown in Table 1.

During assembly of the dewar, the fine thermocouple wires were handled carefully. Before welding the inner vessel to the outer vessel, the thermocouples from the neck were all threaded out through leads in the glass-to-metal seal. The passage, through which the wires were drawn out, was sealed from inside with araldite. The test dewar was then leak tested and baked at 120°C.

The temperature profiles were observed at different liquid levels and with different mass flowrates. In order to check the dependence of temperature profiles on the evaporation rates of the gas, an electrical heater assembly (which somewhat increased the thermal conduction down the neck) was installed, and measurements of evaporation rate as a function of power dissipation were made. The temperatures were also measured when the exit of the neck was plugged with a commercial plug.

RESULTS AND DISCUSSION

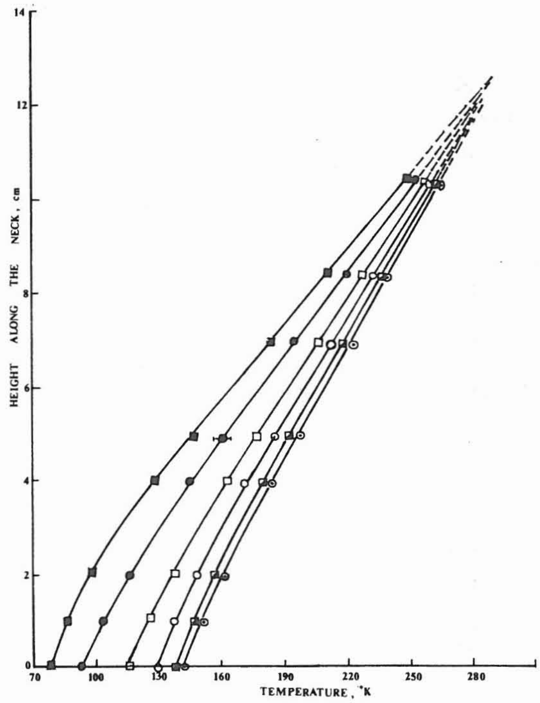
Interpretation of Temperature Profiles

A series of temperature profiles were measured. Typical results are as shown in *Fig. 4*. The graphs show that the temperature at the base of the neck varies with the depth of the liquid nitrogen in the dewar. Hence, it can be deduced that the inner vessel is not in isothermal equilibrium with the liquid bath. In Run 1 and 2 *(Fig 4)*, the mass flow rate was higher than the normal boil-off rate. This could be due to the fact that the funnelling of radiation down the neck tube affects the boil-off of the liquid. However, as the depth of the liquid decreases, the funnelling of radiation down the neck was minimised by the cold effluent gas and much of it was absorbed before reaching the liquid bath. Thus, the boil-off was due to the heat inleak; via neck wall; through multilayer insulation and due to the thermosyphon process.

In theory, the heat exchange between the wall and the vapour is assumed perfect. However, in practice, the assumption fails because the wall and the vapour are not in thermal equilibrium. This could be seen in *Fig. 5*, where the

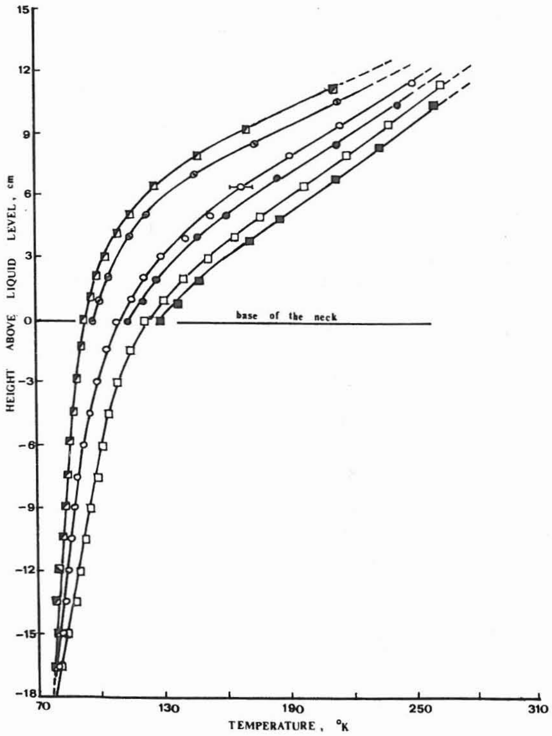
TABLE 1
Details of test Dewar

Number of layer of insulation	Insulation thickness (mm)	Spacer material	Wrapping technique	Other particulars
30	26.6	Glass-fibre paper (GFG 3)	Swissroll	No Snubber tube



■ Run 1 — Level of LIN ~ 12.5 cm below exit.
● Run 2 — Level of LIN ~ 15 cm below exit.
□ Run 3 — Level of LIN ~ 23.5 cm below exit.
○ Run 4 — Level of LIN ~ 34.5 cm below exit.
◪ Run 5 — Level of LIN ~ 41.5 cm below exit.
⊙ Run 6 — Level of LIN ~ 43.5 cm below exit.

Fig. 4. Temperature profiles along the neck (with constant mass flow of effluent gas).

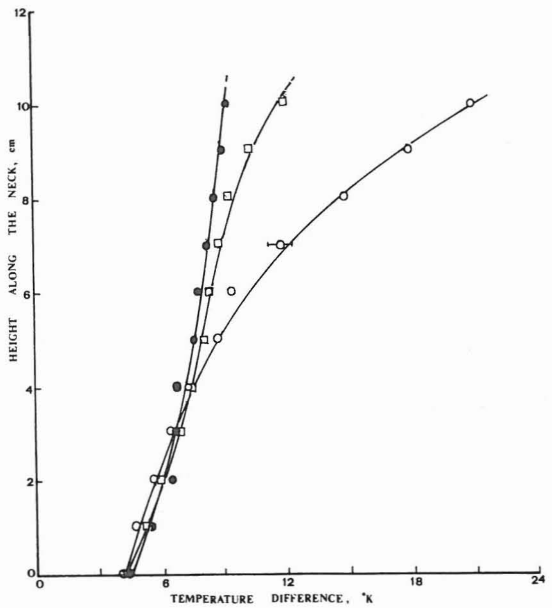


■ Run 1 — Wall temperature. No heat input.
□ Run 2 — Vapour temperature down to liquid level. No heat input.
● Run 3 — Wall temperature. Heat input 1.2 W.
○ Run 4 — Vapour temperature down to liquid level. Heat input 1.2 W.
⊙ Run 5 — Wall temperature. Heat input 3.6 W.
◪ Run 6 — Vapour temperature down to liquid level. Heat input 3.6 W.
(Level of LIN ~ 32 cm below exit.)

Fig. 5. Temperature profiles of the wall and the vapour.

temperatures of the wall are much higher than those of the gas. At a lower flowrate, the difference in the temperatures is fairly constant. At a higher flow rate, the temperature difference between the wall and the vapour increases, as illustrated in *Fig. 6*. This shows that a perfect heat exchange is likely to be approached at low flow rates of a gas. As seen in *Fig. 5*, measurements of the vapour temperature were extended down into the liquid level. It is obvious that as the flowrate increases, a much lower vertical temperature gradient is observed at the region close to the liquid surface.

Measurements of the temperature profiles across the neck are as shown in *Fig. 7*. One obvious result is that there is a marked temperature difference between the wall and the effluent gas and this temperature difference increases towards the neck aperture. The temperature profile is fairly stratified except towards the upper part of the neck where the warm gas mixes before exiting towards the aperture. At the lower part of the neck column, the temperature of the boundary layer is 'lower than that of the bulk temperature of the gas. However, the boundary layer suffers progressive thinning towards the



● No heat input.
□ Heat input 1.92 W.
® Heat input 3.6 W.

Fig. 6. Temperature difference between the wall and the vapour versus height of the wall.

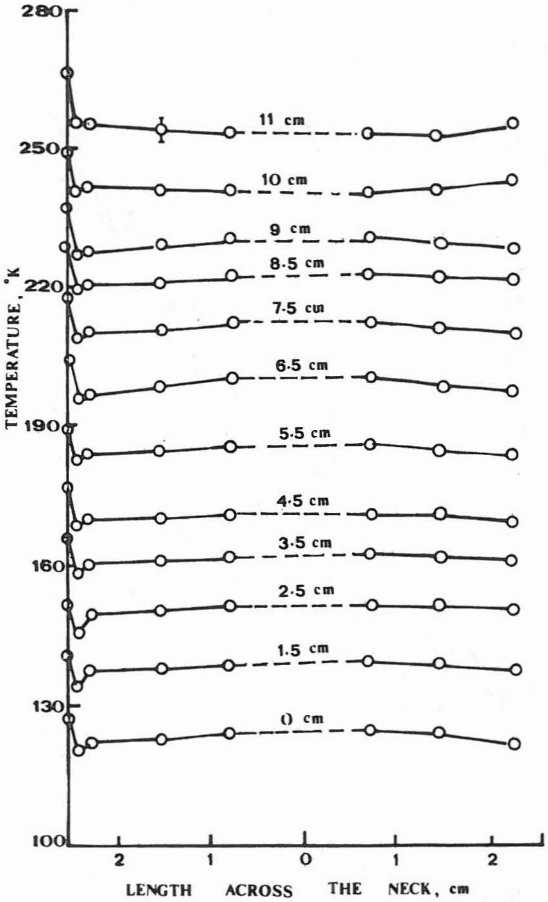
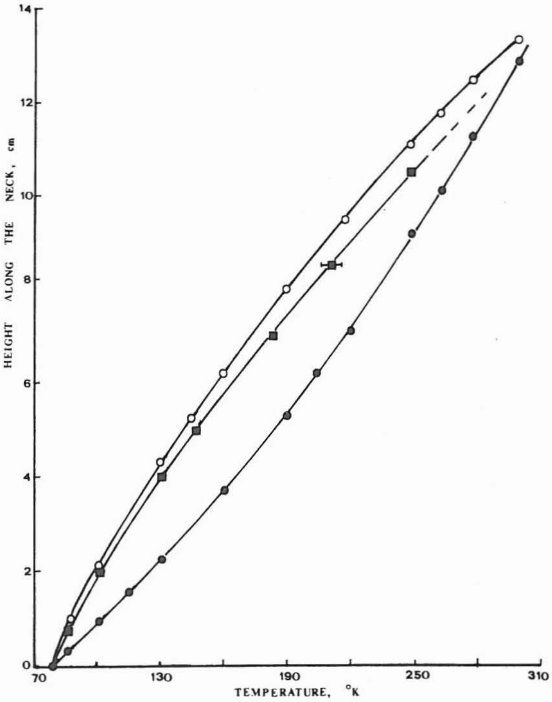


Fig. 7. Temperature profile across the neck (Level of LIN ~ 32 cm. Below exit).

upper part of the neck column and as such the temperature of the boundary layer is fairly equal to the bulk temperature of the gas. This is due to the rapid thermosyphon circulation as was observed by the cryogenic group at Southampton University, where a thorough programme on complex flows in cryogenic vapour column is being carried out.

Calculation of Heat Inleak

Scott (1959) has shown that, assuming 'perfect' heat exchange between cold vapour and neck wall, it is possible to calculate the minimum heat in inleak $\dot{Q}_0$, through the neck wall by using equation (5). Applying this equation to Run 1 in *Fig. 4,* a value of 0.6 watt is obtained for $\dot{Q}_0$. Results of Run were used here because the base of the neck was at the liquid bath temperature, while the other end was at ambient temperature. From the slope of temperature profile of the wall, $\dot{Q}_0$ was found to be 0.85 watt. The difference in the two values clearly implies that the heat exchange between the wall and the neck is not perfect. However, if the cooling effect of the cold vapour is neglected then by using equation (6), a maximum value of heat inleak via neck wall into liquid nitrogen was found to be 1.6 watts. Thus, the value of heat inleak via the wall of the neck, for a practical dewar falls



□ experiment
® calculated from theory for perfect heat exchange.
● calculated from theory for zero heat exchange.

Fig. 8. Comparison of temperature profiles between theory and experimental.

between the two extreme values. This is illustrated in *Fig. 8*. The detailed calculation of heat fluxes is summarised in Table 2. It was found that $\dot{Q}_o$ decreased as the external heater power increased, i.e. as the mass flowrate of the gas increased, Obviously, as the heat conducted along the inner wall, much of it was absorbed by the rising gas.

Measurements of the temperature gradient along the outer layer of the insulation of the wall of the neck enabled us to calculate the radiation heat flux along the neck. The results are shown in Table 3. It was found that an average value of 0.1 watt leaked via the wall of the neck by radiation.

Heat flux due to the vapour recirculation, $\dot{Q}_v$, in the vapour space was found to be another major source of heat inleak. At this stage it could not be measured directly. However, the radiation heat flux into the vessel, via multilayer insulations could be calculated using the equation (Vance, 1962),

$$\dot{Q}_1 = \frac{\bar{K}A(T_1 - T_2)}{t} \qquad (7)$$

where $\bar{K}$ = effective mean thermal conductivity of the insulation.
= $0.5\ \mu W\ cm^{-1}K^{-1}$,

A = area of the inner vessel covered by the multilayer insulation
= $4.135 \times 10^3 cm^2$

t = thickness of multilayer insulation
= 2.66 cm.

T_1, T_2 = temperature at two extremes,

Thus, the heat flux due to vapour recirculation is estimated as,

$$\dot{Q}_v = \dot{Q}_T - \dot{Q}_E - \dot{Q}_0 - \dot{Q}_R - \dot{Q}_1 \qquad (8)$$

where,

$\dot{Q}_T$ = total heat flux

$\dot{Q}_E$ = heater power

$\dot{Q}_0$ = solid heat conduction to the base of the neck.

$\dot{Q}_R$ = radiative heat flux along the neck.

$\dot{Q}_1$ = heat inleak via multilayer insulation.

In column 7 of Table 2, the figures show the estimated values of the vapour recirculation heat flux. To a fair degree, it is found that it is a function of the boil off rate of the gas. This agrees with the flow visualisation work at Southampton, which indicates that the recirculation of vapour in the neck is a function of boil-off rate and temperature gradient and could lead to a heat flux of the order of 0.1 to 1 watt.

CONCLUSION

The data presented here give a clear picture of typical temperature profiles in a 25-litre com-

TABLE 2

Heat fluxes via dewar neck and other sources

Depth or level of the below neck aperture (cm)	Total heat flux in to liquid nitrogen Q_T (watt)	External heat power Q_E (watt)	Net Heat Flux $Q_T - Q_E$ (watt)	Heat flux into Lin via neck conduction Q_o (watt)	Heat flux into ullage space	Heat flux due to vapour recirculation $Q_V = Q_T - Q_E - Q_R - Q_I$ (watt) (5 cases)	Heat flux due to conduction & recirculation (5 cases)
12.5	1.783	0	1.783	0.843	—	0.64	1.483
15	1.783	0	1.783	0.854	—	0.629	1.483
23.5	1.626	0	1.626	—	1.117	—	—
32	1.626	0	1.626	0.821	—	0.505	1.326
34.5	1.626	0	1.626	—	1.075	—	—
38	1.626	0	1.626	—	1.222	—	—
41.5	1.626	0	1.626	—	1.084	—	—
43.5	1.626	0	1.626	—	1.113	—	—
32	1.942	0.3	1.642	—	1.141	—	—
32	2.89	1.2	1.609	0.591	—	0.718	1.309
32	5.119	3.6	1.519	0.223	—	0.996	1.219
34	3.468	1.92	1.548	—	1.13	—	—
38	2.543	0.95	1.593	—	1.165	—	—
38	5.274	3.79	1.474	—	1.014	—	—

Q_R = Heat flux by radiation in the neck column (See Table 3 = 0.1 watt)

Q_I = Heat flux through multilayer insulation (= 0.2 watt)

TABLE 3

Heat fluxes by radiation along neck

Level of liquid nitrogen in the dewar (cm)	Temperature of outer layer of insulation around dewar neck, measured at positions above the neck base at ... (°K)			Mass Flowrates of the nitrogen gas $m(gs^{-1}) \times 10^{-3}$	Heat flux by radiation along the neck (W)
	1.5 cm	4.7 cm	5.6 cm		
12.5	206.2	251.0	262.4	8.948	0.113
15.0	208.8	252.8	264.8	8.948	0.1
23.5	211.6	253.9	265.3	8.159	0.085
32.0	214.8	255.9	267.0	8.159	0.078
34.5	215.5	256.6	267.6	8.159	0.078
38.0	219.6	258.1	269.0	8.159	0.075
41.5	221.7	258.5	269.2	8.159	0.073
43.5	223.5	259.7	270.3	8.159	0.071
32.0	217.6	257.3	267.8	9.746	0.085
32.0	213.5	253.7	264.4	14.097	0.102
32.0	208.8	248.8	260.0	25.69	0.129
34.0	210.6	250.5	261.6	17.404	0.109
38.0	212.2	251.7	262.5	12.717	0.097
38.0	207.9	247.9	259.2	26.468	0.121

mercial dewer. Analysis of the heat fluxes into the dewar shows that;

i.	Conduction along the neck wall	0.85 W
ii.	Multilayer insulation	0.18 W
iii.	Radiation into the neck	0.10 W
iv.	Vapour recirculation process	0.64 W

The total heat inleak is 1.77 W.

In analysing the temperature profile along the neck, the model used simply breaks down due to the fact that there is no thermal equilibrium between the evolved gas and the wall of the neck. However, it would probably fit a long necked dewar such as a helium storage vessel.

Finally it is suggested that the neck wall geometry of the 25 litre dewar be modified so as to achieve lower boil-off rates by reducing solid conduction and vapour recirculation.

ACKNOWLEDGEMENT

This study is part of a project supported by the University of Southampton and the British Oxygen Company, Cryoproducts Division, Crawley, England. The author wishes to thank Associate Professor Dr. Mohd. Yusof Sulaiman, the Deputy Dean of the Faculty of Science and Environmental Studies, Universiti Pertanian Malaysia, for reading the paper.

REFERENCES

CROOKS, M.J. (1969): Temperature Profiles in Experimental Dewars. *Cryogenics.* **9:** 32 – 35.

HENRY, W.E. (1951): A Finite Difference Treatment of a Helium Cryostat design problem. *J. Appl. Phys.* **22:** 1439.

HOARE, F.E., JACKSON, L.C. and KURTI, N. (1961): Experimental Cryophysis. Butterworth, London.

SCOTT, R.B. (1959): Cryogenic Engineering. D. Van Nostrand Company Inc. Princeton. J. Jersey.

SCURLOCK, R.G. and SAULL, B. (1976): Development of Multilayer Insulation with Thermal Conductivities below 0.1 W cm $^{-1}$K $^{-1}$. *Cryogenics.* **16:** 303 – 311.

SHARI, A. HALIM. (1977): Temperature Profiles along the Neck of Liquid Nitrogen Containers. Unpublished M. Sc. Project Dissertation. Southampton University.

WANCE, R.W. and DUKE, W.M. (1962): Applied Cryogenic Engineering. John Wiley and Sons. New York, London.

(Received 9 July, 1984)