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BETWEEN 77 K AND 4.2 K

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TECHNIQUES FOR REDUCING RADIATION HEAT TRANSFER BETWEEN 77 K AND 4.2 K*

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INTRODUCTION

The present day applied superconductivity is a liquid helium based technology. The efficiency of a cryogenic or superconducting device is determined by its rate of consumption of the cryogen. Liquid helium has an extremely small heat of vaporization; thus, its storage almost always requires a state-of-art insulating method. Radiation heat leak becomes significantly more important as larger superconducting devices are built, such as huge high-energy physics analysis magnets, fusion reactor systems and energy storage facilities. For large superconducting magnets, [1-3] it is common practice to surround the liquid helium vessel with a nitrogen shield (at 77K) and wrap MLI around both the liquid helium vessel and the nitrogen shield in an attempt to further reduce the heat leaks (Fig. 1a). Multilayer insulation is inexpensive and generally effective; yet, it is a rather difficult material to apply because its performance depends on a few hard to control parameters such as, the layer density, the compressive loading, and the lateral heat transfer effect. The effective insulation capability obtained in practice is at least a factor of two worse than carefully measured laboratory values (or those claimed by manufacturers). Careless and/or inexperienced application can easily generate heat leak values a few times higher than the predicted value, especially when dealing with peculiarly shaped cryostats.

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Extensive study and test programs^[4-6] had been carried out in an attempt to better understand the thermal performance of MLI but a lack of experimental data still exists for temperatures below 60K. In fact, the only experimental heat transfer data that can be found^[7] by the author is 14.3 mW/m^2 (1.33 mW/ft^2) for a 60 layers/inch Linde system (Al foil + glass paper). A commonly used design parameter taking penetrations and other imperfections into account is 43.0 mW/m^2 (4.0 mW/ft^2) for NRC-2 superinsulation,* which is the cheapest MLI and the easiest to apply. In this paper, the 77K $\rightarrow$ 4.2K performance (in mW/m^2) of NRC-2 as a function of the number of layers is presented. Results indicate that a much smaller number of layers is required to obtain optimum performance, compared to the large number (40 layers/inch for minimum K_p or 120 layers/inch for minimum K) between 300K and 77K. Pushing this concept of complete elimination of solid conduction to the limit would lead us directly to the classical calculation of Scott,^[8] who indicated a heat transfer rate of the order of 21.5 mW/m^2 (2 mW/ft^2) between surfaces at 77K and 4.2K is achievable, provided that both surfaces have sufficiently low emissivities ($\epsilon = 0.01-0.03$). Recent work of Chaussy, et al.,^[9] showed that a value of 18.7 mW/m^2 (1.74 mW/ft^2) could be obtained by covering both the 77K and 4.2K surfaces with a commercial aluminum adhesive tape Eccoshield PST.[†] If we substitute the values for the emissivities confirmed by Chaussy for aluminum ($\epsilon_{77} = 0.03$ and $\epsilon_{4.2} = 0.011$) into the parallel plate formula (for diffuse reflection) in Scott,^[8]

$$\frac{\dot{Q}}{A} = \frac{\sigma(T_2^4 - T_1^4)}{\frac{1}{\epsilon_1} + \left(\frac{1}{\epsilon_2} - 1\right)}, \quad (1)$$

*Manufactured by King Seeley Company, Winchester, Massachusetts.

†Manufactured by Emerson Cuming, Inc., Canton, Massachusetts.

we can actually obtain a heat transfer rate of 16.5 mW/m^2 (1.53 mW/ft^2).

This paper describes a test program set up to further verify low radiation heat loss via this "reflective coating" method and to study the problem of degradation and contamination associated with such low emittance surfaces. We have measured heat transfer ($77\text{K} \rightarrow 4.2\text{K}$) for cleaned copper to cleaned copper, Eccoshield Al tape to Eccoshield Al tape, 3M (Industrial tape #425) Al tape to 3M Al tape. Presented here are also other ideas and experiments leading to a practical scheme for use in the Chicago Cyclotron Magnet (CCM), a large superconducting split solenoid, currently under construction at Fermilab.

RADIATION HEAT TRANSFER AND EMISSIVITIES

Success in reducing radiation heat transfer depends on achieving surfaces of low emissivities. Metals approximate gray bodies rather well in the infra-red region, consequently, we can take the spectral emissivity values to be the same as the total emissivities without any appreciable error. The knowledge of low temperature emissivity and absorptivity values is limited.^[10-12] Note that the emissivity value for a given material is also a function of the wavelength (Fig. 2) of the incoming thermal radiation (in other words, the temperature of the heat source). The emissivity of a practical surface may be quite different from the value predicted for the given material under ideal conditions since it is a strong function of chemical purity and surface roughness, contamination, and method of preparation. Engineering testing of practical reflective surfaces is therefore justified. Good electrical conductors as silver, gold, copper and aluminum have low infra-red absorptivity. Gold is too expensive for practical use, while silver and

copper tend to tarnish easily in air. Aluminum, with its natural protective layer of aluminum oxide (transparent to infra-red radiation of temperature $< 100\text{K}$), is often used. Electropolished copper protected by a transparent benzotriazol layer, a few molecular layers thick, is also a good choice because the nitrogen shields in superconducting devices are typically made of copper whose high thermal conductivity helps to maintain a uniform temperature.

The "floating shield" concept is to insert a second reflective shield between the helium vessel surface and the nitrogen shield. (Fig. 1b). Theoretically, it can reduce the heat leak by a factor of 2. In practice, by attaching this secondary shield through high contact resistance connections, one can reduce the heat leak by 35-40%. Radiation heat load from 77K to 4.2K of less than 10 mW/m^2 is achievable using this method. If one selects to cool this secondary shield with excess helium boil-off, one can easily drive the temperature of the shield to $\sim 50\text{K}$ in which case the heat load to 4.2K would be $\sim 5 \text{ mW/m}^2$.

It has been suggested that a superconductor be used as a reflector. The emissivity should go to zero as the electrical resistivity vanishes. The Bardeen, Cooper, and Schrieffer (BCS) theory^[13] suggests that for a material in the superconducting state there is an energy gap for electron states such that a threshold excitation is required before energy can be absorbed. This relationship takes the form of

$$E_g = h\nu = 3.5kT_C \quad . \quad (2)$$

In other words, a superconductor of a critical temperature T_C would not absorb thermal radiation of the wavelength $\lambda > \frac{hc}{3.5kT_C}$. The average wavelength of the thermal radiation from a surface at a temperature T is governed

by the Wien Displacement Law

$$(\lambda T)_{\text{AVG}} = 4107 \mu\text{m} \cdot \text{K} \quad (3)$$

Simple arithmetic shows that Nb_3Sn ($T_C = 18.3 \text{ K}$, $H_C = 1.85 \times 10^4 \text{ KA/m}$), a superconductor considered having a rather high T_C by present day standards, can only be a perfect reflector for thermal radiation from a surface of temperature $< 15\text{K}$. Hence, a superconductor is of limited value as an infrared reflector for most helium systems.

TEST APPARATUS

A drawing of the test set-up is shown in Fig. 3. The 4.2K surface A is a flat copper plate completely surrounded by a rectangular copper box B at 77K. The sample to be tested is either attached (e.g., MLI), taped (e.g., Al tape) or coated onto both A and B. A capped-off 12.7 mm I.D. copper tube soldered to A is connected to the inner helium vessel C through a coupling. Any heat absorbed by the 4.2K fin A manifest itself as liquid helium boil-off from C because all other sources of heat are intercepted by the outer helium vessel D and the radiation baffles which almost enclose the inner helium vessel. The outermost concentric liquid nitrogen vessel E shields the outer helium vessel D from room temperature. The rectangular copper box B which constitutes the warm temperature boundary is cooled by liquid nitrogen from this outermost vessel E and is insulated with MLI from the outside. The whole apparatus is placed inside a vacuum vessel. When the experiment is running, the vacuum is at least 2×10^{-7} torr. The surface area of the 4.2K fin is approximately 2.26 m^2 (24.375 ft^2). The total heat absorbed by the 4.2K fin is divided by this area to give the experimental heat transfer rate per unit area between the 77K and 4.2K surfaces. Large storage volumes are

provided for both liquid helium and liquid nitrogen so as to facilitate prolonged testing (15-20 days), which is essential for degradation and contamination studies. The pressure in the liquid nitrogen vessel can be reduced to produce a lower warm boundary temperature.

EXPERIMENTAL TECHNIQUE

Instrumentation

The temperature of B is monitored using several copper-constantan thermocouples and that of A with calibrated carbon resistors. Variation of the temperature across A and B are both calculated and measured to be negligible.

The boil-off rate of the liquid helium in C is measured with a calibrated wet test meter (accuracy $\pm 0.2\%$). An electrical signal is sent to a running strip chart recorder upon completion of each revolution of the meter indicator such that continuous (and therefore average) readings can be taken. The temperature of the evolving gas is read directly on the wet-meter and liquid helium boil-off rate at S.T.P. (273.16K and 760 mm Hg) can then be calculated. It is important to note that the ratio of the saturated liquid density to the helium vapor density at 4.2K is only 7.5:1 at 1 atm., therefore, the actual helium evaporation rate should be 1.154 times the boil-off rate measured with the wet-meter.

A typical experimental run produces results similar to that shown in Fig. 5. The loss rate measurement is influenced by the variation of atmospheric pressure, which is therefore monitored continuously. A correction^[14] for the rate of vaporization resulting from small atmospheric pressure changes can be approximated by the general formula

$$\frac{dm_t}{dt} = I V \frac{dp}{dt} , \quad (4)$$

and in the case of liquid helium,

$$ER = 7.9 \times 10^{-3} V \left(\frac{dp}{dt} \right) . \quad (4a)$$

This explains the ± 5 -10% variation in the boil-off rate data. For this reason, each run is generally conducted over a period of a week or more to average out the affect of pressure variations and to insure the achievement of equilibrium conditions.

Calibration

The only major heat source into C is heat conduction down the vent tube F. It was calculated to be 8.3 mW using the method described in Scott.^[16] Background runs of the apparatus without both A and B were performed. In the beginning, we encountered thermal acoustic oscillation (TAO) and a background heat leak value of 5 times higher than calculated was observed. The TAO was removed by filling the vent tube with cotton plugs which broke the gas flow into small flow channels.^[17] The background run then yielded a result of 10.7 mW. The literature on TAO^[18-20] revealed that our system was nearly stable against oscillation and at the expected boil-off rates the background heat leak down the vent tube would only be ~ 0.3 mW. A sample run with the Eccoshield aluminum tape on both the 4.2K and 77K surface gives a heat transfer rate per unit area of 1.40 ± 0.05 mW/ft², which compares well with value obtained by Chaussy.^[9] Six months later, after six other experimental runs, this configuration was run again. A result of 1.38 ± 0.05 mW/ft² was obtained showing good reproducibility. We estimate that the over-all accuracy of our measurements to be $\pm 10\%$.

RESULTS AND DISCUSSION

A series of six runs were made using NRC-2 on a 4.2K surface painted with 3M black Velvet paint^[15] to simulate a dirty stainless steel helium vessel. Eccoshield tape was applied to the 77K surface. The results are shown in Fig. 6.

Other data is given in Table I. By comparing the results of Run #1, 3 and 4, it can be seen that if in the NRC-2 series, one had used bare ETP copper at 77K, the heat flux would have been higher and vice versa for the 3M Al tape.

The measured value from Run #1 (15.1 mW/m^2) is well approximated by Eq. (1), assuming $\epsilon_{77} = 0.03$ and $\epsilon_{4.2} = 0.011$ (16.5 mW/m^2). The geometry of our apparatus, a flat plate inside a rectangular box, may not be adequately represented by Eq. (1). A detailed analysis using configuration factors gives a heat transfer rate per unit area of 17.2 mW/m^2 , using the emissivities above, which indicates that geometrical effects are 4-5%.

Residual resistivity ratio (RRR) measurements of a few samples were made and are included in Table II. This gives a relative indication of how good the sample is as an infra-red reflector because the emissivity of a given material is directly and linearly dependent on its electrical resistivity.

Runs No. 5, 6, and 7 represent an attempt to study the concept of the floating shield and other simple possible schemes to be used in a large superconducting magnet system. The high value of the result from Run No. 5 is attributed to the transmission of heat flux across the single, thin layer of aluminized mylar and possible outgassing of epoxy causing poor vacuum condition between the surfaces. The result of Run #6 was higher than expected and can be explained by a d.c. size effect^[21] in the thin film causing a high value of emissivity. The RRR data in Table II also shows this. The result of Run #7 could be improved by using electropolished aluminum

for the floating shield.

CONCLUSION AND RECOMMENDATIONS

1. The 40 layer/inch quoted by most people for NRC-2 MLI between 77K and 4.2K surfaces is not necessarily true. In Fig. 6, it is shown that there is a broad minimum. For all practical purposes, wrapping the liquid helium surface with 10-15 layers is more effective than filling the gap between 77K and 4.2K with a large number of NRC-2 MLI layers. This smaller number of layers may completely eliminate solid conduction since the outermost MLI layer should not touch the 77K surface. Fewer layers of MLI also represents a reduction in manpower required for the application.
2. The Eccoshield aluminum tape gives poorer performance than using less than 4 layers and more than 74 layers of NRC-2 MLI in a given gap width of 5.08 cm (Fig. 6 and Table I). It is probably comparable to using 40 layers of NRC-2 MLI in a gap width of 2.54 cm. No degradation was observed for samples left part of the time in vacuum and part of the time in the lab for a period of 6 months although there have been past reports^[11] of degradation of highly reflective surfaces. The 3M Al tape, having a cost one-sixth that of the Eccoshield Al tape, is definitely better than NRC-2 MLI in all cases. It offers the hope of reaching heat transfer rates below 10 mW/m^2 without going to complicated floating shield or refrigerated shield systems. Hence, we concluded that commercial aluminum tapes offer a practical alternative for insulating 77K and 4.2K surfaces, especially in the area of penetrations. Good places to further try out these tapes are simple vacuum insulated cryogenic transfer lines and the cold traps of pumping stations.

- Alternatively, aluminum tape with no adhesive can be ordered and epoxied onto the cryogenic surfaces with a non-outgassing epoxy.
3. The floating shield concept may be relatively less attractive because of the complexity and manpower involved in the construction. In the near future, an electropolished Cu (protected by benzotriazol) 4.2K surface facing electropolished Cu 77K surface will be tested.
 4. Through the use of the combination of the various different methods of insulation, we are confident that heat leak rate of the order of $6-7 \text{ mW/m}^2$ can be obtained. This is approximately 15% of the number, 43 mW/m^2 , usually used for design purposes.
 5. The insulating system chosen for the Chicago Cyclotron Magnet will probably be 8 layers NRC-2 MLI on the liquid helium cryostat wall and the 3M aluminum tape on a LN_2 temperature shield. Expected heat load would be of the order of 10 mW/m^2 .
 6. RRR measurement offers a simple method for selecting material to be used as a reflector, e.g., OFHC Cu CDA101 having a higher RRR than ETP Cu is a better choice for the construction of a LN_2 shield in a superconducting magnet. The cost of CDA101 Cu is only 5% higher than that of ETP Cu. In the absence of adequate low temperature emissivity, the best way to evaluate an insulating system to be used in a large superconducting magnet system is to perform engineering testing in devices similar to the one described in this paper.

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NOTATION

A = Cold surface area

c = Velocity of light

$$C_S = \text{Liquid specific heat at saturation}$$
$$\frac{dM}{dt} = \text{Rate of change of mass flow rate}$$
$$\frac{dp}{dt} = \text{Rate of change of atmospheric pressure (mm Hg/hr.)}$$
$$E_g = \text{Energy gap (threshold)}$$

ER = Evaporation rate due to change in atmospheric pressure (SCFH)

h = Planck's Constant 6.626×10^{-34} (Js)

$$h_v = \text{Heat of vaporation}$$

H_C = Critical field of a superconductor

$$k = \text{Boltzman's Constant } 1.381 \times 10^{-23} \text{ (JK}^{-1}\text{)}$$
$$I = \frac{C_S}{V_L h_v} \left(\frac{\partial T}{\partial p} \right)_{SAT}$$

K = Thermal conductivity

K_0 = Thermal conductivity x mass density

P = Barometric pressure

$$\dot{Q} = \text{Heat Transfer Rate}$$
$$\begin{aligned} (\text{RRR})_T &= \text{Residual resistivity ratio} \\ &= \frac{\text{electrical resistivity of a material at room temperature } \rho_{300\text{K}}}{\text{electrical resistivity of a material at temperature } T \rho_T} \\ &\quad (T \text{ usually } < 300\text{K}) \end{aligned}$$

t = Time

T = Temperature

$$T_1 = 4.2 \text{ K (liquid helium temperature)}$$
$$T_2 = 77.4 \text{ K (liquid nitrogen temperature)}$$

T_c = Critical temperature of a superconductor

V = Container volume (liters)

V_L = Volume of liquid involved

Greek Symbols

ϵ_1 = Surface emissivity of the cold surface

ϵ_2 = Surface emissivity of the hot surface

λ = frequency

ν = Wavelength

$\left(\frac{\partial T}{\partial p}\right)_{SAT}$ = Slope of the temperature vapor curves

σ = Stefan-Boltzman Constant $5.67 \times 10^{-8} \text{ Wm}^{-2} \text{ K}^{-4}$

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Table I. Heat Transfer from 77.4K to 4.2K

Run No.	System		Heat Transfer Rate Unit Area in mWm ⁻² (mW/ft ²)	Remarks
	4.2K surface	77K surface		
1.	Eccoshield Al tape	Eccoshield Al tape	15.1 $\pm$ 0.5 (1.40 $\pm$ 0.05)	Calculated value = 16.5 mW/m ² using emissivities values deduced from paper of Chaussy, et al.
2.	Eccoshield Al tape	Eccoshield Al tape	14.9 $\pm$ 0.5 (1.38 $\pm$ 0.05)	Same surfaces after being left in lab and in apparatus for 6 months. No degradation observed. Repeatability of data checked.
3.	Electrolytic Tough Pitch Cu (rinsed with Bright Dip and cleaned with Oakite)	Electrolytic Tough Pitch Cu (rinsed with Bright Dip and cleaned with Oakite)	15.6 $\pm$ 0.8 (1.45 $\pm$ 0.075)	Experiment was run for 11 days continuously. No sign of degradation observed. Typical method used on large magnets previously constructed at Fermilab.
4.	3M Al tape	3M Al tape	12.4 $\pm$ 0.5 (1.15 $\pm$ 0.05)	Industrial Tape #425 seems to have better adhesive property at 77K than Eccoshield Tape.
5.	Blackened with black 3M Velvet paint; then 8 layers of NRC-2 wrapped around the black surface	1 layer of Double-side aluminized Mylar epoxied to 77K Cu surface using Crest #7344 epoxy. [†]	28.8 $\pm$ 0.5 (2.00 $\pm$ 0.05)	Aluminized Mylar, measured to have a 500 Å aluminum layer on each side.* Outgassing of epoxy detected.

*King-Seeley Company, same supplier as NRC-2.

[†]Crest Products Company, Santa Anna, California 92704.

Run No.	System		Heat Transfer Rate Unit Area in mWm^{-2} (mW/ft^2)	Remarks
	4.2K surface	77K surface		
6.	Blackened with black 3M Velvet paint; then 8 layers of NRC-2 wrapped around the black surface	6 layers of NRC-2 attached by nylon screws and polyester threads.	17.3 ± 0.5 (1.60 ± 0.05)	77K surface has 2 inter-weaving areas carefully prepared.
7.	Blackened with black 3M Velvet paint; then 8 layers of NRC-2 wrapped around the black surface	Aluminum floating shield	18.1 ± 0.5 ($<1.68 \pm 0.05$)	Nylon screws with stainless steel nuts were used as the standoffs. Floating shield made from 0.127 mm thick aluminum sheets.

Table II. Aluminum Surface Characterization
with RRR Measurements

Aluminum Surface	$RRR_{77K} = \frac{p_{300K}}{p_{77K}}$	$RRR_{4.2K} = \frac{p_{300K}}{p_{4.2K}}$
Eccoshield Aluminum Tape (0.076 mm thick without adhesive)	8.26 ± 0.25	30.1 ± 0.4
3M Aluminum Tape #425 (0.089 mm thick without adhesive)	8.71 ± 0.20	33.2 ± 0.2
0.127 mm thick Aluminum Sheet for Run No. 7	8.22 ± 0.10	26.1 ± 0.6
Double Aluminized Mylar MLI (500 Å aluminum on each side of 0.0127 mm thick Mylar)	1.81 ± 0.02	1.94 ± 0.06

FIGURE CAPTIONS

- Fig. 1a Typical arrangement of insulation system in large superconducting devices.
- Fig. 1b The floating shield concept.
- Fig. 2 Dependence of the emittance of a surface on its temperature and the average wavelength of the incoming thermal radiation.
- Fig. 3 Thermal radiation test set-up.
- Fig. 4 Exterior view of the thermal radiation test apparatus (without the insulation shroud and one side wall of the nitrogen box) with the large diameter vacuum chamber in the background.
- Fig. 5 Typical data obtained from a run.
Run #10: 8 layers of NRC-2 on blackened 4.2K surface, Eccoshield Al tape on 77K surface. Average heat load into the 4.2K surface per unit area $\dot{Q}/A = 13.45 \pm 0.5 \text{ mW/m}^2$.
- Fig. 6 Radiation heat flux vs. number of layers of NRC-2 in a 50.8 mm gap from 77K to 4.2K. There is a broad minimum in the curve.

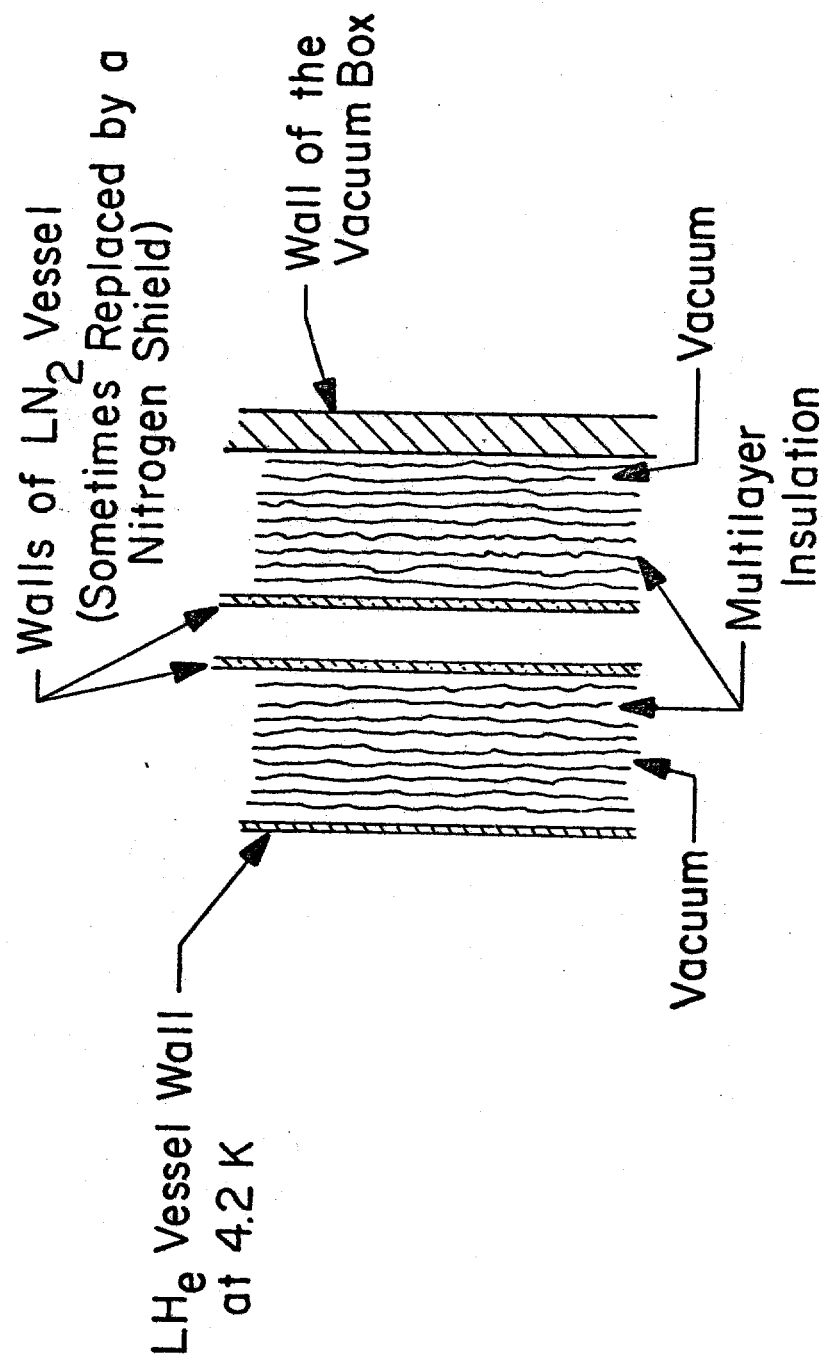


Figure 1a

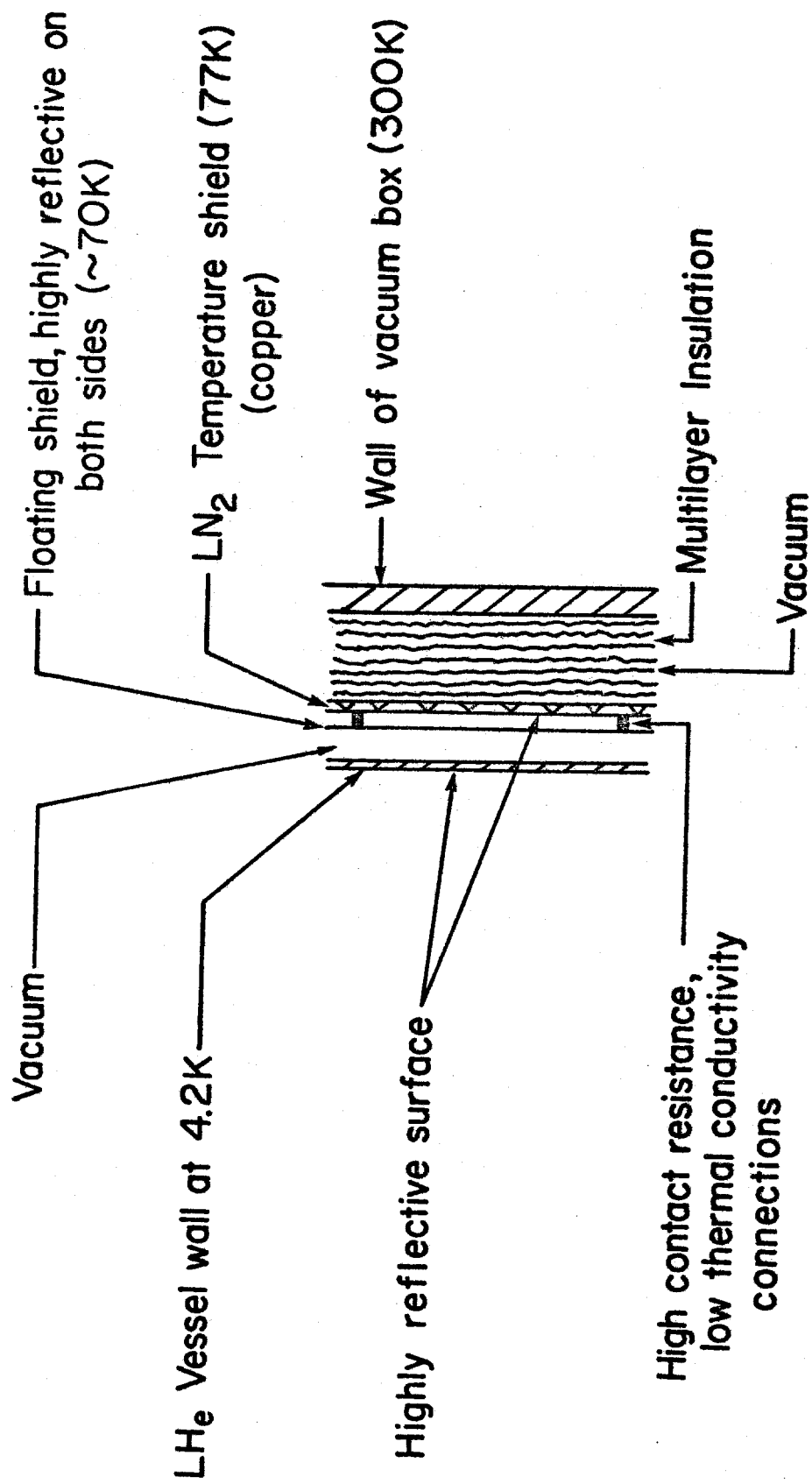


Fig. 1b

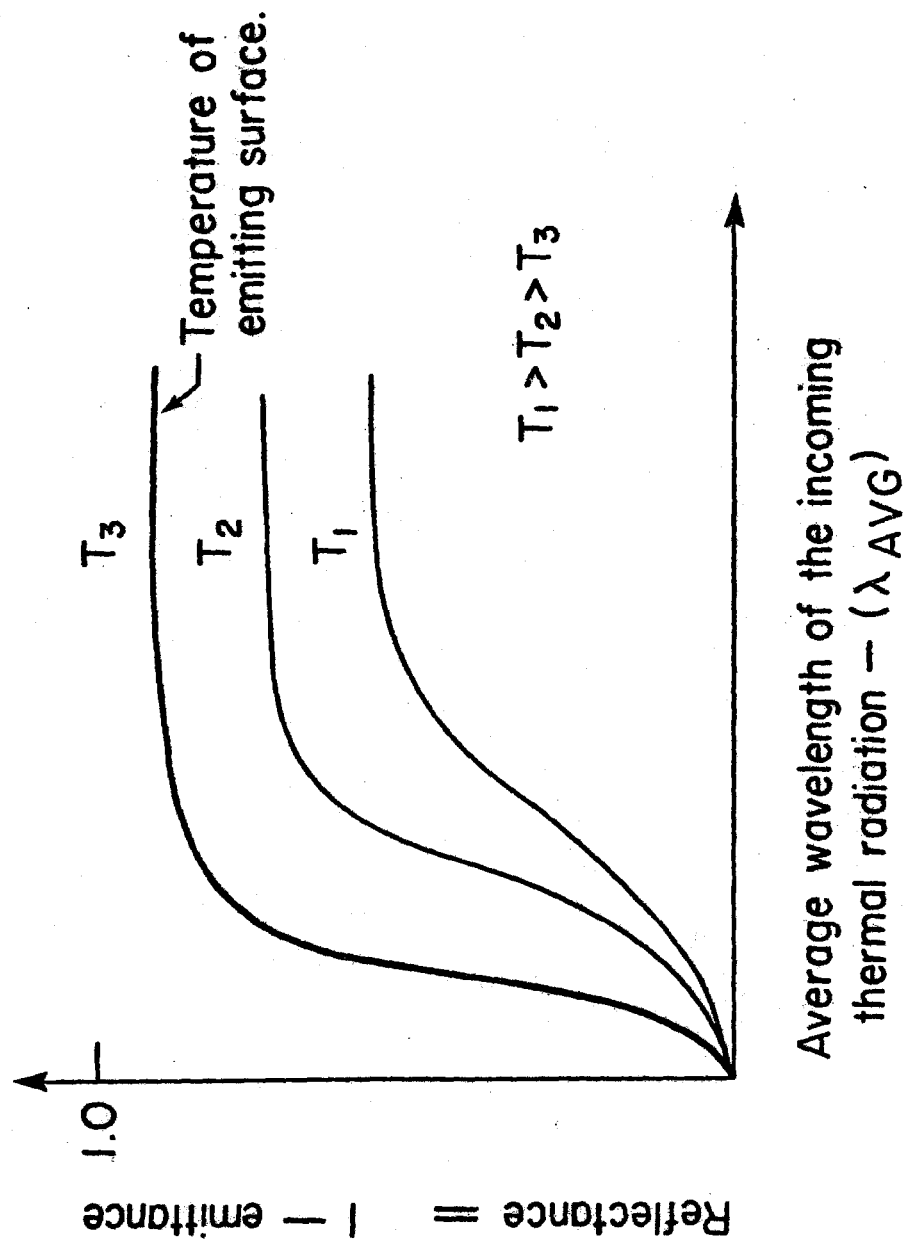


Figure 2

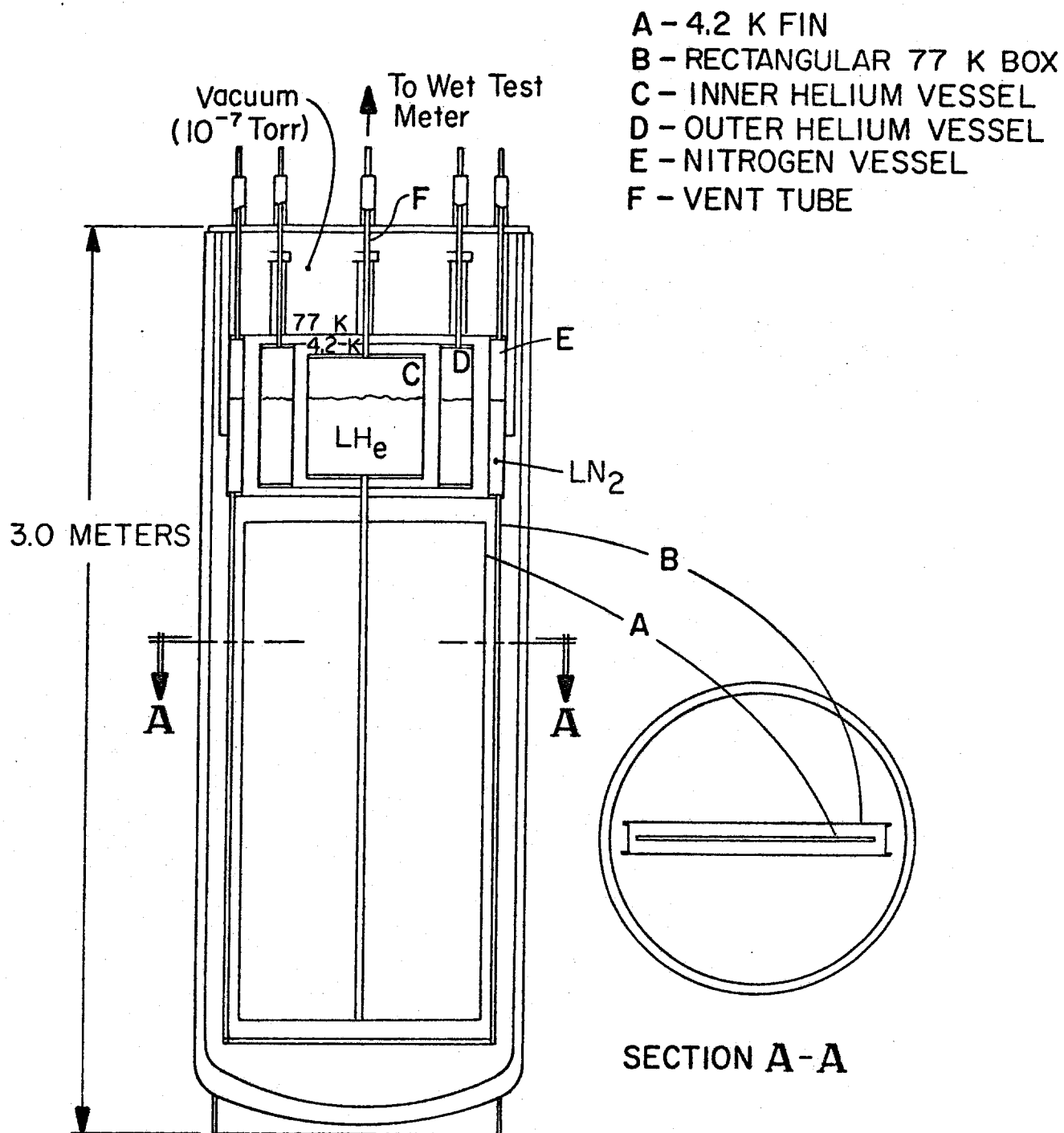
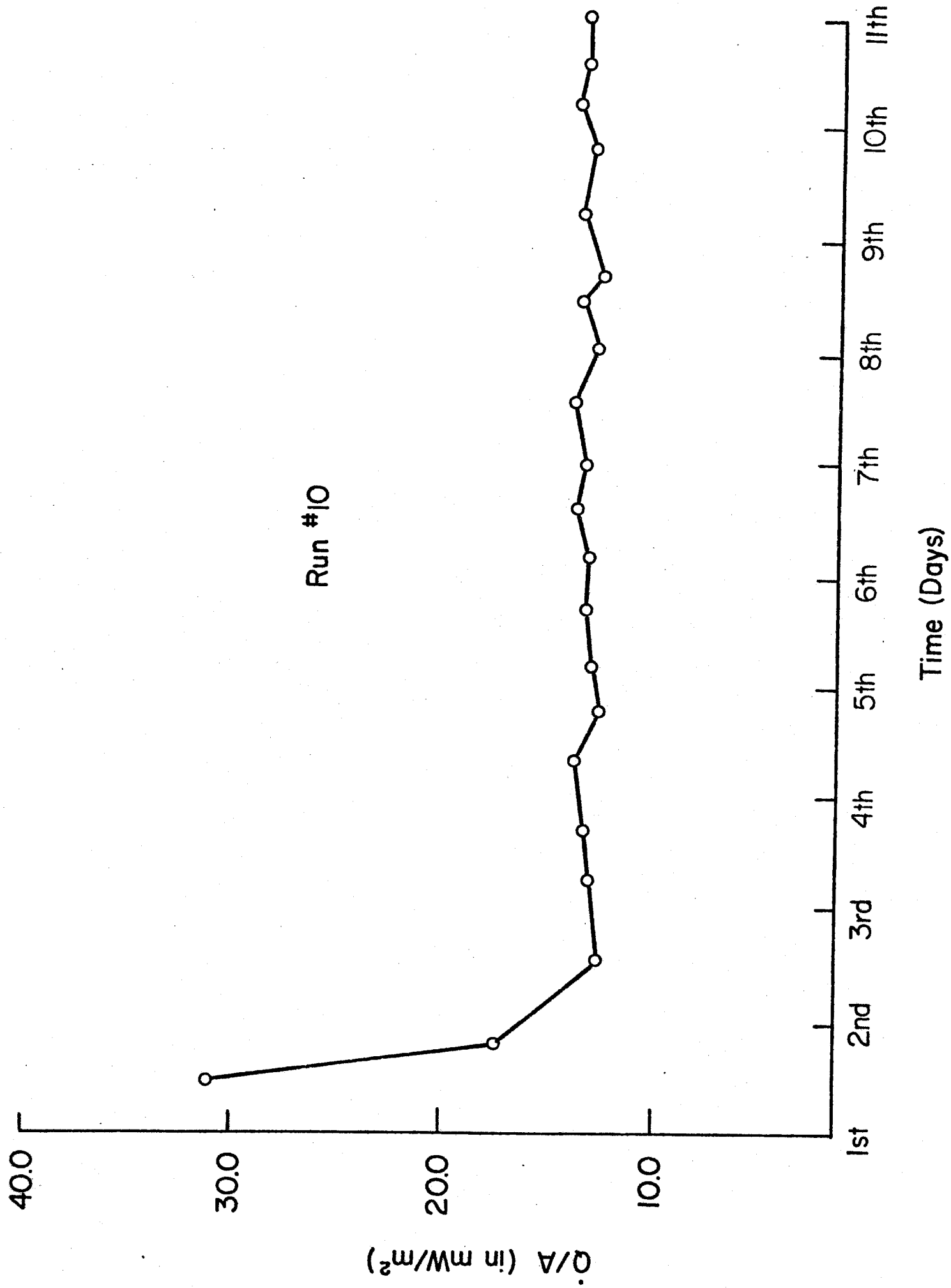


Figure 3



Figure 4



NRC-2 SERIES RUNS

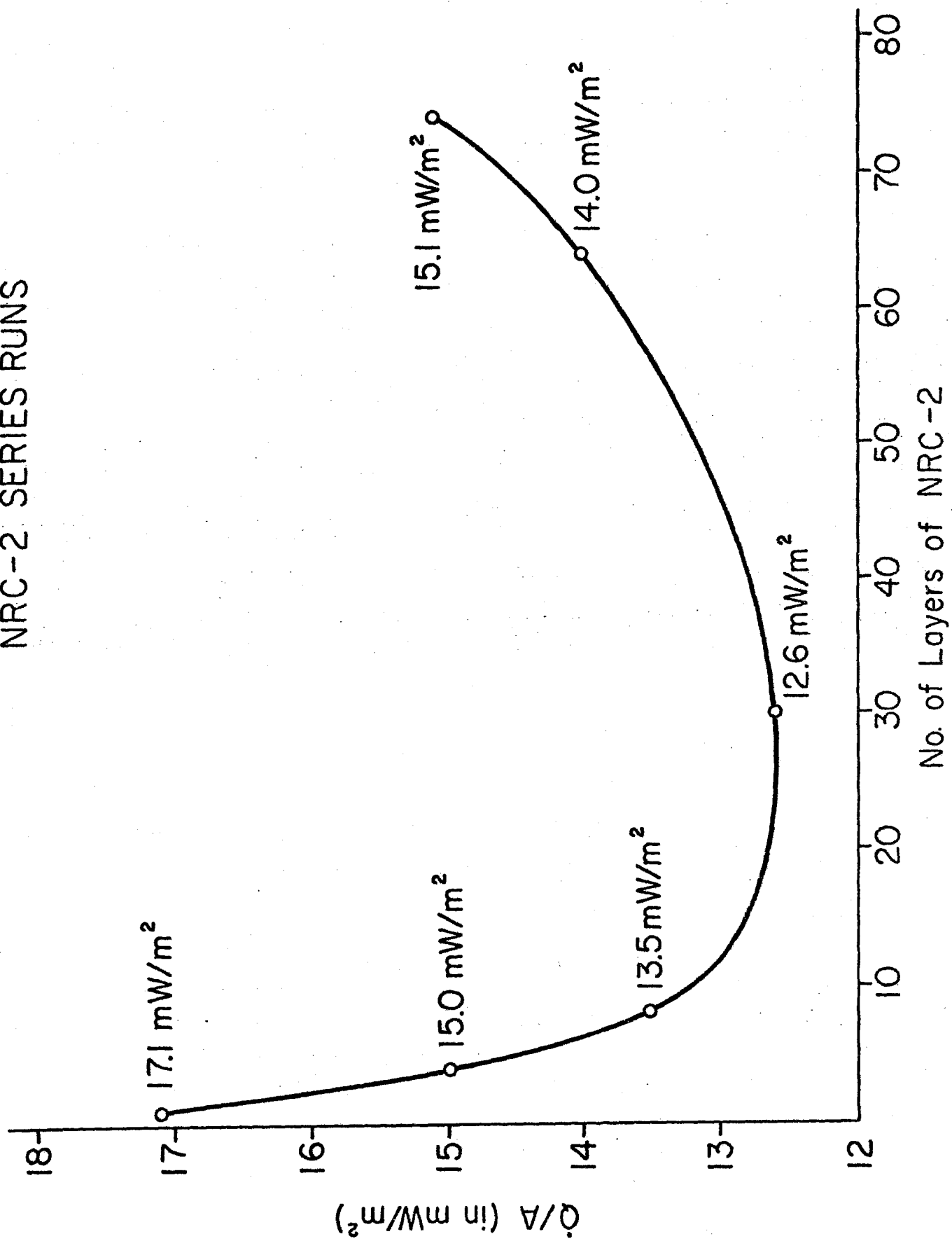


Figure 6