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# Fridge Manual: Screen Room W061 Dilution Fridge Operation

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July 15, 2021

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# Introduction

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## 1.1 Description of the Fridge

The fridge is an old 1986 Oxford Instruments 'wet' helium dilution refrigerator, it has six stages of cooling with defined 'plates' of decreasing temperature to anchor to, these are:

1. 300 K - Room Temperature Plate (RT)
2. 4.2 K - Liquid Helium Bath (4K)
3. 1.4 K - 1K pot (1K)
4. 0.8 K - Still Plate (Still)
5. 100 mK - Heat Exchangers (HX)
6. 25 mK - Mixing Chamber (MC) and Experiment (EX)

The fridge has been modified significantly since instillation. The stages EX - 4K are held within the internal vacuum can (IVC) with the 4K plate forming the top of this vacuum can. The IVC is then immersed in a volume of helium liquid or 'bath' which keeps the outside of the IVC at 4K. The IVC is connected through the bath to the fridge 'top plate' (RT) via a series of stainless steel (SS) tubes that contain everything needed for cooling, measurement and control within the IVC. The helium bath is contained within a super-insulating dewar or outer vacuum can (OVC), which covers the IVC and forms a seal with the fridge top plate. When running or 'closed' this OVC hangs centrally within the first screen room in W061, with pumping lines and electronics feeding in from above. When the fridge is 'open' the OVC is stored directly below the dilution unit of the fridge in a pit below the floor. The fridge does not have a nitrogen jacket, so when running only the helium bath must be refilled to keep it above the level of the 1K pot intake.

Table 1.1 below shows a list of key values for normal operation of the fridge, as a quick reference. More details for running will be in later chapters, and description of the pumping and control setup of the fridge will be in section 1.3. All valves and gauges will have unique names, most of which can be found on the diagrams in section 1.3. This manual will only contain information relevant to fridge control, see other reports for details of the RF measurement setup [1].

**Table 1.1:** Important Values for Normal Running of Fridge

Item	Shorthand	Value	Unit
Base Temp	$T_{base}$	25	$mK$
1K Pot Temp	$T_{1K}$	1.4	$K$
1K Pot Pick-up Depth	$LvL_{min}$	375	$mm$
Bath Max Level	$LvL_{max}$	1125	$mm$
Still Power	$C_{Still}$	10	$\%$
Turbo Current	$C_{Turbo}$	1-2.5	$A$
Turbo Pressure	P1	$8 \times 10^{-3}$	$mbar$
1K Pot Pressure	G6	2.5-3	$Torr$
Dump Pressures	$P_{D1} + P_{D2}$	830	$mbar$
IVC Pressure	$P_{IVC}$	$1 \times 10^{-6}$	$mbar$

## 1.2 Known Problems with Operation

The list below is of know issues with the operation of the fridge, many from the age of the fridge and extensive modifications. These are issues that have yet to be resolved or are not practical to resolve, none are significant enough to prevent running of the fridge or create safety issues. If significant work arounds are required they will be mentioned in the relevant sections for running.

- Leak through valve 13, gas in 'back side' (between valves 12 / 13 / S\* and valve 8 / 8A, via G4 1.1) of  $^3\text{He}$  circuit will flow into fridge. Leak is small but  $mbar$  of pressure will build up over days/weeks.
- Leak from atmosphere into fridge. This leak is small and behind the  $\text{N}_2$  cold traps, so the leak can be largely ignored. However more gas than expected will be frozen into the cold trap, which can easily overpressure the cold trap if warmed up. To stop this the cold trap should be pumped on through the B system when removed from  $\text{LN}_2$ .
- O-rings deteriorated on B system pumping station. The B system diffusion pumping station is unreliable and leaks are frequent. The Fridge has been isolated from this system, so that the cold traps can still be pumped out but using a pumping trolley through the 'vent' on the main  $^3\text{He}$  circuit.
- Pressure gauges are not zeroed / accurate. All operating values given will be gauge dependent and not absolute. Only P1 or a pumping trolley should be used to determine whether there is good vacuum in any part of the  $^3\text{He}$  circuit ( $< 10 mbar$ ).

- There is not enough  $^3\text{He}$  in the mix, if running in single shot mode the still empties very quickly. This may be limiting the base temperature / cooling power, but cooling to  $25\text{ mK}$  is still possible.
- The 4K plate can quickly rise above 4.2K once the bath level is below this plate, causing NbTi coax to become normal under microwave signal. A few things have been added to mitigate this: a copper braid attached to the 4K plate hangs down into the bath, some of the holes in the bath baffles have been covered to impede the flow of gas out, and a small heat leak has been added between the 1K pot and hot end of the NbTi. This seems to eliminate the problem but keeping the  $^4\text{He}$  level high will also prevent this.
- The adjustable end of the LHS cable for raising the dewar is stuck. This end should be set to a level such that only the RHS cable need adjusting. The LHS can be adjusted when not attached to the dewar.
- It is unknown how effective the air cushions for vibrational isolation of the fridge are.  $25\text{ mK}$  is still achievable and vibrational isolation has not been a problem yet.
- The  $^4\text{He}$  fill line gas handling system has leaks and there is not a good enough nitrogen trap to stay cold overnight. This requires a solution as it is dangerous to leave experimental cells, that are full of liquid helium, either close or open to something that is leaking air in.

### 1.3 Diagrams and Photos of Fridge Setup

Figure 1.1 shows the  $^3\text{He}$  dilution gas handling system for the fridge. Not shown are the  $^4\text{He}$  bath cold trap and dilution unit within the fridge, this figure shows the rt sections of the circuit outside of the OVC. Shown in red is the circuit taken by the  $^3\text{He}$  in normal circulation mode, shown in grey is the OVC with condensing and return side labelled. The main pumping force is provided by a large turbo pump (housed behind the  $^3\text{He}$  gas handling board) backed by the  $^3\text{He}$  rotary (located in the pit). There are two cold traps to allow cleaning of one whilst the other is used in circulation, this allows continuous running for long periods. Valve 5 is left open along with the dumps during circulation in case of a blockage within the fridge. When the pressure on G4 exceeds  $800\text{ mbar}$  the power is cut to both pumps and the bypass opens allowing gas to flow from the condenser side of the circuit into the dump. There is no bypass on the back side of the circuit as the running pressure is low enough and volume is large enough to take the mix.

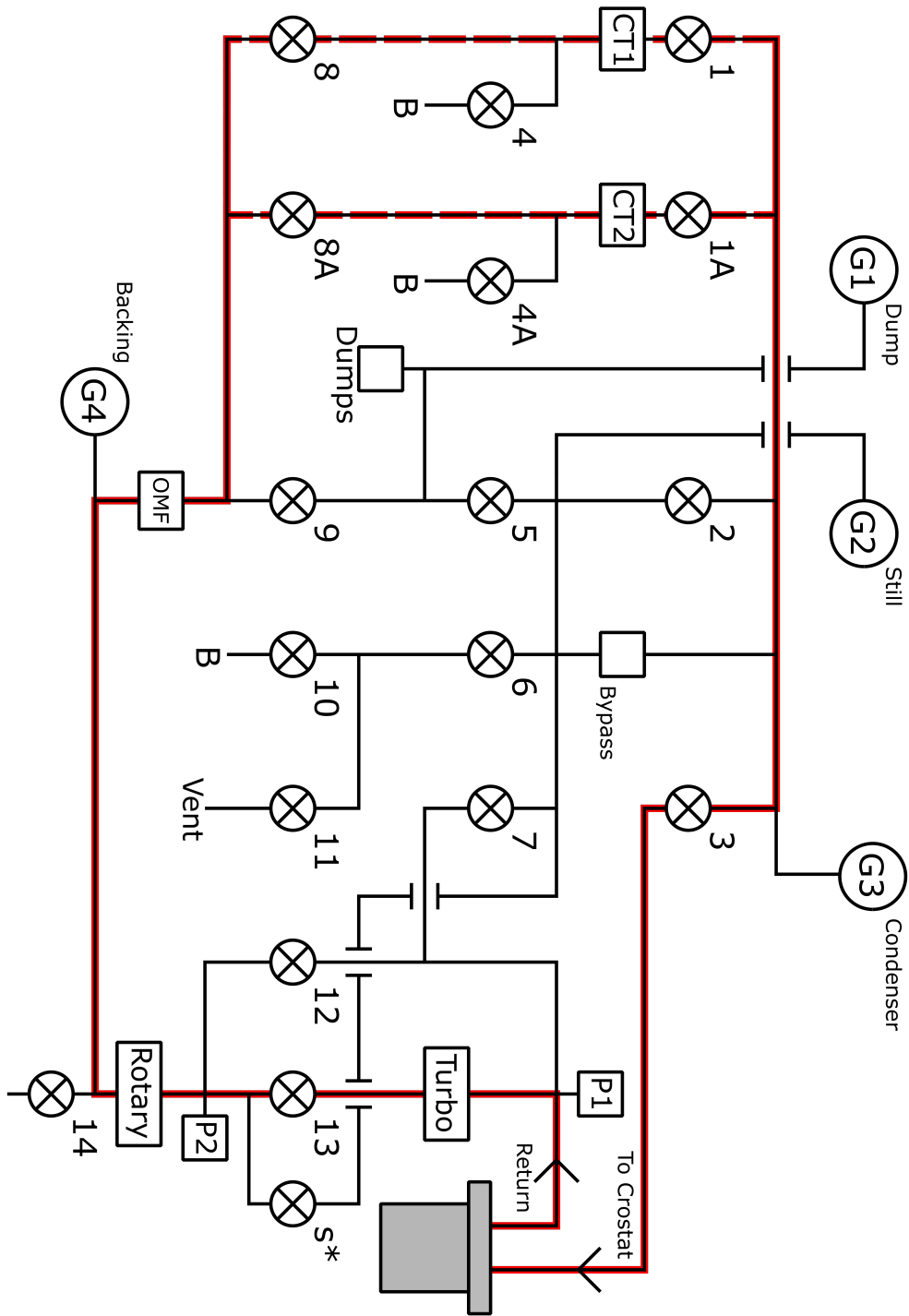
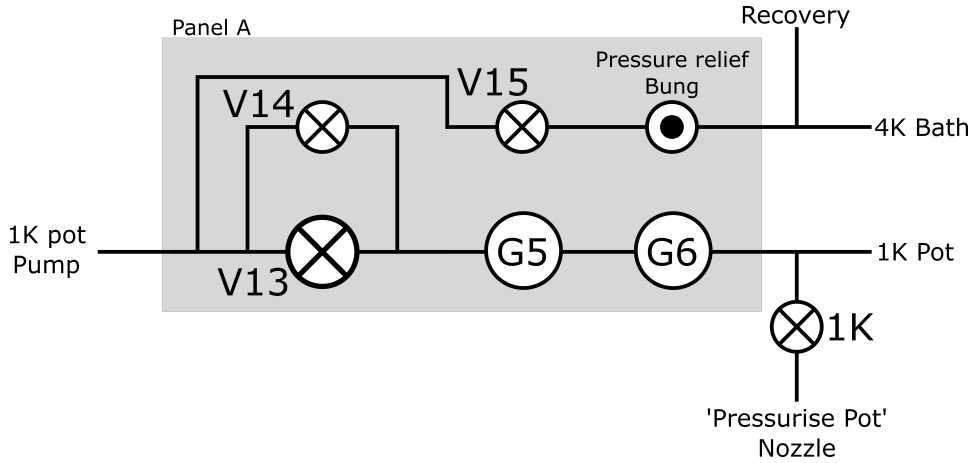


Figure 1.1:  $^3\text{He}$  pumping system, inside of fridge not shown.

G1-4 are pressure gauges from 0 – 1000 mbar. P1 is the pressure in front of

the turbo and measures pressure down to **pressure** and P2 is the pressure behind the turbo / in front of the  $^3\text{He}$  rotary and measures pressure down to **pressure**. The 'vent' labelled section ends in an external KF flange allowing a pumping trolley to be attached. This allows the pumping out of any air in the system and the cleaning of the cold traps through the connected 'B' system. All valves bar 14 are found on the main  $^3\text{He}$  gas handling board along with all pressure gauges. There are two dumps to help extraction of all mix and to avoid overpressure within the system.



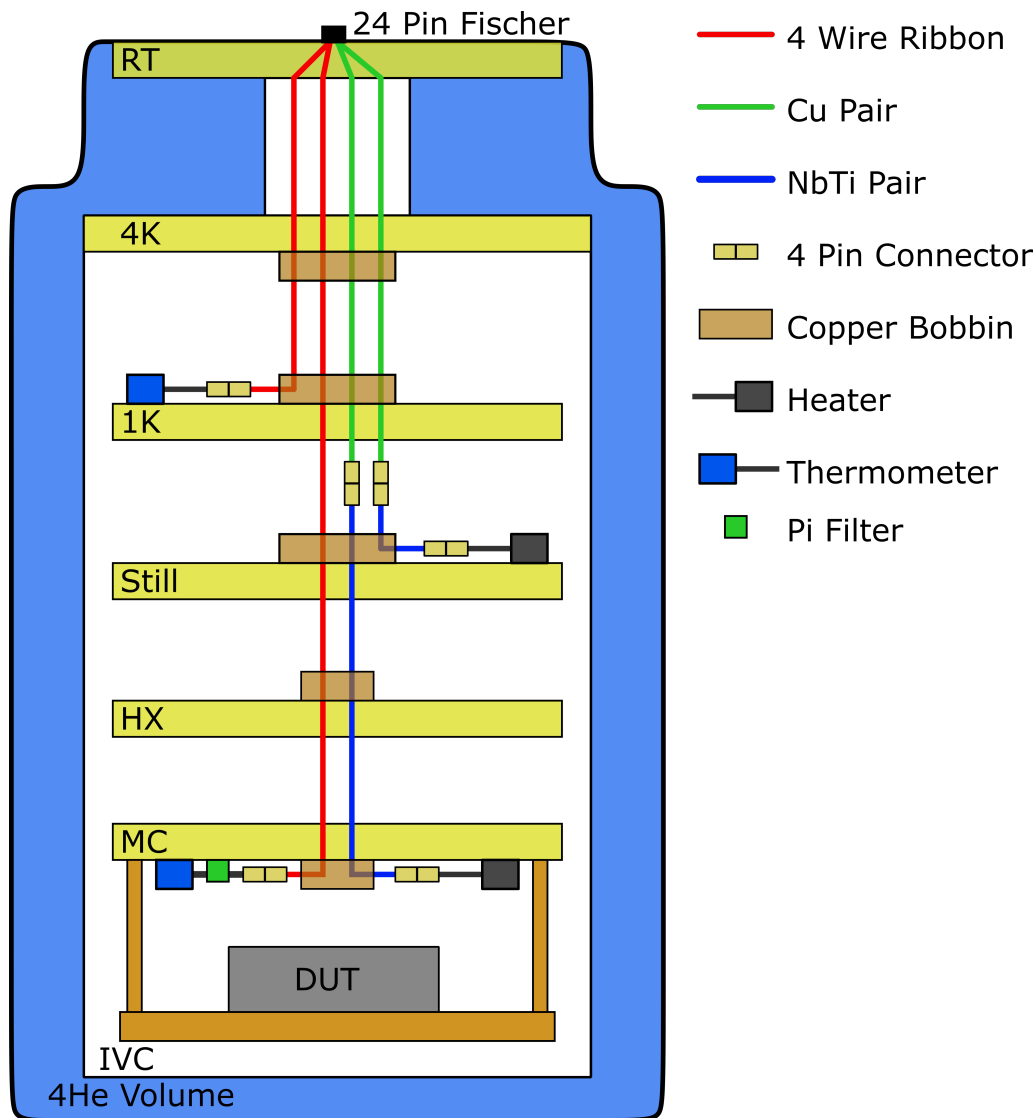
**Figure 1.2:** 1K pot pumping system, which is linked to the 4K helium bath.

Figure 1.2 shows the gas handling system for the 1K post pumping line outside the OVC. It also shows a section of the  $^4\text{He}$  recovery system, this allows recycling of the  $^4\text{He}$  used for the 4K bath and 1K pot. The bath is connected to the pressure relief bung at all times which prevents overpressure at around 1.5 bar, though this safety feature is mainly present to protect the OVC when pressuring the bath to blow out  $\text{LN}_2$ . G5 is the rough 1K pot gauge from 0 – 1000 mbar whilst G6 is the fine gauge from 25 – 0 Torr. The 1K pot pump is housed beneath the fridge in the pit, and the recovery leads through gas meters and to the cryogenics facility where the  $^4\text{He}$  is re-liquefied.

Figure 1.3 shows the wiring of all thermometers and heaters within the IVC. There are currently only two thermometers and two heaters. The heaters are 1  $K\Omega$  resistors located at the Still and MC plates and wired with 44 SWG / 81  $\mu\text{m}$  diameter copper twisted pairs from rt to 1K then with superconducting NbTi (100  $\mu\text{m}$  core, CuNi insulation) twisted pairs to short copper twisted pairs soldered directly to the resistors. The thermometers are located at the 1K and MC stages; A *Cernox CX-1030-AA-0.3L* thermometer purchased already calibrated from rt down to 300 mK, is located on the 1K plate; and a Ruthenium Oxide *RX-102B-CB* thermometer calibrated down to 8 mK (against a noise thermometer on ND2), is located on the MC plate. Both thermometers are wired from rt to their respective plates with 4 wire 36 AWG phosphor bronze ribbons, transitioning to their factory wiring close to the thermometer. The MC ther-



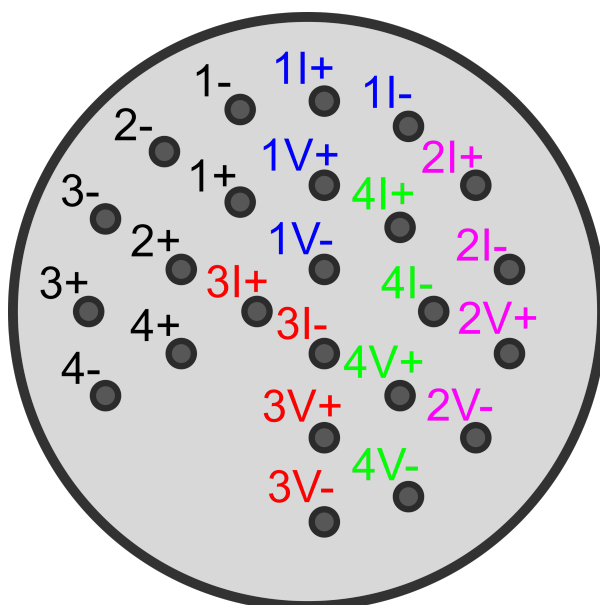
mometer line also has a (details) pi filter (low pass), thermalised to the MC, to reduce RF heating directly on the thermometer. Transitions between different wire types have been achieved with small 4 pin plugs to allow easy modification. All control wiring is thermalised at each plate with 10+ turns around copper bobbins and coated in GE varnish, using a small piece of rizla between the wires and copper bobbin to help electrical isolation. Dental floss and kapton tape have been used to secure wiring to the fridge structure within the IVC.



**Figure 1.3:** Control wiring for fridge showing both thermometers and heaters. Turns around the copper bobbins are not shown, all wires are thermalised to the same bobbin at each stage.

The figures 1.4, 1.5 and 1.6 show wiring schemes. Figure 1.4 shows the 24 pin

Fischer cable wiring scheme, shown is the view into the male cable, this also corresponds to the same orientation as the back of the 24 pin vacuum Fischer connector, providing the transition into the IVC through the rt plate. The thermometers 3(1K) and 4(MC) are used along with resistors 2(Still) and 4(MC). The connection between the 24 pin Fischer and scanner box of the *Lakeshore 372 AC Resistance Bridge and Temperature Controller*, is provides by a 12X individually shielded twisted pair cable. One end of this cable is the male 24 pin Fischer connector, the other is split into the 25 pin male scanner connector (Figure 1.5 for female), and 4 individual twisted pairs for the heaters. Figure 1.5 shows the scanner input wiring scheme, matching wiring in Figure 1.3. Shields are soldered from the individual twisted pairs, and twisted pairs are formed from the positive and negative of matching measurement lines (e.g. 3V+ and 3V-).



**Figure 1.4:** 24 pin Fischer connector wiring, as looking into male cable, female vacuum Fischer on rt plate will be mirror image, or same from back where fridge wiring is soldered to.

Black numbers are heater wires and coloured are thermometers, numbers refer to which device, I and V are current and voltage, while + and - are positive and negative connections.

Figure 1.6 shows the wiring scheme for the 4 pin connectors (*CMD Direct, 4-way connector for cryogenic use*) used for transitions within the IVC. The figure is labelled for one of the thermometers. These connectors are small enough so as to not take up too much space in the fridge, whilst providing robust connections and the ability to easily swap out thermometers / heaters between runs. For all wiring standard multi-core lead solder is used along with *Future 315 No Clean Flux*, for soldering into buckets this provided easy wetting with reduced risk of corrosion from flux residue.

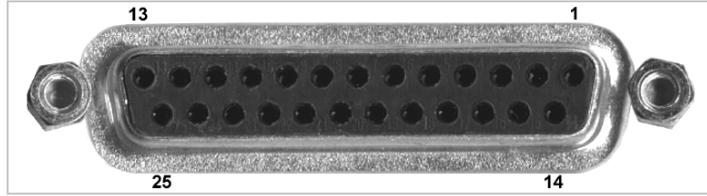
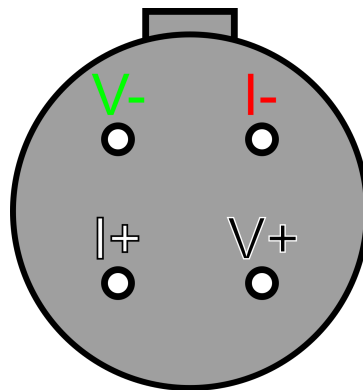


FIGURE 3-9 Scanner input connector

Pin	Symbol	Pin	Symbol
1	Shield	14	Shield
2	Shield	15	1I-
3	1I+	16	1V-
4	1V+	17	Shield
5	Shield	18	2I-
6	2I+	19	2V-
7	2V+	20	Shield
8	Shield	21	3I-
9	3I+	22	3V-
10	3V+	23	Shield
11	Shield	24	4I-
12	4I+	25	4V-
13	4V+		
Body	Shield		

Guards are not carried through the scanner. Shields are connected to isolated measurement common.

**Figure 1.5:** Lakeshore scanner input connector, pins on Fischer to scanner cable are soldered to match.



**Figure 1.6:** 4 pin female connector wiring, for wire transitions within the fridge. View from the back into which the colour coded 4 wire ribbons are soldered. Pins are also labelled with their corresponding wiring to the Lakeshore. For resistors only the top two pins are used.

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# Closing The Fridge

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## 2.1 Cleaning Cold Traps + $^3\text{He}$ Circuit

The fridge has two cold traps CT1 and CT2 (figure 1.1), both cold traps should be left under vacuum at the end of a run. However due to the leak through 13 it is likely some of the mix will have escaped the back side of the  $^3\text{He}$  circuit, and that some air has leaked into the system. Therefore it is advised to ensure the mix is all in the back side and clean before starting to run the fridge. Only one cold trap is required for this so the other can be pumped out via the 'vent' using a pumping trolley beforehand. The process for this is as follows (assuming CT1 used):

- All valves should be close, the mix should be in the dumps with some remaining in the back side.
- Fill the CT1 with  $\text{LN}_2$  open 8.
- Turn on  $^3\text{He}$  rotary and open 1,2,3,6,7,10,12,13 to circulate via 7 and pump out B (if still isolated).
- After a few minutes all air should be frozen into CT1, close 8.
- Open 4 to pump out CT1 from both sides.
- Pump mix into back side until P1 reads  $< 1 \text{ mbar}$ , close 12 and start the turbo.
- Leave for an hour or until P1 is low (details) then close all valves.
- Connect a pumping trolley via vent and begin pumping trolley rotary (ptr), open 4,10,11.
- Remove CT1 from the  $\text{LN}_2$ , the evaporation of frozen air inside can be sped up with a heat gu, though this is not necessary.
- Once pumping trolley pressure (ptp) is  $< 1E - 1 \text{ mbar}$  (and CT1 is warm) start the pumping trolley turbo (ptt). This step can be skipped if time is a concern.
- Once satisfactory pumping achieved close all valves, stop and remove pump (follow pump shut-down procedure).
- Fridge state should be all valves closed, all  $^3\text{He}$  circuit free of air and mix all in back side, with G4 reading  $\sim 180 \text{ mbar}$  (matching end of previous run). The  $^3\text{He}$  rotary should be left running to prevent the mixing leaking out of the back side.

## 2.2 Electrical Checks

Electrical checks should be done before closing the IVC. Currently only the resistances of devices connected to the Lakeshore, and cryogenic pre-amp need be checked before closing up. Depending on the microwave setup it may also be possible to check the measurement circuit, though currently this can only be tested at 77 K. The cryogenic pre-amp must be checked carefully according to its data sheet. Then the resistances of thermometers and resistors can be checked with a multimeter. Nominal values for the thermometers just before the scanner (Figure 1.5) and the heaters at the back of the Lakeshore (disconnecting first) are:

**Table 2.1:** Electrical check resistances on control wiring, heaters have been labelled according to Lakeshore setup.

From	To	Resistance ( $\Omega$ )
3I+	3V+	30.4
	3I-	68.2
	3V-	67.6
3V+	3I-	67.9
	3V-	67.4
3I-	3V-	30.8
4I+	4V+	59.6
	4I-	1069
	4V-	1068
4V+	4I-	1069
	4V-	1068
4I-	4V-	60.2
Still Heater		1150
Sample Heater (MC)		1261

## 2.3 Closing IVC

If significant changes were made within the IVC since the last run it may be worth electrical / leak checking these before closing the IVC. Changing experimental cells will require re-soldering the  $^4\text{He}$  fill line near the MC, to check this is successful the cell can be filled with  $^4\text{He}$  gas and a leak detector in 'sniff' mode can be used. If the cell is a microwave cavity the change from vacuum to  $^4\text{He}$  gas should cause a visible change in resonance. A more sensitive leak check can be carried out once the IVC is closed.

- Ensure nothing is sticking out from and plates enough to contact the inside of

the IVC, and that the IVC and 4K plate are clean where the indium seal will be formed.

- To close the IVC the circular plate directly below the fridge must be removed, along with the circular 'lid' plate covering the bath volume of the OVC. It is easiest to open the large square pit plate, remove the OVC lid, return the pit plate, then remove the circular plate from the centre of the pit plate.
- Lower the IVC through the hole to then raise it over the plates of the fridge, recovering the centre of the pit plate and using a jack the IVC can be brought up close to the 4K plate.
- Wrap a 1 mm diameter piece of indium around the groove at the top of the IVC (where the seal is formed), ensuring the indium is tight and overlapping the ends slightly where they meet.
- Raise to contact and screw the IVC to the 4K plate, ideally this is done by two people tightening the screws slowly, starting from opposite sides of the IVC and rotating around the IVC in the same direction.
- Once all screws have been tightened the IVC should be left for an hour before tightening screws once more.
- The IVC should now be leak tight and ready to pump down. Attach a turbo pump to the top of the IVC and begin pumping. The IVC can be flushed with N<sub>2</sub> or <sup>4</sup>He to speed up this pumping, a pressure below 10<sup>-4</sup> on the IVC pressure gauge (P<sub>IVC</sub>) is desirable before cooling down.

## 2.4 Pumping out OVC

Connected a pumping trolley with working penning gauge to the OVC whilst still in the pit (this can be done once the OVC is up, but the opportune time is as soon as the IVC is closed). Do **NOT** open OVC valve until pressure in line is below 10<sup>-2</sup> mbar, otherwise the pressure shock could damage the super-insulation within the dewar. The OVC maintains pressure well enough that it is safe to start the turbo pump on the pumping trolley before opening the OVC valve. Overnight pumping should yield a pressure around 5 × 10<sup>-5</sup> mbar, which is acceptable for running.

## 2.5 Leak Checks

The entire fridge circuit has been leak checked and easy to identify leaks resolved, there is likely some leaking of air into the <sup>3</sup>He circuit through old O-rings which can be ignored

for this step. Ensure when leak checking that leak detectors are appropriately set to either  $^3\text{He}$  or  $^4\text{He}$ . Leak checks that can be performed are:

- Should be turbo pumping on IVC with pumping trolley, back turbo pump on IVC with leak detector. Leak checks can be performed whilst pumping down the IVC for cooling.
- Allow small amount of mix into dilution unit, if no change on leak detector is seen then there is no leak from the dilution unit into the IVC.
- Alternatively if there are concerns of a significant leak a small volume of  $^4\text{He}$  from a cylinder can be let into the dilution unit via 'vent', 11, 6, and 7. Then pumped out via the same route using a pumping trolley.
- The experimental fill lines should be pumped out the pressurised with  $^4\text{He}$  to leak check them.
- Spray  $^4\text{He}$  around the IVC seals and around the RT top plate of the fridge to check for leaks from the bath / atmosphere into the IVC.
- Close 1K pot needle valves, pump out the 1K pot with 1K pot pump (V14+V13 open, V15+1K closed, 1K pot pump exhaust to the pit not recovery), pressurise 1K pot with cylinder through 'pressurise pot' nozzle (1K).
- Stop cylinder on 1K pot, open needle valves, and G5 should return to *Atm* if unblocked.
- The reverse of these tests can be conducted where  $^4\text{He}$  is put into the IVC, and fill lines / dilution unit are pumped on with a leak detector backed pumping trolley. Though care should be taken to isolate the mix as in Section 2.1.

## 2.6 Throughput Test

Assuming the mix is all in the back side and the fridge circuit is clean and dumps are closed, open 3, G3 should remain steady. Then open 9, 5 and 7 to allow this mix into the still side of the dilution unit. A pressure change should be seen in G3 on the condenser side showing that the impedances are not blocked. The mix can then be returned to the back side via pumping as in Section 2.1.

## 2.7 Raising Dewar

The dewar (OVC) must be raised using the steel cable assembly and counter weight. The counterweight is balanced with the dewar, however it is safest to have one person holding the dewar when trying to move it. There are two sets of screws for attaching the dewar: smaller screws which tighten the o-ring seal with the RT plate, and larger ones that attach to the frame which hold the weight of both the dewar and IVC. The pit should be open underneath the IVC, the OVC lid should be removed, and the OVC should be pumped down with nothing attached to its valve. The procedure is then:

- Check that the dewar o-ring and underside of the RT plate are clean, the dewar o-ring can be cleaned and re-greased at this stage.
- Attach the copper braid to the 4K plate then tape it and anything else loose to the IVC.
- Free both ends of the dewar raising cables, check that the cables are in the pulley system and not caught on anything.
- Attach the cables to the dewar loops, the RHS cable may need to be adjusted slightly.
- Once attached tighten then RHS adjustable end, taking the weight of the dewar.
- Once the weight of the dewar is taken by the counterweights remove the stopper rod from the counterweights.
- Slowly raise the dewar by hand being careful not to impact the IVC, baffles or RT plate. Stop just as the o-ring contacts the RT plate.
- Line up the RT plate screws with dewar and thread the screws but do not tighten.
- Line up and then screw on the heavy bolts to the frame, take the weight of the dewar with these but do not tighten.
- Let slack into the steel cables and remove the RT plate pillars from the top bar. So the weight of the dilution unit is entirely taken by the dewar which in turn is taken by the frame.
- Tighten both sets of bolts. The bath should now be relatively air tight and the dilution unit should be isolated from vibrations.



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# N<sub>2</sub> Cooling to 77 K

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## 3.1 Ready State For Cooling

The dewar should up and sealed over the top of the IVC, the IVC should be pumped down to below  $1 \times 10^4$  *mbar*, and the bath should be disconnected from the recovery. The <sup>3</sup>He circuit should be clean and the mix in the back side, valves on the circuit should be closed and the rotary left on. The process from here is:

- Let some exchange gas into the IVC. This can be done carefully by blowing <sup>4</sup>He from a cylinder into the IVC valve on top of the fridge, closing the volume with a palm and opening the valve. Doing this twice should provide enough exchange gas, roughly 4 *mbar* at RT and 1 *mbar* at 77 K. It is safest to do this now as multiple <sup>4</sup>He supplies may not be available and the 1K pot must be pressurised while N<sub>2</sub> is in the bath.
- Pump out the 1K pot through V13 and V14 (Figure 1.2). Ensure V15 and both needle valves closed, with the 1K pot pump's exhaust is to the pit.
- Remove the 'pressure relief bung' (Figure 1.2) to open the bath volume to atmosphere.
- Close V13 and V14, connect <sup>4</sup>He cylinder to '1K' valve and overpressure by  $\sim 200$  *mbar*.
- Open both needle valves **half** a turn to start blowing <sup>4</sup>He through the 1K pot and into the bath. This prevents air freezing and blocking the pot input and also the needle valves locking up due to thermal contraction.
- G6 should continue to read 1200 *mbar* if not increase the cylinder pressure.

## 3.2 Transfer N<sub>2</sub> In

It is now safe to transfer the LN<sub>2</sub> into the bath, it is probably a good idea to turn on the screen room extractor fan on while transferring. The process is then:

- Open and insert the N<sub>2</sub> transfer SS tube through one of the transfer ports on the RT plate, the tube should hit the bottom of the dewar then be raised up a few centimetres and locked in place.

- Attach the output of a N<sub>2</sub>dewar to the exposed end of the N<sub>2</sub>transfer tube with orange tubing.
- Pressurise the N<sub>2</sub>dewar with dry air by  $\sim 250$  mbar.
- The N<sub>2</sub>should be pushed through the orange tubing into the fridge, you should be able to see boil off through the pressure relief bung port.
- A 50 L N<sub>2</sub>dewar will be enough to cool and keep cool the fridge to 77 K overnight. It may also be possible to fill the bath with N<sub>2</sub>in the morning and have time to let it cool, blown it out, and start <sup>4</sup>He transfer in the same day.
- If leaving overnight replace pressure relief bung and attach one way valve to a bath port on the RT plate. One can be made from a short length of orange rubber tubing with a bung at one end by cutting a slit into the side of the tube with a razor blade.

If there are significant concerns it may be worth leak checking at 77 K, if not it is probably best to do this when pumping out the exchange gas.

### 3.3 Blown N<sub>2</sub> Out

Once the thermometers inside the IVC read  $\sim 77$  K the N<sub>2</sub>can be blown back out. Note this should be done just before transferring in <sup>4</sup>He. The Process is:

- Replace pressure relief bung.
- Open empty N<sub>2</sub>dewar (use same as before).
- Attach orange rubber tubing to end of N<sub>2</sub>transfer tube, have other end loose.
- Insert transfer tube into transfer port, once it hits the bottom of the dewar raise it up  $\sim 10$  cm.
- Use a small SS tube to pressurise the dewar with dry air from supply. Only use  $\sim 250$  mbar as the dewar is not designed for pressure within the bath.
- Insert the other end of the orange tube, attached to transfer tube, into the N<sub>2</sub>dewar, hold this with a glove while it freezes in place such that there are no harsh corners and the orange tubing is not deep inside the N<sub>2</sub>dewar.
- Once the N<sub>2</sub>level has dipped below the end of the transfer tube the orange tubing will begin to unfreeze. When this happens fully insert the tube until it is  $\sim 1$  cm from the bottom, then switch from pressurising the dewar with dry air to <sup>4</sup>He from the supply.

- Once the orange tubing begins to unfreeze again all the N<sub>2</sub> should be blown out, this can be checked by lowering the transfer tube to touch the bottom. The temperature inside the IVC should also be rising. Remove the transfer tube and tube used to pressurise, ensuring transfer ports are closed.
- The fridge is ready for <sup>4</sup>He transfer.

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# $^4\text{He}$ Cooling to 4.2 K

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## 4.1 First $^4\text{He}$ Transfer

The bath should now be empty and the thermometers should read  $\sim 80\text{ K}$  although something around  $100\text{ K}$  is okay. If the  $^4\text{He}$  transfer tubes have not been pumped out in some time this should be done first, use a pumping trolley, run the turbo for  $\sim 30\text{ mins}$ . The process for the first transfer is:

- Ensure the bath is fully closed then open the  $^4\text{He}$  recovery. There are two large orange valve handles. The higher symmetric is open when parallel to pipe and the lower asymmetric one is 'pointing' away from the handle, so should be pointing towards the recovery gas meter.
- Ensure the 1K pot pump's exhaust is set to the recovery and not pit.
- Collect a helium dewar from cryogenics facility.
- Fully insert the long transfer tube into the fridge through a transfer port to  $\sim 5\text{ cm}$  above the bottom of the dewar. Insert the transfer tube slowly to stop the air in it freezing and blocking the bottom.
- Close the dewar to the recovery, insert the shorter transfer tube with flexible end into the helium dewar slowly until  $^4\text{He}$  gas is heard blowing out the other end.
- Very slowly lower the transfer tube lower into the liquid  $^4\text{He}$  in the dewar. There should be an audible frequency to the gas being blown out the other end, this will increase until the inside of the transfer tube is cold enough. At which point a 'blue flame' of cold helium gas should be seen at the other end. Connect the dewar transfer tube to the bath transfer tube.
- The transport dewar tube can be slowly lowered until it is a little over  $10\text{ cm}$  from the bottom. This is to leave enough liquid  $^4\text{He}$  to keep the dewar cold until it is refilled.
- The recovery gas meter should be increasing quickly, and the thermometers should start recording a temperature drop.
- Pressurise the transport dewar from a  $^4\text{He}$  cylinder up to  $250\text{ mbar}$ .
- Once the Thermometers start reading  $\sim 10\text{ K}$  begin pumping on the IVC.

- Once the bath is full of  $^4\text{He}$  gas the 1K pot can stop being pressurised. If only one source of  $^4\text{He}$  gas is available a helium balloon can be used to start the pressurisation of the dewar.
- The transfer is stopped by de-pressurising the transport dewar, separating the transport tubes, removing the transport dewar tube and then slowly removing the bath tube.
- It is good practise to do a second transfer before starting the 1K pot, this also gives the IVC time to pump down.

## 4.2 Leak Checks

Leak checks can be carried out in a similar way to Section 2.5, once the IVC has been pumped to a low enough pressure. Although note that it becomes increasingly hard to pump out  $^4\text{He}$  as temperatures lower, so a small amount of the mix can be used to leak check the  $^3\text{He}$  circuit, the 1K pot is already full of  $^4\text{He}$  so is automatically checked, and the fill line may be checked using high purity helium.

## 4.3 Transfers When Running

A second transfer is normally done before running as IVC will take a little time to pump down. The 1K Pot pick-up is at  $\sim 375\text{ mm}$  on the bath depth meter so if the level is much higher than this the fridge could be started, however significant evaporation will come from condensing the mix so normally the bath is filled to the top before continuing, this will also lead to quicker condensation as the more of the SS pipes running through the bath will be at  $4.2\text{ K}$ .

When transferring after the first transfer it is even more important to lower the transfer tube slowly into the bath. Firstly the transfer tube should very slowly for the first few  $\text{cm}$  so that air inside isn't suddenly frozen to the end blocking it. Secondly to avoid unnecessary evaporation, watch the helium gas meter as the transfer tube is inserted, when this begins to speed up the tube is close to the bath level. At this point wait a short amount of time for the tube to cool and then proceed slowly. Unfortunately there is no way to transfer without inserting the tube to near the bottom of the bath, so care should be taken to cool the tube in the cold  $^4\text{He}$  vapour before inserting into the liquid. As mentioned the 1K Pot pick-up is at  $\sim 375\text{ mm}$ , the rate of helium evaporation overnight is around  $55\text{ mm}$  but during the day this can be increased significantly when running experiments. Transferring if the level meter is around  $600\text{ mm}$  is a safe approach, although for key microwave experiments it is worth transferring just before to minimise temperature of all coax. There is no clear maximum helium level, however around

1000 *mm* the RT plate will begin to freeze; slowing down the transfer rate 1125 *mm* is easily achievable. Care should be taken to not freeze the IVC ports as this could lead to a leak through frozen o-rings. Other than these small differences the transfer process is the same as above.

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# Cooling to Base Temperature

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## 5.1 Starting 1K Pot

Pumping the exchange gas out the IVC should have begun once the thermometers read  $10\text{ K}$ . The pot should be full of liquid helium with both needle valves half a turn open and the bath level well above  $375\text{ mm}$ .

- Once the pressure in the IVC is  $\sim 1 \times 10^{-6}\text{ mbar}$  the IVC can be closed, pumping on the fridge below  $4.2\text{ K}$  is very slow and there is a risk of cryo-pumping any small leaks in the pumping trolley line.
- Ensure V13, V14, V15 and '1K' (Figure 1.2) are closed and the 1K pot pump exhaust is to the recovery, then start the 1K pot pump.
- Close secondary needle valve and move primary needle valve to a quarter turn open.
- Slowly open V14, pressure on G5 should drop slowly.
- Once V14 is fully open and G5 is **low** open V13.
- G5 should head towards  $0\text{ mbar}$  and at low pressure G6 should start dropping. G6 should level out around  $3\text{ Torr}$ .
- primary needle valve can be reduced to  $\sim$ one eighth open.

## 5.2 Condensing Mix

There should be  $\sim 650\text{ mbar}$  in dump 1 and  $\sim 180\text{ mbar}$  in dump 2, G4 should roughly match the pressure in dump 2. If lower mix has been lost, if higher air has gotten into the system. To condense:

- Condense through CT 1 or 2, filling  $\text{N}_2$  trap and cooling CT, onto the larger still side of the fridge.
- Open dump 2 and valves 2,7,8.
- Slowly adjust the valves 1 and 9 so that G4 is around  $100 - 200\text{ mbar}$  whilst G2 & G3 are kept  $50 - 100\text{ mbar}$ .

- When G1 drops to  $\sim 50$  *mbar* close dump 2, 2 and 9. Then open dump 1 and repeat the condensation process.
- Because the fridge is running low on mix it is worth thoroughly condensing everything in the dumps.
- Once both dumps are low close 2, 7 and 9.
- Open both dumps, then 5 and S\*. This should start the  $^3\text{He}$  rotary pumping on both dumps.
- If pressure on G3 becomes  $> 100$  *mbar*, 3 can be opened.
- After pumping both dumps down below 1 *mbar* (P2) close s\*. 5 should be left open so the bypass is connected to the dumps in case of an over pressure in the circuit.
- All the mix should now be condensed and all valves on the red  $^3\text{He}$  circuit (Figure 1.1) can be opened apart from 13 (only using whichever CT is in  $\text{N}_2$ ).

### 5.3 Starting Circulation

Once the pressures in G1-4 are  $\sim 70$  *mbar* or below the mix should be condensed enough to start circulation. The  $^3\text{He}$  rotary should already be turned on.

- Slowly open 12, G3 should be kept below  $\sim 200$  *mbar*.
- When P1 and P2 are reading  $\sim 1$  *mbar*, open 13, close 12, and then start the turbo. The turbo should read  $\sim 16$  *A* when running up, with a operational frequency of 600 *Hz* once it has ramped, and 1 *A* when running in low circulation mode.
- We have been keeping the Turbo current under 3 *A* once ramped, though the fridge operates well at lower current.

The fridge should now be running in low circulation mode and approaching base temperature. The entire red  $^3\text{He}$  circuit should be open along with both dumps and 5 (enabling the bypass). See the table in the next section for expected gauge readings when running at base temperature in low circulation mode.



## 5.4 Normal Running Parameters

Below is Table 5.1 of normal running parameters (e.g. pressure gauge readings) for the fridge when running at base temperature in low circulation mode. There is some variation as the optimal running parameters of the fridge have not yet been found. Additional important parameters can be found in Table 1.1.

**Table 5.1:** Expected parameters for low circulation operation. Note  $C_{still}$  is % of max current.

Item	Shorthand / Gauge	Value	Unit
Base Temp	$T_{base}$	25	$mK$
1K Pot Temp	$T_{1K}$	1.4	$K$
Still Power	$C_{Still}$	10	%
Turbo Current	$C_{Turbo}$	1	$A$
Turbo Pressure	P1	$8 \times 10^{-3}$	$mbar$
$^3\text{He}$ Rotary Pressure	P2	$1 \times 10^{-2}$	$mbar$
Dump Pressure	G1	20	$mbar$
Still Pressure	G2	40	$mbar$
Condenser Pressure	G3	50	$mbar$
Backing Pressure	G4	50	$mbar$
1K Pot Pressure	G6	2.5	$Torr$
IVC Pressure	$P_{IVC}$	$1 \times 10^{-6}$	$mbar$
1K Pot Valve	$V_{1K}$	1/8	Turns

G2 isn't actually still pressure when running, it is the dump pressure. Running in higher circulation modes (higher  $C_{still}$ ) should give a higher cooling power on the MC although it is not clear how severely the possible lack of  $^3\text{He}$  affects this. In higher circulation mode all pressures in the  $^3\text{He}$  circuit will be higher along with the turbo power. A higher circulation may also need a slightly more open 1K Pot valve.

## 5.5 Using Fill Lines

There are two fill lines attached to the fridge, they run through the 1K pot and continuous heat exchanger down to the experimental stage. An additional heat sink has been added for line **Y?** at the MC stage. The gas handling system for these fill lines leaks, and the  $\text{N}_2$  trap does not stay cold overnight. Therefore it is not currently advisable to leave cells full of helium for extended periods. Once full at base temperature cells cannot be pumped out and so the fridge must be warmed to get the helium back out. The gas handling is being replaced with a high pressure one for future experiments.

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# Warming Up and Opening

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## 6.1 Removing Mix

These instructions proceed from a low circulation mode assuming the  $^4\text{He}$  bath level is still above the 1K pot intake. The fridge can be warmed from any any state using this method, however care should be taken with local heating and sudden changes in pressure. To recover the mix:

- Turn off the turbo and close 3.
- Allow turbo to run down then turn off the 1K pot.
- Slowly open V15 to make sure the 1K pot is open to the recovery.
- Close dump 2 and open 2, this passes the mix through a CT one last time before recovering it to a dump.
- Open both 1K pot needle valves.
- Increase the still power to 30%.
- Slowly increase the sample heater to 30 *mW*. Keeping an eye on the pressures and temperatures of the fridge.
- Once G2 reads above  $\sim 650$  *mbar* close dump 1 and open dump 2.
- If filling a cell with  $^4\text{He}$  the MC should be held just above 4.2 *K* for a while to recover the  $^4\text{He}$  into the gas handling system, using a rotatory to pump the last bit. This should be done before letting exchange gas in.
- Once  $T_{MC}$  is close to 4.2 *K* exchange gas can be let into the fridge as in Section 3.1.
- Wait for mix to be recovered from inside fridge, this can be seen by P1 dropping and the combined dump pressures nearing 830 *mbar*.
- $C_{Still}$  at 10% and mixing chamber power of 5 *mW* is safe overnight with  $P_{IVC}$  around  $2 \times 10^{-2}$  *mbar*.
- Once most of the mix is recovered close 8 and 5. Then open 9, then 7 and 3 slowly.

- All sections of the  $^3\text{He}$  circuit should now be being pumped on (apart from the back side), into the dumps through 9. 6, 10 and 4 can be opened to pump on the cold trap from both sides.
- As P1 approaches 1 *mbar* shut 9 and dump 2.
- The  $^3\text{He}$  circuit and cold traps can then be cleaned as in Section 2.1.

At the end of this process  $\sim 830$  *mbar* should be the combined dump pressures and all the mix should either be in the dumps or back side of the  $^3\text{He}$  circuit. All valves on the main  $^3\text{He}$  gas handling system should also be closed.

## 6.2 Warming Up

Once the mix has been recovered it is just a waiting game until all the  $^4\text{He}$  in the bath has boiled off. Small amounts of heat can be applied to the heaters inside the IVC to speed up this process. There is currently not a warm up heater on the fridge to speed up this process. Water cooling to the turbo can also be shut off at this step. Once the bath is empty close the recovery, V13, V14, V15 and switch the 1K pot to backing to the pit. Wait until the fridge has warmed up to RT to close the 1K pot needle valves.

## 6.3 Dropping Dewar and Opening IVC

Wait until the temperature inside the IVC has returned to RT before opening. The dewar can be dropped back into the pit while the IVC is still under partial vacuum if changes only need to be made outside the IVC. Alternatively if modification need to be made to the top plate the IVC vacuum can be broken whilst the dewar is still in place. The process for lowering the dewar and then removing the IVC is:

- Recovery should be closed along with all valves and 1K pot needle valves. Microwave lines should be disconnected. The  $^3\text{He}$  rotary should also be off.
- Remove RT plate screws so weight is taken by top of dewar.
- Re-attach steel bar to RT plate so that steel frame is taking weight.
- Re-attach steel cords to top of dewar and tighten so they are ready to take the weight.
- Remove dewar bolts from steel frame putting weight on pulley system.
- Remove pit plate below dewar.

- Lower dewar into pit and re-cover.
- The SS IVC can now be removed.
- Slowly open the IVC speedivalve to break the vacuum.
- Place the jack and books below the IVC to take the weight when the seal it broken.
- Remove screws between IVC and 4K plate.
- Use plastic screws between IVC and 4K to break the indium seal.
- One person holds the IVC up while the other person removed the jack, books and circular pit plate. The second person can then entirely remove the IVC by first lowering it part way through the hole.
- Removing the indium straight away is easier than waiting until the start of the next run. The indium can be saved to be recycled.
- Remove the pit plate, return the dewar circular plate, return the pit plate and central circular plate.

The fridge should now be in the fully open state ready for modification or the switching of experimental cells.

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## Bibliography

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- [1] S. Spence, “Third year report,” tech. rep., Royal Holloway, 2020.