MODEL 400 TL COMPLETE DILUTION

REPRIGERATOR

INSTRUCTION MANUAL

Supplied to : Triumf 4004 Wesbrook Mall UBC Campus Vancouver Canada V6T 2A3

Project No : 28677

OXFORD INSTRUMENTS LIMITED OSNEY MEAD OXFORD OX2 ODX ENGLAND

TELEPHONE: (0865)241456TELEX: 83413CABLE: HELIUM OXFORD

July, 1987 28677SC.VWF

CONTENTS

1. Introduction 2. Principle of Operation 3. Drawings of insert and dilution unit 4. The pumping systems 4.1 Helium-3 gas handling system Helium-4 and high vacuum system 4.2 Cooling down procedure 5. 5.1 Fitting experiment and closing the insert 5. 2 Checking capillary throughput 5. 3 Room temperature tests 5. 4 Liquid nitrogen tests 5. 5 Cooling to 4.2K 5. 6 Cooling to 1.2K 5.7 Introduction of the sample 5. 8 Condensing and starting circulation 5. 9 Changing temperatures and flow rates 5.10 Operation of the continuous fill needle valve on the 1K pot Changing and cleaning cold traps 5,11 5.12 Temperature control on a Dilution Refrigerator 6. Top loading procedure 6.1 Loading the sample into the refrigerator 6.2 Unloading the sample 6.3 Operating procedures - Co⁶⁰ sources 7. Closing down, and warm-up procedure 8. Maintenance and routine checking operations 8.1 Introduction 8.2 Use of the cryopump Security, safety and trouble shooting 9. 9.1 Active security systems 9.2 Passive security systems Action after automatic shutdown 9.3 9.4 Loss of base temperature 9.5 Fault finding 10. Dilution Refrigerator Test Results 11. Electrical wiring 11.1 Insert and dilution unit 11.2 Pumping system

INTRODUCTION

The helium-3/helium-4 dilution refrigerator first proposed by London (1951) has now become a standard laboratory instrument for producing ultra-low temperatures for a variety of research purposes. While the number of scientists interested in the thermo-dynamics of attaining these low temperatures is decreasing, more and more scientists are interested in experimenting at these temperatures. The aim of Oxford Instruments is to provide Dilution Refrigerators for these users by producing a reliable and comparatively simple system which works with the minimum amount of expert knowledge and associated 'black magic' still connected with these instruments.

The present manual contains however a short description of the theory of operation of a dilution refrigerator which will enable the user to gain a fuller understanding of the working and operation of his system. This might also be useful during fault diagnosis when the system, for one reason or another, does not reach the specified temperatures.

The present design of this refrigerator takes into account several years of practical experience and development. The system is now as reliable as present day technology permits. We at Oxford Instruments hope you will be entirely satisfied with both the performance and reliability of the refrigerator and wish you prolonged and successful operation.

Principle of Operation

The principle of operation of the dilution refrigerator was originally proposed by H. London in 1951, but the first working systems were not built until more than ten years later. Since that time, the performances of these systems have steadily improved, and the physical processes involved have become much better understood.

When a mixture of the two isotopes of helium is cooled below a critical temperature, it separates into two phases. The higher "concentrated phase" is rich in helium-3 and the heavier "dilute phase" is rich in helium-4. The concentration of helium-3 in each phase is dependent upon the temperature as shown in figure 2.1. Since the enthaplies of the helium-3 in the two phases are different, it is possible to obtain cooling by "evaporating" the helium-3 from the concentrated phase into the dilute phase. This process continues to work even at the lowest temepratures because the equilibrium concentration of helium-3 in the dilute phase, is still finite even as the temperature approaches absolute zero.

When the temperature of the helium-4 is below about 0.5K, it can be regarded as being in its quantum mechanical ground state. It has zero spin and is superfluid. The helium-3 atoms however, have a spin of one half and in the dilute phase their behaviour can be described as that of an ideal Fermi-Dirac gas. They move through the helium-4 (which is both thermally and hydro-dynamically inert) as if they were in a vacuum. In the dilution refrigerator this gas is formed in the mixing chamber at the phase boundary, and the cooling obtained by this process is used to cool a sample.

In a continuously operating system, the helium-3 must be extracted from the dilute phase (to prevent it from saturating) and returned into the concentrated phase keeping the system in a dynamic equilibrium. Figure 2.2 shows a schematic diagram of a continuously operating dilution refrigerator. The helium-3 is pumped away from the liquid surface in the still, which is maintained at a temperature of 0.6 to 0.7K. At this temperature, the vapour pressure of the helium-3 is about 1000 times higher than that of helium-4, so helium-3 evaporates preferentially. A small amount of heat is supplied to ensure that the still does not cool as this evaporation takes place.

The concentration of the helium-3 in the dilute phase in the still therefore becomes lower than it is in the mixing chamber, and consequently there is an osmatic pressure difference which drives a flow of helium-3 to the still. The helium-3 leaving the mixing chamber is used to cool the returning flow of concentrated helium-3 in a series of heat exchangers. In the region where the temperature is above about 50mK, a conventional coiled tubular heat exchanger can be used effectively, but at lower temperatures than this the thermal boundary resistance (Kapitza resistance) between the liquid and the solid walls increases with T^{-3} , and so the contact area has to be increased as far as possible. This is done by using sintered silver heat exchangers, which are very efficient even at the lowest temperatures.

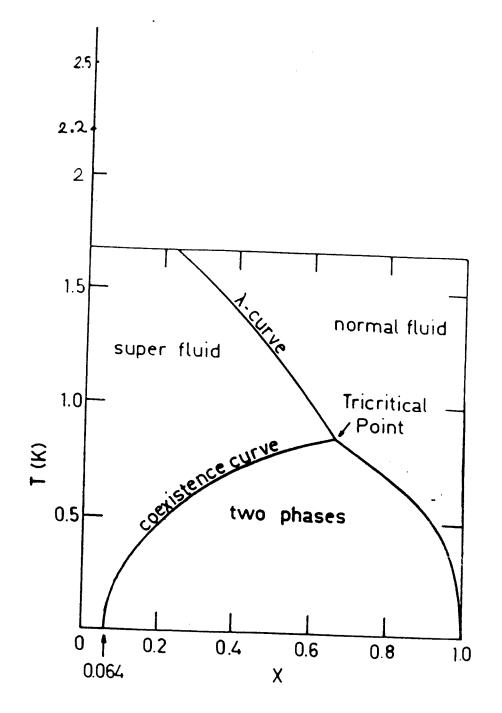
-2.1-

The room temperature vacuum pumping system is used to remove the helium-3 from the still, and compress it to a pressure of a few hundred millibar. Since the cooling power of a dilution refrigerator is directly proportional to the flow rate of helium-3, the large cooling power refrigerators must have very powerful pumps, and wide pumping lines are needed in the low pressure regions.

The gas is then passed through filters and cold traps to remove impurities and returned to the cryostat, where it is precooled in the main helium bath and condensed on the 1K pot. A flow impedance in the form of a capillary tube is used to maintain a high enough pressure in the 1K pot region for the gas to condense.

The experimental apparatus is mounted on the mixing chamber, ensuring that it is in good thermal contact. All connections to the room temperature equipment must be thermally anchored at various points on the refrigerator to reduce the heat load on the mixing possible base giving the lowest minimum, the chamber to If the experiment is to be carried out at higher temperatures. temperatures, the mixing chamber can be warmed by applying heat to it directly, and a temperature controller can be used to give good stability.

This description should enable the operator to understand the detailed description of the operating procedure of the system, which will be found later in the manual.

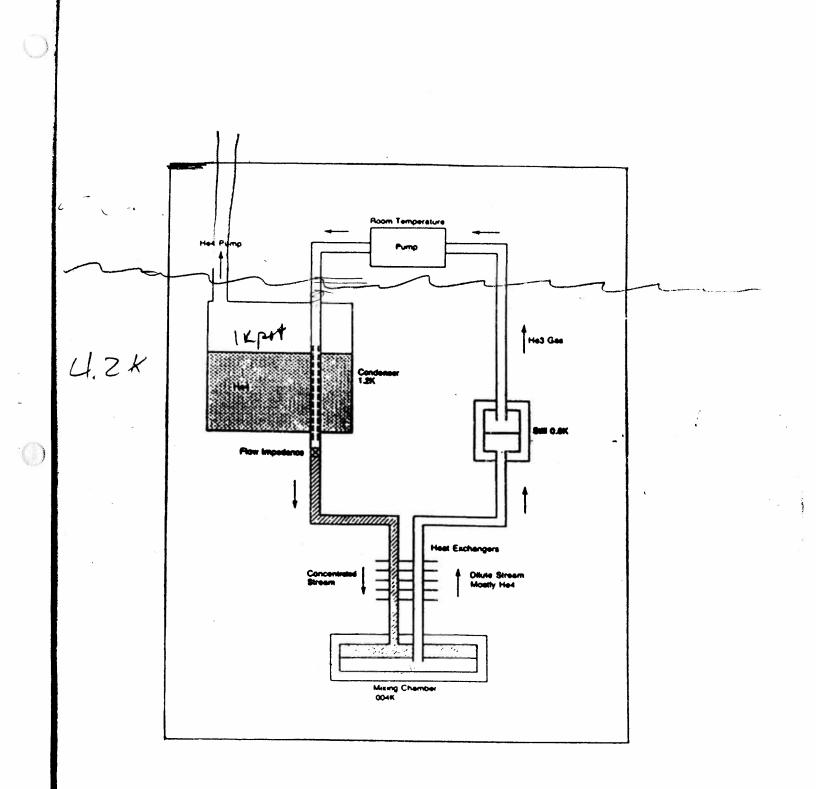


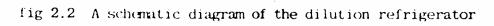
Phase diagram of ${}^{3}\text{He}/{}^{4}\text{He}$ mixtures, x = n₃/(n₃ + n₄) is the ${}^{3}\text{He}$ concentration. The tricritical point is at 0.86 K. figure 2.1

1)

)

,t





3.1 Description of insert and dilution unit

Enclosed with the manual are drawings of the top plate, the top of the IVC and the bottom of 1K pot, with a description of all ports and services needed for the operation of the D/R or for customers use.

Drawing	No.	AFA0410	System GA
		AFA0601	Sample Holder
		ARZ0403	Pumping Cabinet

100 micromoles / See 0.12 lit/mis

4.1 The helium-3 gas handling system

The circuit diagram for the helium-3 gas handling system as it is drawn on the panel is shown in ARZ0403.

The helium-3 is circulated by a 9B3 booster pump, backed by a Leybold $65m^3$ rotary pump. This combination is capable of circulation rates up to approximately 800 micromoles/sec. The pumps are switched from the panel.

Two Pirani gauges are fitted to measure the inlet pressure Pl and the booster backing pressure P2.

The inlet pressure can also be seen on G2 via valve 7. The helium-3 is filtered through oil mist filters and the outlet pressure is seen on G4 which is fitted with a trip switch normally set at around 950 mbar. When triggered, the safety circuit is activated, and it will stop the pumps and open a by-pass valve (parallel to valve 2) between the condenser line and the still pumping line.

In continuous operation the helium-3 is filtered through one of the two nitrogen cold traps (CTl or CT2) and flows back into the system via the return line and a helium cold trap. After passing through these cold traps the helium-3 is returned to the cryostat.

The mixture is stored in the dumps which are accessible from the panel via valves 5 and 9. The dump pressure is read on Gl. The dump vessels themselves are individually supplied with shut off valves, so that each dump can be isolated from the others.

Air or exchange gas can be pumped out of the system through values 6 and 10. The points marked "B" on the panel are all connected together, and an auxiliary pump can be connected to the manifold. A leak detector can be fitted to the point marked "Vent 1" giving access to the system through value 11. The warning lights on the panel indicate the following faults.

- 1. 1K pot rotary pump failed.
- 2. Backing pump for high vacuum diffusion pump failed.
- 3. Diffusion pump failed.
- 4. Booster pump over-heated.
- 5. Helium-3 rotary pump failed.

4.2 Helium-4 and high vacuum system

7 E W J

The circuit diagram as shown on the left side of the panel is reproduced in ARZ0403.

As can be seen, there are five lines going to the insert and dewar:

- The OVC line that connects to the Outer Vacuum Can of the dewar.
- The vacuum lock line should be used while inserting the top loading probe.
- The recovery line, which is connected to the main helium-4 bath, and can be connected to the customers recovery system. The pressure is measured on G5.
- The right-hand line runs to the IVC, inner vacuum can, which surrounds the dilution unit and the 1K pot.
 - Next to that runs the 1K pot line, which is used to pump the helium-4 from the 1K pot thereby reducing its temperature to about 1.2K, and enabling the helium-3 to condense. The system is fitted with an Edwards E1M18 rotary pump. Gauge G6 indicates the pot pressure.

After assembly of the tails and closing of the dewar the OVC should always be the first to be pumped. In any case <u>BEFORE THE MAIN BATH</u> <u>IS PUMPED</u>.

Both OVC and IVC are pumped with a 180 l/sec vapour diffusion pump, which is backed by an Edwards E2M8 roughing pump. Either this pump or the 1K pot pump cam be connected to line B which connects to the Helium-3 gas handling system.

The diffusion pump circuit includes a Penning and Pirani gauge (P3) to monitor the inlet and backing pressures respectively.

All the different spaces 1K pot, OVC and IVC can be vented with exchange gas from the recovery system or an external source via Vent 2.

1S, PROPERLY , DR/ INSERT INNER / PUTER For bend LANGE HERI ARG NMEM Far Section 5 EWS ATTE DA mark The Cooling Procedures

F

A134

baild.

Main mal

L'an

25 1

This section assumes that the system has been properly installed and the gas handling system, interconnecting lines and insert have all been leak tested and are leak tight. Furthermore it is assumed that the system has been run before.

Fitting experiments and closing the insert 5.1

The insert is fitted with 3 x 18 electrical leads which end in flat connectors under the 1K pot. Twelve leads are permanently in use for the operation of the dilution unit, the others are available for experiments.

Before closing the IVC the system should be carefully checked for ate any radiation leaks from room temperature through holes in the IVC top flange. Stainless tubes act as good wave guides for this radiation whose intensity is 46 mW/cm². 3

Also check all electrical connections to the dilution unit (see section 11) and your experiment.

Before closing the IVC it is advised that the IK pot needle valves are checked. This is done as follows. Close the needle valves (17, and 18 in AFA0410) and using the He4 pump evacuate the 1K pot to 0.1 mbar (zero on G6). Close valve 24 and check that each needle valve can be opened smoothly and monitor the throughput by checking how quickly the pressure rises on G6. Throughput values greater than 10mbar/min should be expected.

Having checked the 1K pot and the electrical connections, we advise that the ³He capillary throughput is checked as described in the 13 next section.

Checking capillary throughput 5.2

The dilution unit is fitted with a flow impedance below the condenser in the form of a restricted capillary. This should be checked to ensure it has the right throughput and is not blocked by any contaminants.

Using the high vacuum pumping system evacuate line B which connects the helium-3 system via valve 10 with the auxiliary pumping system. Then open valves 2, 3, 6, 7 and 10 and evacuate the dilution unit. If the system has been run recently and the lines or mixing chamber have not been opened, the pressure inside the dilution unit should te less than a few mbar. If the pressure is higher than this, it is likely that there is some contamination in the system.

(lose valves 2, 6 and 10. Connect a supply of pure helium-4 (99.99%) to vent 1. Open valves 10 and 11 to evacuate the line, Connect a supply of pure helium-4 cose valve 10 and fill it with pure helium gas.

Open value 6 slowly to allow the pressure in the still line to rise to 1 - bar, which can be seen on gauge G2. The capillary throughput can now be compared with the test results as given in Section 10 and should be within approximately 25%.

2 2 L n

After verifying the capillary throughput open valve 2 to equalise the pressure in the still and condenser lines. Close valve 11, open 10 and re-evacuate the dilution unit via line B.

Close the IVC and fit the dewar tails. Then check all the electrical connections again.

5.3 <u>Room temperature test</u> !! MAKE SULE THAT IVC PUMP LINE COMMAL VALUE IS OPEN, TURN CLOCKWISE!! Using the roughing pump/diffusion pump system, pump the OVC. When the pressure is less than 1 mbar close the OVC and open the values to the IVC.

Check the pressure in the dilution unit (D/U) on G2 and G3 is less than 1 mbar. If not open values 10, 6, 7, 2 and 3 to connect the D/U to the auxiliary pump via line B and pump to less than 0.1 mbar. Make sure the helium cold trap values in the return line are open.

Connect a leak detector to the port below valve 36 on the auxiliary panel, and pump the IVC and OVC to a high vacuum.

Close the needle valves to the 1K pot. Evacuate the helium can and 1K pot using the helium-4 pump. Stop pumping when the pressure is below 1 mbar. Vent the helium bath to 1000 mbar with helium gas (leaving the 1K pot under vacuum), while checking the O.V.C and I.V.C for leaks with the leak detector. This procedure can be performed by drawing gas from the recovery line, or using a separate gas bottle attached to vent 2.

Leave the leak detector connected to the IVC and by opening a meedle valve, vent the 1K pot to 1 bar with helium from the main bath, while checking the leak detector signal. Close the needle valve and re-evacuate the 1K pot using the helium-4 pump. Similarly, check that all other needle valves work correctly. Check that the needle valves seal properly be watching for a pressure rise on G6 when valve 24 is closed.

When all the above tests have been completed satisfatorily, (i.e. no signal on leak detector observed), close valves 2 and 10. It is advised that the capillary throughput is checked again before cooling with nitrogen.

After verifying the capillary throughput, open value 2 which will equalise the pressure in the still and condenser lines. At this point, check that no signal has appeared on the leak detector.

Re-evacuate the dilution unit and 1K pot. Then disconnect the leak detector and connect a supply of dry nitrogen gas to vent 2. Fill the 1K pot with one atmosphere of clean helium gas, and maintain this pressure while the system is cooled to 77K. Make sure that the top loading entry baffles are closed and start to cool the system.

Continue to pump the O.V.C. with the diffusion pump while the system is cooled.

Liquid nitrogen tests 5.4

11 IUC COAXIAL PUMP VALVE

The helium vessel can be filled with nitrogen, to pre-cool the system to 77K.

Cooling to 77K

The port labeled magnet

Open the main bath helium recovery port to air $\mathcal J$ Insert a tube into the main helium reservoir through the siphon fentry and screw it into the cone mounted on the top of the I.V.C. Connect a liquid nitrogen dewar to the top of the tube and slowly begin to transfer nitrogen into the main helium bath. It takes about 1/12 of the longe (1502?) LN2 dewars to bill completely. Fill slowly over about 6-3 hours, e (1502?)

Half fill the main bath with nitrogen, and when the boil off has settled, fit a non-return valve to the helium recovery port, remove the tube from the siphon entry port and replace the bung. Wait for the dilution unit to cool to 77K by noting the resistance values. Pre-cooling time will depend on the exchange gas used in the IVC, (typically overnight for nitrogen and 6 hours for helium).

77K Test procedure IVE CONTAL VALUE STILL OPEN

Evacuate the IVC and connect the OVC and the IVC to the leak detector.

N.B. Do not evacuate the IVC before closing the OVC cryostat hand valve, as there should be high vacuum in the OVC. (thin window between

IUCYOU Insert a tube through the siphon port, ensuring that it scews into the cone on the top of the I.V.C. Connect the top of the tube to one of the liquid nitrogen reservoir filling bushes using a length 2 th of silicon rubber tube. Pressurise the helium reservoir with a small overpressure of helium gas and blow all the liquid nitrogen out of the main bath into the nitrogen reservoir. Pressurisation is conveniently done using a gase bottle connected to the vert of the exchange gas manifold.

When the liquid nitrogen level is below the level of the blow-out tube in the main bath, the pressure drops. This can be checked by stopping the gas flow at vent 2, removing the rubber tube from the top of the blowout tube and ensuring that no liquid comes from the blowout when the bath is pressurised again.

Remove the blowout tube and put the bung in the siphon entry. The bath should now be evacuated using the helium-4 pump, and the pressure should fall steadily to less than 1mbar. If the pressure hesitates at approximately 100mbar, it is likely that there is still some liquid nitrogen in the bath, and this should be removed by inserting the blow-out tube and pessurising the bath again.

When the main bath pressure has been reduced to less than <u>imbar</u> the IK pot should also be evacuated, also using the helium-4 pump. Then close the valve between the IK pot line and recovery line (valve 31 in ARZ0403) and connect the helium recovery line back to the cabinet. Vent the main bath to 1000 mbar with helium gas as previously described while watching the leak detector. Open a needle valve and vent the pot to 1000 mbar with helium gas. Close the needle valve and check that the other needle valve is not blocked. Connect both the IK pot (via valve 31) and the main bath to the recovery line.

10 ~ Smbar i

Evacuate the dilution unit via line 'B' using the rotary pump by opening valves 10, 6, 7, 3 and 2. Purge the line, close valves 6 and 2, and vent the still pumping line to 1000 mbar of pure helium gas, via the front panel vent, using the method described previously. Check that the capillary throughput on gauge G3 is between x6 and x8 of the room temperature throughput. Open valve 2 to vent the condenser line to 1000 mbar, checking that no signal has appeared on the leak detector. Evacuate the dilution unit using the roughing pump and then close valves 10, 11 and 6 on the helium-3 panel leaving 3, 7 and 2 open.

Charpe lite fort when @ -20% full. (So that come no longer used) 5.5 Cooling to 4.2K

Isolate the I.V.C. and admit approximately 3 litres (a balloon of 6 or 7" diameter) of helium exchange gas to the I.V.C. using the exchange gas manifold. Continue to pump the O.V.C. until liquid helium collects in the main bath.

" When the label reaches 350 recipe transfer take (in DR) about 2" Insert the helium transfer siphon ensuring that it fits into the cone on the IVC. Transfer helium at about 100-200 litres of gas per values, begin pumping the exchange gas from the I.V.C. (As an indication, this should happen at about the time liquid starts to collect in the main bath). Continue the transfer until the desired helium level is achieved. Top up the liquid nitrogen jacket.

 μ_{L} ef The I.V.C. should be pumped for at least 5 hours using the diffusion putt pump. The helium signal should then be less than 10^{-6} mbar l/sec, as determined by the leak detector. During this time, the 1K pot should not be cooled to below 4.2K.

N.B. The above describes one method of cooling from 77K to 4.2K. Several other methods may be used, such as the "20K transfer" method or by employing different exchange gas in the I.V.C. The exact method to be used depends on personal preference and experience will indicate the exact point at which exchange gas should be pumped away in order to produce the most efficient cooldown cycle.

NOTE: WHEN THE OVC HAS BEEN CHECKED FOR LEAKS FROM THE MAIN BATH, THE O.V.C. HAND-VALVE ON THE CRYOSTAT SHOULD BE CLOSED, MAKING IT DIPPICULT TO VENT THE O.V.C. ACCIDENTALLY.

A. Sta

5.6 Cooling to 1.2K

Make sure that the 1% pot needle valves are closed and start pumping the pot, checking that the pressure drops to below 1 mbar. Connect the leak detector to the IVC and watch for leaks as one of the needle valves is opened. The pressure on G6 will rise to a steady value as the pot fills, and will suddenly increase after a few minutes, indicating that the pot is overflowing. Close the needle value and nump the pot to base temperature. valve and pump the pot to base temperature.

5.7 Introduction of the Sample

The sample may, now be introduced into the refrigerator. procedure for top loading is described in section 6. The

AFB. INC BAFFLE IS OPEN WHEN

THE WORD "OPEN" IS PARRALLEL TO EVE PUMPMING LINE

5,5

- 5.5 -

5.8 Condensing mixture and starting circulation

When the 1K pot is filled with helium and pumped to below 10 mbar, check that no signal has appeared on the leak detector. If not then close valve 2. Check valves 3 and 7 and the valve on top of the helium cold trap is open. Open the dump hand valves and slowly open valve 5, to allow the mixture to condense into the still and Gauge G3 should indicate 20-50 mbar after a few minutes. Condense the mixture at about 150-200 mbar pressure on gauge C2. It may be necessary a re-fill the 1K pot as previously described. Continue to condense the mixture until valve 5 is fully open and the still pressure is about 100 mbar, (which typically During this time, refer to section 5.11 and clean out the cold traps.

Switch on the sealed rotary pump. A small amount of mixture may have passed back through the pump to its inlet side. should return to the pressure it reached at the end of the previous run. If G4 is more than 100 mbar condense this via valve 9 with the mixture from the dumps. When G4 and G2 show the same pressure of about 100 mbar close valve 9 again.

Fill the liquid nitrogen cold trap dewar. G4 is set to activate at approximately 950 mbar. Put the cold traps Check that the switch on in the dewar and open valve 8, allowing the mixture in the exhaust of the sealed pump to enter one of the traps and cool to 77K. Check that the helium cold trap has been lowered into the cryostat and is open Connect the back of the sealed pump to the condenser line by Begin circulation by cracking valve 12 open keeping the pressure on G4 below 950 mbar; the pressure at which the bypass valve is activated. Gradually open valve 12 until it is fully open and P2 is less than 1 mbar. The pressure on G4 should

"But clive to it

Transfer to the booster pump by opening valve 13, closing valve 12, and opening the booster isolation valve (Q.S.B.). Check that the booster has not stalled, by noting Pl < P2. When Pl falls below 0.1 mbar, energise the still heater to promote the circulation required.

The refrigerator should now cool to its base temperature. For overnight operation, ensure values 5 and 7 are open, together with the hand valves on the storage dumps. Check that sufficient

liquid tefrigerants are in the cryostat and cold trap dewar. (To prevent boosta stall, keep ass loss 10pen 13 for abour 20 min.

5.9 Changing temperature and flow rates

The flow rate is primarily determined by the amount of heat put into Gismu? the still and varies approximately linearly with the still power. Тс

reach the base temperature the flow rate should be set indicated on and all extra heating experiments should be minimised. as from the

If the basic heat load from the experiment is higher the flow rate will have to be adjusted accordingly, and the final temperature will

The temperature can now be varied by either changing the flow rate or by putting extra heat into the mixing chamber via the M/C heate or the experiment. An electronic temperature controller can be used, but one should be aware that below 20 mk, the time to reach equilibrium will be long - sometimes up to an hour.

Experience will teach you what is the best operating mode for your application.

5.10 Operation of the continuous fill needle valve on the LK pot

Measure the helium boil off from the 1K pot in the single shot mode. Open a needle valve sufficiently to increase the boil-off by just more than a factor of two. At this rate the pot will fill continuously.

The level in the 1K pot can be conveniently seen on the level meter provided. Note that the electronics are set differently for the probe operating in superfluid. The 1K pot level indicator and the main bath indicator electronics are not interchangeable.

Top Loading Procedure

The 400 TL dilution refrigerator is designed to allow samples to be loaded into the cryostat without warming it above approximately 2.5K. Four electrical connections can be made to the sample through the contacter block on the mixing chamber. A special "top loading siphon" is used to insert the sample holder, and in conjunction with the "delivery siphon" it is also used to pre-cool the sample to liquid helium temperatures. This is done by drawing liquid from the main bath through the siphons, using a small pump. Note that care must be taken not to let air into the top loading access as this will prevent the proper operation of the system. Also, if the baffles are damaged during top loading, (so that they will not close properly), the refrigerator will not reach base temperature.

6.1 Loading the sample into the refrigerator

The aligning sample holder should be somewed firmly anti-clockwise onto the lower end of the top loading siphon and drawn into the vacuum lock. The two parts of the sample holder should be screwed together finger tight (refer to AFA0601). Note that this connection has a left hand thread. Mount the vacuum lock on the gate valve on the top of the cryostat. The gate valve must closed, maintaining the vacuum in the IVC. Make sure that the source of over valves are also closed.

Rough out the vacuum lock and sliding seal, and transfer to the diffusion pump. When the pressure is reduced to 10^{-4} mbar, close the valve to the vacuum lock and open that gate valve and lower the top loading probe until the sample holder is a few inches above the 4.2K baffle, taking care not to touch it. During this procedure the sliding seal should be pumped to prevent any air which passes the first '0' rings from entering the IVC.

Insert the delivery siphon into the main bath and connect the delivery siphon to the top loading siphon. Pressurise the main bath slightly and check that the two siphons are not blocked, by observing the gas coming out of the siphon exit. If there is no blockage, connect this port to the top laoding pump using the polythene line. Pressurise the main bath slightly, by closing off the recovery valve and using a bladder.

Prepare the refrigerator by closing, value 5, the QSB valve, and valve 13. Maintain helium-3 circulation on the rotary ramp only, and turn off the still heater.

Draw liquid helium through the top loading siphon to cool the sample holder. This should take approximately 30 minutes when loading the sample or approximately 15 minutes when the siphor is being cooled to remove the sample. When the sample is at about 4.2K the note of the pump may change, as liquid helium is drawn out of the siphon exhaust. If the pump line is removed from the siphon a jet of liquid will be seen. The next stages are most easily performed with two operators, one for the top laoding probe and one for the pumping cabinet. The siphon operator opens the radiation baffles (19 in AFA0410) and disconnects the top loading siphon from the delivery siphon after the main bath has been de-pressurised. He withdraws the delivery siphon from the main bath and lowers the top loading siphon through the dilution unit to the mixing chamber. The cabinet operator watches the pressure on G4 carefully during this procedure. When the sample holder touches the mixing chamber, the pressure on G4 will rise. If the pressure approaches 1 atmosphere some helium-3 should be allowed into the dumps by opening valve 9. Adistance from should the siphon operator then rotates the siphon clockwise to load the

The siphon operator then rotates the siphon clockwise to load the sample (anti-clockwise to unload). The clockwise motion firstly unlocks the thread between the two parts of the sample holder and then locates and locks the upper part of the sample holder into the tail piece (refer to AFA0601). The locking of this upper piece ensures that thermal contact between the sample holder and tail is obtained. Once the sample holder is tight in the tail piece the connection between the sample holder and siphon will break free. The siphon must be rotated further until it is free to be withdrawn, and it is pulled up to a point above the 4.2K baffle. The baffles are then closed, and the probe can be withdrawn completely and the gate valve closed.

When the sample holder cools to the temperature of the mixing chamber the pressure on G4 will begin to fall. When it has dropped to about 500 mbar the helium-3 from the dumps can be allowed back into the circuit by slowly opening valve 5. When P2 drops to about 1 mbar, transfer circulation back to the booster pump as described in section 5.8. energise the still heater when necessary, to promote the required circulation, and the refrigerator should cool to base temperature.

6.2 <u>Unleading the sample</u>

The sample is unloaded by following the same procedure, pre-cooling the siphon before screwing it onto the sample holder. Note that the siphon must be rotated in the opposite direction.

> Le choubler on the loading siphon 120 cm is just-above baffles On insertion 1-9.6 cm is seated in "mounting cage" in D.P. MC extension 1-atter pressure plug insterted your after 2nd "reak") ~7.8 cm E top of lock nut On removal - Final touch C 8.5 cm - After insertion into pressure plug & field (reake 7.8 cm - After insertion into pressure plug & field (reake 7.8 cm - After insertion into pressure plug & field (reake 7.8 cm - After insertion into pressure plug & field (reake 7.8 cm - After engaging "translation sciew" until tight ? - After still seated 9.5 cm (same as the beginning of insertion)

6.3 <u>Operating Procedures - Co⁶⁰ Sources (15 micro-curies)</u>

A. General

- 1. The procedure for mounting the source in the cryostat must be carried out as quickly as possible.
- 2. Keep source in container/safe box until required.
- Handle source with tweezers. Avoid direct contact hands and other parts of the body must not come into contact with the radiation source.
- 4. Do not stare directly at the source.
- 5. Do not leave crystal unattended without identification.
- 6. Wash hands thoroughly immediately after contact.

B Mounting of Co⁶⁰ Sources

- 1. Ensure that the surface on which the crystal is to be mounted is machined and tinned with Woods Metal.
- 2. Using a soldering iron, tin the crystal with Woods Metal.
- 3. Place the crystal on holder and press down with soldering iron, ensuring that the crystal is flat on the holder surface. Do not touch the crystal at any point in this procedure.
- 4. Do not use a naked flame in the vicinity of the crystal.
- 5. Wash hands, and any tools used to touch the crystal.
- 6. Avoid all direct contact with the crystal.

C. Removal of Co⁶⁰ Sources

- Remove the source from its holder and immediately place it in the container. Do not touch the source - use tweezers at all times.
- 2. Wash hands and clean all tools used to touch the crystal.

Section 7 !! BEFORE CLOSING DOWN OF WARMING OPEN INC CONTAL PUMPLINE VALUE (CLOCKWISE) Closing down and warming up procedure ALL THE WAY!!

To stop the refrigerator, close the booster isolation valve (QSB) and valve 13. Equalise the still and condenser pressures by opening valve 2. Connect the still and condenser lines to the dumps by checking that valves 3, 5 and 7 and the dump hand valves are open. The system will tend to warm slowly under these conditions. speed the warming, stop pumping the 1K pot and open a needle valve. To when the 1K pot pressure is 1 bar open valve 31 to connect the 1K pot to the main bath and the recovery system. Admit a few ccs of helium exchange gas into the I.V.C. Close valves 8, 8A and 5, open valve 9, and slowly open valve 12 to bring the mixture back to the dumps, using the sealed rotary pump. Check that valves 1 and 1A are both open. As long as there is liquid helium in the main bath it will be very difficult to get all the mixture out. To help the boil off of the mixture energise the still heater and the mixing chamber heater to their maximum. If one does not want to wait until all mixture has come out it can be left like this while the helium in the main bath boils off completely. It is advised however to remove all the mixture before the unit has warmed to 60K. To do so open valve 13 and switch to the 9B3 booster, and pump at full speed. Check that all the mixture has been recovered by noting that the dump pressure Gl, has returned to its original value. Close all the hand valves on the dumps for security.

If wished, the O.V.C. may be softened with about lcc of helium gas to speed up the warming procedures.

Switch off the pumps, bearing in mind the usual procedures for switching off diffusion pumps. Check that the vacuum system, helium-4 bath, 1K bath and helium-3, system are all safe, particularly with respect to warming and possible build up of pressure. Switch off the helium-4 pump, ensure that the 1K pot is safe by opening a needle valve, and the bypass valve, to the main He bath (valve 31). The system should be left open to the helium recovery line, until the cryostat has warmed to room temperature.

Allow the refrigerator to reach room temperature before attempting to remove the cryostat tails.

7-1

<u>Section 8</u>

Maintenance and routine checking operations

8.1 Introduction

It is recommended to keep a good record of the operation of the refrigerator. Especially check the amount of mixture in the storage (dump) vessels by noting Gl at the beginning and end of each run.

At the beginning of each run open valves 2, 3 and 7 (these should normally be left open) and check the pressure in the dilution unit. Unless the dilution unit has been opened the pressure should be less than a few mbar. If more pressure is indicated, either there is an air leak in the system, which should be traced and cured, or the mixture was not properly removed during the close down at the end of the previous run. Any contamination should be removed via line B as described in section 5.2.

Before the start of the run the cold traps should be warmed up to 100° C and pumped to below 10^{-2} mbar as described in section 5.11.

Periodically, check for contamination of the oil mist filter. It is unlikely that this will ever require attention. The cryostat should be kept clean.

The dilution unit should be kept in a dry state, and warmed to room temperature before the tails are removed. This will prevent excessive condensation of moisture on the cold sections. None of the seals should require any attention.

The gas handling system contains several components not made by Oxford Instruments e.g. rotary pumps, vacuum gauges and valves. For these components we recommend that the manufacturers instructions for maintenance (which are sent with the dilution refrigerator) are followed.

8.2 <u>Use of the Cryopump</u>

A small cryopump is supplied with the system, and it should be used to remove the mixture left in the pipes between valves 5, 8, 8A and the sealed rotary pump if maintenance work is to be carried out on this part of the system.

The flexible line of the cryopump should be connected to "vent 1" on the helium 3 panel, and the line should be evacuated through line "B" and valves 10 and 11. Valves 2, 7, 8 8A, 10 and the individual dump valves should all be closed. Lower the cryopump into a liquid helium storage dewar and open the "Hoke" valve, then open valves 11, 6, 5 and 9. When the pressure on G2 has dropped to a steady value, valve 9 and the Hoke valve should be closed, and the cryopump should be lifted out of the storage vessel. Open the dump valves and slowly open the Hoke valve. When the pressure has reached equilibrium, close the dump valves and repeat the process until all the mixture has been removed from the pump, and stored safely in the dump vessels. Then use the cryopump to store the mixture which remains in the pipework. 01

IT IS IMPORTANT THAT THE CRYOPUMP IS ONLY USED TO STORE THE SMALL AMOUNT OF MISTURE WHICH WAS IN THE PIPEWORK. IF TOO MUCH MIXTURE IS PUMPED INTO THE CRYOPUMP, IT IS POSSIBLE TO DEVELOP DANGEROUSLY HIGH PRESSURES.

When the mixture is allowed back into the system it is important to make sure that the high pressure "Hoke" valve is only opened slowly, and that the other valves i.e. 6, 7 and 11 are already open. In this way the only part of the pipework which is subjected to a high gas pressure is within the cryopump, which is designed for this purpose. The other seals in the the system are only reliable for vacuum operation.

Security, safety and trouble shooting

9.1 Active security systems

As indicated in section 4 the helium-3 gas handling system is fitted with several sensors which, when triggered, will stop the helium-3 circulation and allow the system to warm up safely. For that purpose an electrical bypass valve is mounted parallel to valve 2, between the condenser line and the still pumping line.

This valve is "normally open" and is activated to close during running of the dilution refrigerator, i.e. when the power is switched on and the cooling water is running.

In table 9.1 we have summarised the status of the different pumps and valves after one of the sensors has been triggered.

When a fault indicator is illuminiated the fault should be rectified immediately. If one of the pumps fails it will normally blow a fuse first but is is most likely to be a failure associated with the pump or its motor. This should be checked before resetting the refrigerator. The "N₂ level low" indicator will light up if nitrogen level in the cold trap dewar is too low and no other actions are triggered. Refill immediately.

ACTION TABLE OF SAFETY SENSORS

	Booster Pump	QSB	rotary	bypass
AC power fail	off	closed	off	open
Overpressure G4	off	closed	off	open
Water fail	off	off	on	open
³ He rotary fail	off	closed	off	open
Nitrogen	on	open	on	closed
Booster Overheat	off	closed	on	closed
Booster fail	off	closed	on	closed

*

Table 9.1

-)

•

9,2

9.2 Passive security systems

The 1.2K pot pumping circuit

This circuit is protected in the event of pressure rise by an overpressure relief valve fitted to the pumping line within the cabinet. A second pressure relief valve is fitted on the cryostat top plate, connected to the 1K pot by an independent line. This means that even if the pot pumping line should block for any reason, the pot itself can vent safely.

The liquid nitrogen jacket

A dangerous state could arise if the nitrogen ports on the cryostat become blocked with solid air. In order to avoid this situation, check that all ports are free during each nitrogen transfer. The one of the four nitrogen ports. If not then during normal running to each nitrogen bush. One of these tubes should be closed with a atmospheres which are excessively humid, all tubes should be closed with a sharp knife, thus allowing for the normal nitrogen boil off.

During helium transfers the nitrogen boil off may temporarily fall to almost zero. Therefore we advise to check 10 or 15 minutes after the transfer that the nitrogen is boiling off again and none of the ports are blocked.

The main dewar vacuum space (0, V.C.)

This is protected by means of an integral vacuum/vent valve. The main helium reservoir of the cryostat has been supplied with aluminium strengthening rings. These rings will prevent accidental collapse of the reservoir under a pressure difference of one atmosphere. However, it is recommended that the helium reservoir should not be pumped unless there is a vacuum in the O.V.C.

9.3 Action after automatic shutdown

Apart from pump failures or interruption in power or water supply, an automatic shut down will be triggered by too high a backing pressure of the helium-3 rotary pump. This is indicated on G4 which triggers the safety circuit when pressure rises above 950 mbar.

Before pressing the reset button, switch off the helium-3 rotary pump. Close valves 12 and 13, and the Q.S.B. valve.

A high G4 can be caused by either.

 <u>IK pot empty</u>. The pirani gauge P4 will indicate a pressure below 0.1 mbar. When the IK pot warms to above 3K the helium-3 will not condense and the pressure rises, triggering the shut down.

Action : Refill the 1K pot using one of the needle valves and restart the refrigerator as described in section 5.8.

 There is a blockage in the return line. This can be in the nitrogen cold trap, the helium cold trap, or in the flow impedance in the dilution unit (the capillary).

Action : Locate the blockage and remedy it, as described below.

Checking the nitrogen cold trap

Make sure that values 12, 13 and the Q.S.B. are closed. Start the helium-3 pump and by cracking value 12 open slowly start to circulate again. If a pressure difference develops quickly between G4 and G3 (the condenser pressure), the nitrogen trap is blocked. Close value 12. Close the trap which is in use and open the other one. Crack value 12 again and start circulation. Blocked traps should be cleared as described in section 5.11.

Checking the helium cold trap

Start to circulate slowly as described above. No pressure difference should develop between G4 and G3 but the bath boil-off will rise quickly. Stop circulation and switch over to another helium trap by closing and opening valves 3 or 3A, and the corresponding valves on top of the cold trap themselves. Restart circulation. If the pressures on G4 and G3 start to fall to normal pressures continue the circulation otherwise the blockage is in the dilution unit.

A blocked helium cold trap should be removed from the dewar and cleaned as described in section 5.11.

Capillary blocked

If the blockage is not in the cold traps but in the dilution unit it is most likely to be the flow impedance below the 1K pot condenser. The system will have to be warmed up. Sometimes the blockage will disappear at around 80 or 90K. If not the system should be warmed completely and carefully checked for leaks of air and water which could have caused the blockage.

Another possible source of contamination is sometimes the vapour booster pump. If the temperature has become too high because of a fault in the electrical circuitry or because there was not enough oil in the pump, the oil may overheat and start to crack releasing hydrogen. This will condense in the dilution unit and eventually block the capillary. A careful examination of pumps instruments.

9.4 Loss of base temperature

Leak detection must always be performed with a helium leak detector. Merely watching for pressure rises on a Penning gauge is

Providing the refrigerator has no touches, vacuum leaks or additional heat leaks, and the capillary has the correct throughput, the system should operate successfully. At the end of a run it is advised that Gl is read and recorded, to check that no losses or gains are made to the mixture. Operation of the helium-3 circuit above 1000 mbar is not recommended. The chance of losing helium-3

Losses of base temperature are caused normally by:-

- 1. Dirty thread on the sample holder or mixing chamber.
- 2. Damage to, or incomplete radiation baffles.
- 3. Wrong circulation rate.
- 4. Wrong helium-3/helium-4 mixture.
- Heat leaks caused by additional wiring not properly thermally anchored at 4.2K and 1.2K.
- 6. Wrong capillary throughput.

7. Bad sample mounting on the sample rod, or superconducting joints between the sample and the mixing chamber. (Note: even certain types of silver solder become superconducting below about 50 mK). Joints should be either copper/copper screwed or solder joints exposed to magnetic fields sufficiently high to suppress

To help finding and curing a fault, a short list of common faults is presented in the next section.

9.5 Fault finding

This is a summary of common faults and what to do to cure each one. If the problem cannot be solved and another operational fault is suspected a full set of test data should be produced and sent to Oxford Instruments for diagnosis.

1) Cold point in wrong Too little helium-3 (or place. too much - unlikely). Dilution unit will not cool further in the single shot mode. 2) Still warms drastically Still empty - too little on applying about 1 mW, helium-4. See sympton 1 as also low circulation rate. to whether more $\hat{h}e\hat{l}ium-3$ is required. 3) Circulation rate and 1.2K condenser not condenser pressure operating efficiently remain very high. can be confirmed by measuring temperature of return helium-3 as a function of circulation rate and comparing it with the pot temperature. 4) Low circulation rate Capillary impedance too and high condenser high - check room pressure with slow temperature and 77K cooling. throughputs and re-adjust as necessary. 5) Poor percentage of See symptons 1 and 2. helium-3 circulated. Check film burner current is correct. Check dilute side flow impedance, having first removed mixing chamber base. 6) 77K leak on dilution Check all indium seals are unit. tight. Seals should not require replacing. Poor base temperature. Check for heat leaks from radiation, wiring, vibration, residual exchange gas. Plot base temperature as a function of circulation rate, allowing approximately 3 hours for each point. Perform single shot and watch for further cooling. If no effect, check symptoms 1 to 6. Finally

check thermal contact and

temperature thermometer.

operation of low

- 8) Oscillations in still pressure which do not disappear after several hours.
- 9) Oscillations in condenser pressure.

Level of liquid in still rises as condenser pressure falls - remove some helium-4.

,

Check symptom 8 and sympton 3.

action 10

<u>section 10</u>

<u>pilution Refrigerator Test Results</u> Magnet Switch Heaters Table 10.1 <u>Resistor values</u> (including leads) (ohms) Maynel \$10 910 leads RICION 1) R2 R3 R5 R6 Film Still Cold External Burner Plate Mixing Chamber 717-710 (1996) 703 740 360 R.T. 20.3 118 116 488 763 790 780 6 80 796 841 354 374 18:5 319 319 77K 490 1240 1180 1240 380 4.2K 6 Z O 107 109 Base 2530 2800 20.4K 6330 Temp 7100K Magnet Fisa Cooling power results Table 10.2 LN2 Jun- 97 Temp Cooling power flow rate still power film burner $(\mathbf{m}\mathbf{R})$ (microwatt) micromol/sec (mW) (mW) 9.5 Û S 0 0 0 Base temperature in sample position 9.9 mK (at 150 micromoles/sec) SEE. BACK 4.-Room temperature capillary throughput 27-28 (AFTER REPAIR 12 mbar/min IK pot needle valve throughput - mbar/min M.C. Mixture volume (at approx 16% conc.) 149 lts Dump volume 200 lts Liquid nitrogen bath lifetime >48 lts Liquid helium bath lifetime in >48 hrs (approx) static mode Helium boil off with fridge operational 243 cc/hr (approx) Cooling water flow 3 1/min Compressed air requirement 3 bar CAPILLARY FLOW @ 77 % 750 mBAR PRESS = 90 m BAF INCREASE/MIN. WITH min 18,400 r PV=nRT

đ

Ma

Dilution Refrigerator Electrical Wiring

The dilution unit is equipped with several diagnostic carbon resistor thermometers. These have been positioned on the still, the cold plate and the mixing chamber.

From room temperature down to the 1.2K pot level, these resistors are wired using 42 s.w.g constantan wire. This wiring has been thermally dumped at 4.2K and at 1.2K, before being soldered to the flat connectors on the underside of 1K pot.

11.1 Insert and dilution unit

500 ohm nominal resistance constantan heaters are fitted to the film burner, the still and the mixing chamber. 40 swg copper wire has been used to wire the heaters on the film burner and the still, but the mixing chamber heater is terminated below 1.2K with superconducting wire. The electrical wiring to the dilution unit is demountable via a 20 way "Radio spares" flat pack connector also terminated at 1.2K on 20 way "Radio spares" connectors, after being dumped as described earlier. (These connectors are also

The solid co-axial lines are terminated in RADIALL subminiature series co-axial plugs. These are compatible with other manufacturers miniature co-axial connectors. The part number of the mating connectors is R 114 003.

wire from : California fine wire company Erover eity (305) 489-5144 The 3 Fischer connectors situated at room temperature are labelled 1-3 on their respective housings. 3 "flat pack" 20 way connectors are stacked on the base of the 1.2K pot. The wiring from Fischer connector number 1 is wired to the 20 way connector closest to the The wiring from the other Fischer connectors proceeds logically, connector number 4 being wired to the flat pack connector Fischer connector number 1 This connector is used for the dilution unit wiring via the top Film burner 1 **R1** 2 Still R2 Cold plate 3 R3 54 Mixing chamber 65 Mixing chamber R4 R54 or Oxford controller 6 4 is Nic 7 Common line for resistors 8 9 10 11 12 13) Film burner heater (500 ohm nom.) 14) 15) Still heater (500 ohm nom.) 16) 17) Mixing chamber heater (500 ohm nom.) 18) outside messures x 700 r Lines 1-10 are wire in constantan, 11-18 in copper, to the 1K pot. Fr Country - Conector # 2 is all constant ant. Connector # 3 is all copper writing All leads on the other Fisher connectors (2-4) are spare for \dot{C} ಶ Y. $9^{+}...$ A 9 b 2 ß С 11,2

11.2 Pumping System

A full set on electrical diagrams of the gas handling system is included in the manual.

.

FUSE DETAIL

 \bigcirc

O

28677

			20011
NO	FUNCTION	FUSE	FUSE HOLDER
14 15 16 $-$ 1 8 H 9 H 0 F 1 M 2 B 0 F 1 M 2 D 1 C 0 C 0 D 1 C 0 C 0 D 1 C 0 C 0 H 1 C 0 C 0 H 1 C 0 C 0 H 1 C 0	MAINS IN RED Ø MAINS IN YELLOW Ø MAINS IN BLUE Ø COIL C1 PHASE DET. RED PHASE DET. YELLOW PHASE DET. BLUE TX PRIMARY TX SECONDARY GAUGES L2 LOOP COIL C2 HE ³ ROT HE ³ ROT HE ³ ROT HE ³ ROT HE ³ ROT HE ³ ROT COIL C3 BOOSTER BLUE Ø BOOSTER BLUE Ø BOOSTER BLUE Ø BOOSTER BLUE Ø BOOSTER BLUE Ø COIL C4 DT 'A' PUMP DT 'A' PUMP DT 'A' PUMP DT 'A' PUMP DIL C5 FF PUMP IL RL 13-2 IL C6 4 ROT 4 ROT	30 A TIA 30 A TIA 30 A TIA 30 A TIA 1 A HBC 60 M/A QB 60 M/A QB 60 M/A QB 1 A HBC 3 A HBC 2 A HBC 3 A HBC 2 A HBC 5 A HBC 750 M/A T 16 A NIT 16 A NIT 16 A NIT 16 A NIT 10 A NIT 10 A NIT 100 M/A T 100 M/A T 100 M/A T 10 A NIT 10 A NIT	RS 32 P RS 32 P RS 32 P PANEL MTG 1 [‡] " PANEL MTG 1 [‡] " PANEL MTG 1 [‡] " PANEL MTG 1 [‡] " PANEL MTG 1 [‡] " SAKS SAKS SAKS SAKS SAKS RS 20 P RS 20 P RS 20 P RS 20 P RS 20 P RS 20 P RS 20 P SAKS SAKS SAKS SAKS SAKS SAKS SAKS SAK

