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Operation of the Kelvinox Dilution Fridge Under Different Temperature Regimes

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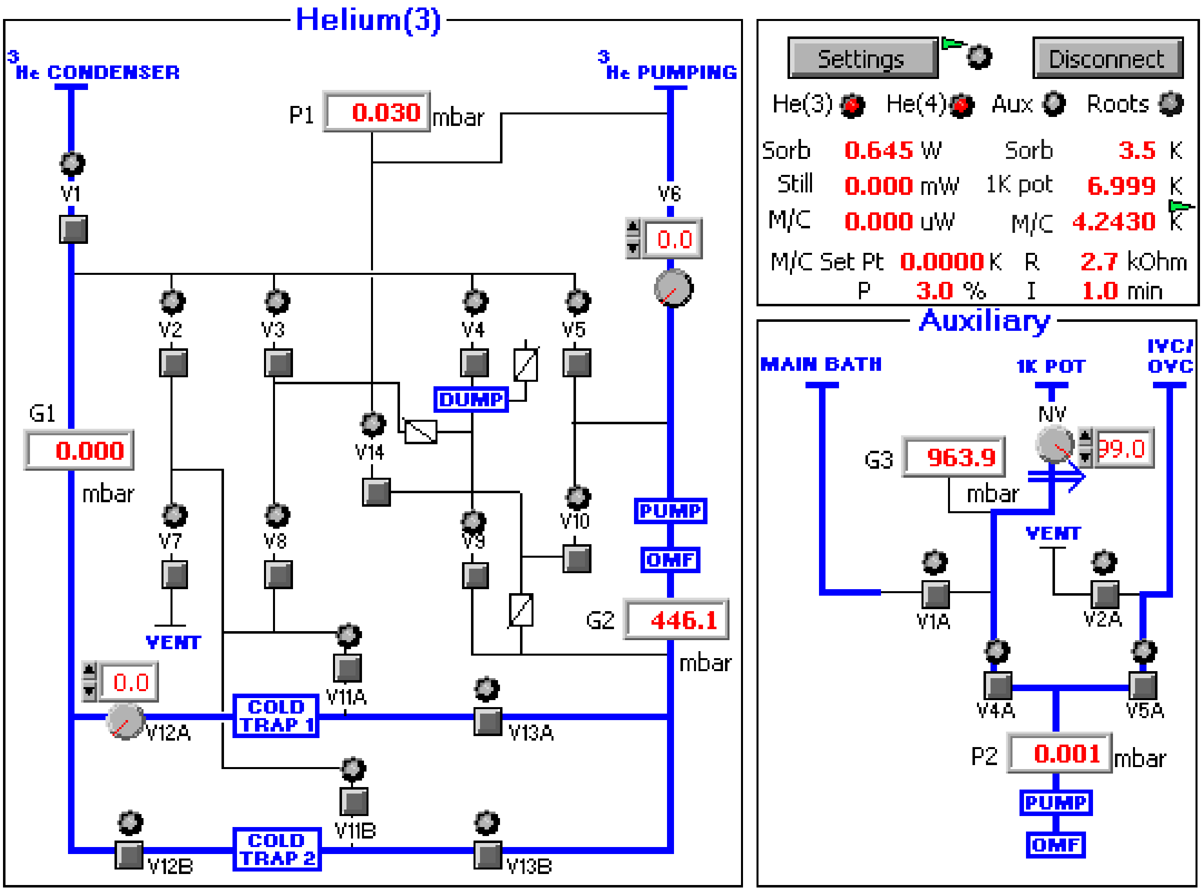

Figure 1: Schematic of the IGH front panel showing all the valves, pressures, temperatures and heater powers which will be referred to in this manual.

1 Introduction

A dilution fridge is a scientific instrument which uses a mixture of helium 3 and helium 4 to reach temperatures in the miliKelvin range. Although the operation of such machines in general is relatively straightforward, running a dilution fridge under certain regimes can become tricky and is more of an art than a science. This guide is designed to explain how to run the Kelvinox 25 dilution fridge in the Halle Vittoz under several different regimes. In this manual it is assumed that all the different vacuum tubes are connected, the two helium pumps are running and all manual valves are open. Throughout this manual we will refer to many valves and pressures which are found on the gas handling system (IGH). A schematic of the IGH is shown in Figure 1.

2 Getting to base temperature

This section deals with the procedure used to introduce the mixture into the system followed by cooling the fridge down to base temperature. This can be divided into two sub-sections: Condensing the mixture into the mixing chamber and then circulating the mixture.

2.1 Condensing the Mixture

2.1.1 Cooling the 1K pot

The condensing phase is where the bulk of the mixture is taken from the dump and condensed into the mixing chamber. Before condensing can begin, the 1K pot must be cold. The optimal temperature for the 1K pot is typically between 1.4 K and 1.6 K. To cool down the 1K pot valve V4A has to be opened followed by the needle valve. As the software for the dilution fridge changes the needle valve setting almost at random, the needle valve control has been disconnected from the IGH and must be opened manually. The needle valve must be opened until the pressure in G3 rises to something in the region of 10mbar. If at any point in time this pressure drops to zero, the needle valve must be opened some more as the 1K pot will be empty and rapidly warm up.

2.1.2 Condensing the mixture

With the 1K pot cold, the mixture can be introduced into the fridge. Before doing this, make sure that the nitrogen cold trap dewar has a sufficient amount of liquid and that the helium cold trap is fully inserted into the cryostat. To start condensing the mixture, first open valves V9, V13A and V1 then slowly begin to open V12A. The reason why we want to open the valves slowly is that if too much mixture is allowed into the fridge, the 1K pot will most likely warm up too much due to the large flow of gas through it. Thus the pressure on G1 should ideally be kept below 200mbar by controlling V12A. V12A can normally be opened initially to 9% and then opened by 1 to 5 % intervals, making sure the pressure never increases too much. Continue opening V12A until it is fully opened and the LED on the IGH turns green. Before circulating the mixture, it is a good idea to wait until the pressures on G1 and G2 drop to around 100mbar.

2.2 Circulating the Mixture

With pressures G1 and G2 below 100mbar the circulation of the mixture can begin. This circulation will cause the mixing chamber to cool below 1K. To start circulating, first V9 must be closed and then V14 opened. The pressure gauge P1 will probably show 1000 mbar even before opening V14, this is not a problem as the pressure gauge saturates with only a few millibar of pressure. The next step is to slowly open V6 while keeping the pressure in G2 relatively low. It is a good idea to aim for a pressure of at most 150mbar. The low pressure is desired as theoretically if V6 were opened quickly, the sudden rush of gas to the helium pump could cause oil to be blown out of the pump and into the system. Generally V6 can be opened to near 11% initially and should then be opened by 0.2% intervals. Once the pressure reading on P1 is below 100mbar, V6 can be fully opened. Once V6 is fully opened the pressure on P1 will slowly decrease. When this pressure reaches zero valve V14 can be closed as all the mixture has been removed from the dump.

2.3 Typical operating parameters at base temperature

Once the mixing chamber temperature has gone sub Kelvin, the still heater can be turned on using a power from 4-6 mW. In general with this kind of heating power the pressure on p1 is in the region of 0.01 mbar due to the extra evaporation of the mixture. The typical base temperature achieved is 50mK and should be achieved within one and a half hours of starting the condensing procedure. Additionally when the system is running smoothly at base temperature the pressures of G1 and G2 are in the range of 100-150 mbar. Obviously at higher temperatures, these pressures are expected to be somewhat higher. It is normal to have a difference in pressure between G1 and G2 with G1 5-10 mbar lower due to the finite time it takes for the helium gas to circulate through the cold traps.

3 Temperature Control

Dilution fridges work as there is a phase separation between liquid helium 3 and helium 4 which takes place at $\sim$780 mK. Unfortunately, this means that for temperatures above this separation it becomes increasingly difficult to control the temperature of the system accurately. This section deals with how to stabilise the temperature of the mixing chamber in different temperature ranges.

3.1 Base Temperature - 1.2K

Between base temperature and 1.2K it is normally sufficient to use the temperature control for the mixing chamber without any adjustments to the operation of the system. To do this, first click on the button in the IGH front panel software labeled *Settings*. This will cause a new window to open which has the settings for all the heaters present. In this window press the button labeled *M/C Set Point (K)*, which will open a further window with the settings for the mixing chamber temperature control. Inside this window, enter the desired mixing chamber temperature and then press *OK* to change the temperature. If the mixing chamber for some reason does not stabalise at the required temperature, but instead oscillates, it may be necessary to adjust the PID parameters manually. This can be done in the window with the mixing chamber temperature controls. The *P* term is the only one which should need to be adjusted and should be in the range of 3 to

30. Unfortunately at this point we do not have a very good idea of the ideal parameter settings, so the only way to set the PID's correctly is via tweaking.

3.2 1.6K - 6K

At high temperatures, the best way to achieve a stable temperature is to essentially stop pumping on the mixture. With the 1K pot as cold as possible (typically in the range of 1.5K) closing V6 will lead to the mixing chamber stabilising at a temperature very close to that of the 1K pot. From here by using the procedure found above, the temperature can be controlled between 1.6K and 6K with a good accuracy. Due to the fact that the cooling power is now much less than when the dilution fridge is running under normal operation, the heater range of 2mW should be sufficient except for the highest temperatures. If the temperature is very unstable this time round, it is most likely due to the heater range being set too high and should thus be manually changed until the correct range is found.

3.3 1.2K - 1.6K

This range of temperatures has been left until last as it is definitely the temperature range where control is most difficult. The main reason for this is that in this region, the cooling power of the fridge changes between 2 and 20mW and the PID tables seem to have a hard time to keep a stable temperature. In addition to this, at temperatures above $\sim$1.5K the heater installed on the fridge is not sufficiently powerful. To combat these two problems, the amount of mixture circulating in the system needs to be reduced. This is done by first of all opening V9 to let some of the mixture back into the dump. In addition to this, the valves to the condenser line and to the pump after the still need to be partially closed. Typically good settings are having V12A opened to 25% and V6 to 15%. When running the fridge in this temperature range, it is necessary to keep an eye on the heater power being used. If it appears that the fridge will run out of heating power, the two valves should be opened a bit more; typically opening the valves by $\sim$1% is enough to increase the cooling power.

4 Tips to improve performance

This section deals with some methods which are used to improve the performance of the fridge to get a better base temperature. In general the settings here do not need to be tweaked, although it is possible to decrease the base temperature by a few milikelvin by patiently adjusting the settings.

4.1 1K pot temperature

The 1K pot can theoretically be tuned to improve the cooling power and base temperature. This is done by using the needle valve to change the temperature of the 1K pot, which will affect the mixing chamber temperature. Unfortunately, there is no golden rule for which 1K pot temperature gives the best results, although having a temperature of $\sim 1.8\,K$ has been beneficial, particularly when there is a large thermal mass to cool down.

4.2 Heating the still

The still heating power is critical to an optimal running of the machine. As has been previously mentioned, typically 5 mW of heating power gives the optimal results. As the performance of the fridge seems to change slightly with each cool down, the required still power can also change. It is recommended to start with a still power of 5 mW and then make adjustments in 1 mW intervals to improve performance. It is important to wait for a few minutes between changing the heating power as the fridge can take quite a while to reach a stable temperature.

4.3 Single shot technique

The single shot technique allows for the base temperature to be lowered significantly for a short amount of time. The idea behind this technique is to run the fridge in a regime where no gas enters into the system, thus reducing the heat load. To perform a single shot, the fridge has to be running normally, with all the mixture condensed and base temperature achieved. To initiate the single shot, close V1 and open V5 to stop the circulation of the mixture. the base temperature will almost immediately start to decrease and will continue to do so until all the ^{3}He has been removed from the mixing chamber. During the single shot it may be useful to increase the still heating power, effectively increasing the cooling power.

5 Problem solving

The final section of this document focuses on identifying the causes of bad cooling performance and gives possible solutions.

5.1 Large pressure difference between G1 & G2

A block in the nitrogen cold trap will present via large difference between pressures G1 and G2. If G2 is consistently more than ∼15mbar larger than G1, action should be taken to clean the cold trap. Fortunately this can be done without stopping the cirulation of the mixture. The pressure of P1 must be noted down as this will be used to determine when all the mixture has been removed from the cold trap. The procudure is to first open up valves V12B and V13B and close V12A and V13A, causing the mixture to circulate through the second cold trap. The next step is to pump out the mixture which is still in the primary cold trap. This is done by opening valves V11A, V8 and V3 until P1 has dropped back to its initial value and then close these three valves.

To remove the block from the cold trap, first attach a pump to the vent port on the back of the IGH and open the manual valve and evacuate upto the vent port. It is also possible to use the ^{4}He rotary pump if another pump is not available; in this case open vavles V5A and V2A. Regardless of which pumping method is used the following procedure remains unchanged. With the tubes upto the vent valve evacuated, open valves V7 and V11A to start pumping on the cold trap. Slowly remove the cold trap from the nitrogen dewar, being careful to not remove it so quickly as to create an overpressure. If the pressure becomes too high, the cold trap can be lowered into the dewar once again. With the cold trap out of the liquid nitrogen, it should be heated up to 100^{o}C (once again paying attention to the pressure). The nitrogen cold trap will probably need to be pumped for at least an hour until the pressure becomes reasonably low. Once the pumping is finished the valves used for pumping can be closed and the cold trap can be replaced. The mixture can then be returned through the primary cold trap if desired by opening V12A and V13A, closing V12B and V13B then opening V11A, V8 and V3 to pump the mixture out of the second cold trap.

5.2 High Pressure on G1 & G2

A high pressure on G1 and G2 can indicate a block somewhere in the system. Such a block will decrease the flow of mixture and decrease the cooling power and lead to a poor base temperature. The most likely source of this block is in the helium cold trap. To remove the blockage, first close V13A then open V5 and V9. This will start to pump all the mixture back into the dump. Once all the mixture is back in the dump (P1 will show a pressure of zero), close V6, V12A and V4A. With the valves closed, open V2, V7, V2A and V5A to begin pumping on the cold trap. Slowly raise the cold trap out of the Dewar, being careful not to remove it so fast that an over pressure is created. Once the cold trap is completely removed, it can be heated up to 100^oC remove any water in the cold trap. Once the pressure on P2 is zero, the heating can be stopped, the cold trap replaced and the mixture re-condensed. If this doesn't solve the problem and the pressure on G1 and G2 are still very high and a god base temperature is unachievable, the whole fridge must be taken out to remove the blockage.

5.3 When all else fails

This final paragraph is dedicated to ideas which may be able to help when the fridge isn't behaving nicely and the other problems weren't present. Two ideas are presented to attempt to improve performance.

5.3.1 Pumping the IVC

Although it is unlikely that it would be possible to condense the mixture if the IVC had too much exchange gas in it, experience has taught us otherwise. Occasionally we have found the fridge to have erattic behaviour when there is too much exchange gas in the IVC. To pump the IVC, attach a pump directly to the valve and pump the tube to a pressure of at most $10^{-5}\,mbar$. Then open V9 to the dump and heat the sorb up to 40K. Once the heater has been switched on, the manual valve to the IVC can be opened. If it is preferred to keeep the mixture condensed, pulse the heater for 10-30 seconds then turn it off again. Once the pressure in the IVC while heating has dropped below $\sim 10^{-4}\,mbar$ the heater can be turned off and the IVC valve can be closed again.

5.3.2 Recondensing

If the fridge is not working as it should be or normally does, it is quite often a good idea to simply heat it up to above 4K and then recondense. The theory is that it could be possible that the mixture has condensed in an unusual configuration and recondensing it will result in a smoother running. To evaporate all the mixture quickly, first open V9 and close V13A along with the needle valve for the 1K pot. Begin to heat the mixing chamber to 6K, the still with 10mW and the sorb to 40K simultaneously. The majority of the mixture should have evaporated within a couple of minutes and then the procedure for condensing the mixture can be followed to recondense.