Oxford Instruments *Triton 300* 64913 Yoshida Operating Instructions and Tips

Figure 1: Main cryostat of Triton system with partial OVC shield

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October 30, 2018

Triton 300 64913 Yoshida

Chapter 1

Preface

Written by Ken on October 18, 2018
Edited by Ken on October 30, 2018

1.1 WARNING - READ THIS BEFORE USING!

- NEVER EVER OPEN V10 - The consequences of doing so may break the dilution refrigerator - loss of expensive ^{3}He gas, 11L - $\sim$ ¥5,500,000!
- Make sure that you understood the basic dilution refrigerator principle and read this manual thoroughly before using the dilution refrigerator.
- V13 is usually close and V14 is usually open, please contact Oxford Instruments/-experts when you are not sure. There is no need in any normal operation where V14/V13 are needed to be adjusted.
- In the event of sudden power outage, please contact Oxford Instruments - This system does not have a UPS to support the Triton system control in these events.
- Always use a glove when working with the main cryostat.
- Always leak check of the OVC after reassembling the OVC.
- A good mastery of English is recommended as operating control system, manuals are all in English.

1.2 Acknowledgement

Many thanks to Yuji Yamanaka from Oxford Instruments for teaching the know-how of operating a dilution refrigerator and final installation procedures. Many Thanks to Takayuki Kawamura from Oxford Instruments for the assembly of Triton main system.

1.3 Triton Dilution Refrigerator Yoshida 64913 system

- Installation Completion Date - 19 September 2018

- ^{3}He/^{4}He ratio in Mixture Tank - 11L^{3}He, 64L^{4}He (Total 75L)
  P1 $\sim$ 0.748bar (74.8L of mixture gas)
- Storage tank capacity - 100L
- OVC target pressure - $\sim 10^{-3}$mbar
- Triton liquid N_2 dewar capacity - 50L
- Maximum load in Mixing Chambers - 20kg, same as M3 threading shear limit.
- Base temperature measured using nuclear orientation thermometry, 9.8$\pm$0.1mK
- Throughput test at 300K, P2 - 3bar, ΔP3 - 0.3mbar, at 10K ΔP3 should be around - 10mbar
- *Triton 300* Cooling Power, 250μW at 100$\pm$1mK

1.4 Diagrams and Figures

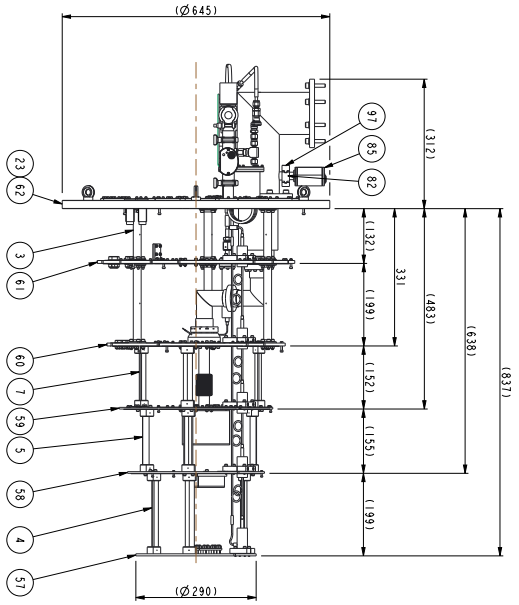
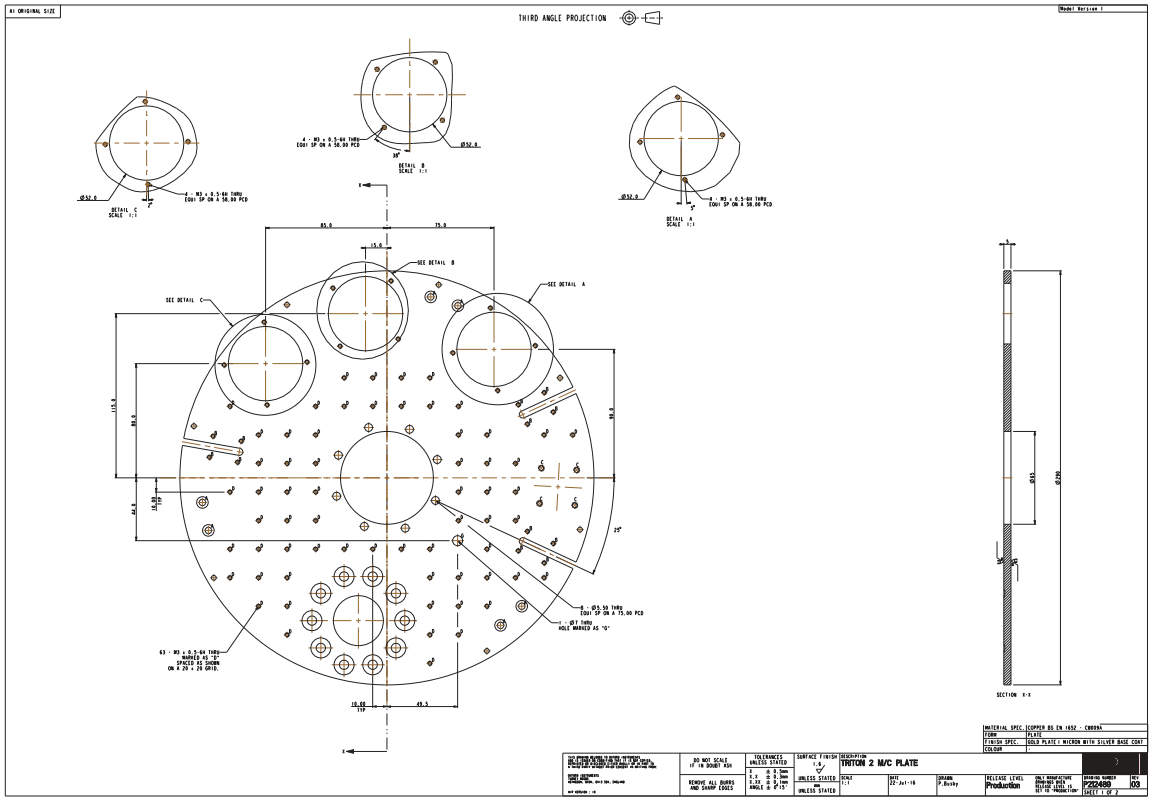


Figure 1.1: Engineering diagram of the main cryostat of Triton 300 System. The plates from top to bottom:- Top plate(r.t.), PT1 plate($\sim$ 40K), PT2 plate($\sim$ 4K), Still plate($\sim$ 1K), Cold plate($\sim$ 100mK), Mixing Chambers plate($\sim$ 10mK)



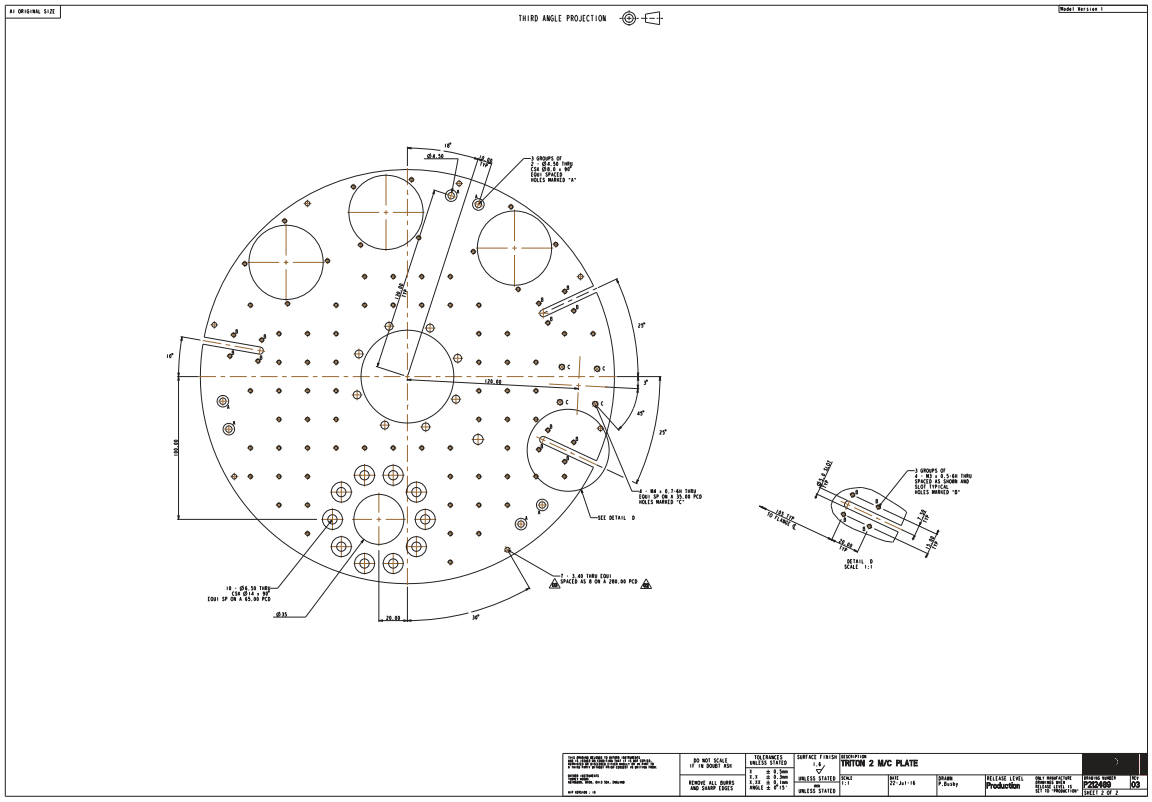


Figure 1.2: Engineering diagram of Mixing Chamber plate of Triton 300 System.

Chapter 2

Dilution Refrigerator for Dummies

2.1 ^{3}He/^{4}He Dilution Refrigerators

[1] 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 performance of these systems has steadily improved, and the physical processes involved have become much better understood.

When a mixture of the two stable isotopes of helium is cooled below a critical temperature it separates into two phases. The lighter 'concentrated phase' is rich in ^{3}He and the heavier 'dilute phase' is rich in ^{4}He. The concentration of ^{3}He in each phase depends upon the temperature. Since the enthalpy of the ^{3}He in the two phases is different, it is possible to obtain cooling by 'evaporating' the ^{3}He from the concentrated phase into the dilute phase.

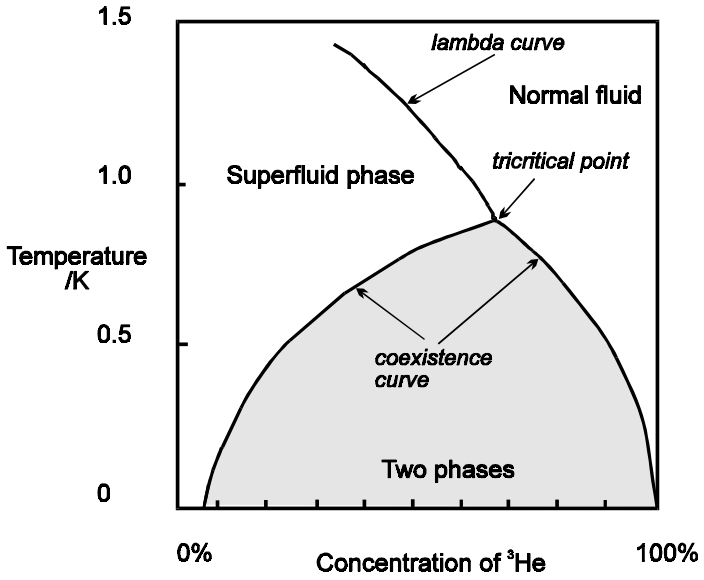


Figure 2.1: Phase diagram of ^{3}He/^{4}He mixtures

[1] Taken directly from "Practical Cryogenics - An Introduction to Laboratory Cryogenics by N H Balshaw"

The properties of the liquids in the dilution refrigerator are described by quantum mechanics and the details will not be described here. However, it is helpful to regard the concentrated phase of the mixture as liquid ^{3}He, and the dilute phase as ^{3}He gas. The ^{4}He which makes up the majority of the dilute phase is inert, and the ^{3}He 'gas' moves through the liquid ^{4}He without interaction. This 'gas' is formed in the mixing chamber at the phase boundary. This process continues to work even at the lowest temperatures because the equilibrium concentration of ^{3}He in the dilute phase is still finite, even as the temperature approaches absolute zero. However, the base temperature is limited by other factors, and in particular by the residual heat leak and heat exchanger performance.

When the refrigerator is started the 1 K pot is used to condense the ^{3}He/^{4}He mixture into the dilution unit. It is not intended to cool the mixture enough to set up the phase boundary but only to cool it to 1.2 K. In order to get phase separation, the temperature must be reduced to below 0.86 K (the tri-critical point). The still is the first part of the fridge to cool below 1.2 K. It cools the incoming 3He before it enters the heat exchangers and the mixing chamber, and phase separation typically occurs after a few minutes. Gradually, the rest of the dilution unit is cooled to the point where phase separation occurs.

It is important for the operation of the refrigerator that the ^{3}He concentration and the volume of mixture is chosen correctly, so that the phase boundary is inside the mixing chamber, and the liquid surface is in the still. The concentration of ^{3}He in the mixture is typically between 10 and 20%.

In a continuously operating system, the ^{3}He 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 typical continuously operating dilution refrigerator. The ^{3}He is pumped away from the liquid surface in the still, which is typically maintained at a temperature of 0.6 to 0.7 K. At this temperature the vapour pressure of the ^{3}He is about 1000 times higher than that of ^{4}He, so ^{3}He evaporates preferentially. A small amount of heat is supplied to the still to promote the required flow.

The concentration of the ^{3}He in the dilute phase in the still therefore becomes lower than it is in the mixing chamber, and the osmotic pressure difference drives a flow of ^{3}He to the still. The ^{3}He leaving the mixing chamber is used to cool the returning flow of concentrated ^{3}He in a series of heat exchangers. In the region where the temperature is above about 50 mK, a conventional counterflow 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 often done by using sintered silver heat exchangers, which are very efficient even at the lowest temperatures.

The room temperature vacuum pumping system is used to remove the ^{3}He from the still, and compress it to a pressure of a few hundred millibar. 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 1 K pot. The primary impedance is used to maintain a high enough pressure in the 1 K pot region for the gas to condense.

The experimental apparatus is mounted on or inside the mixing chamber, ensuring that it

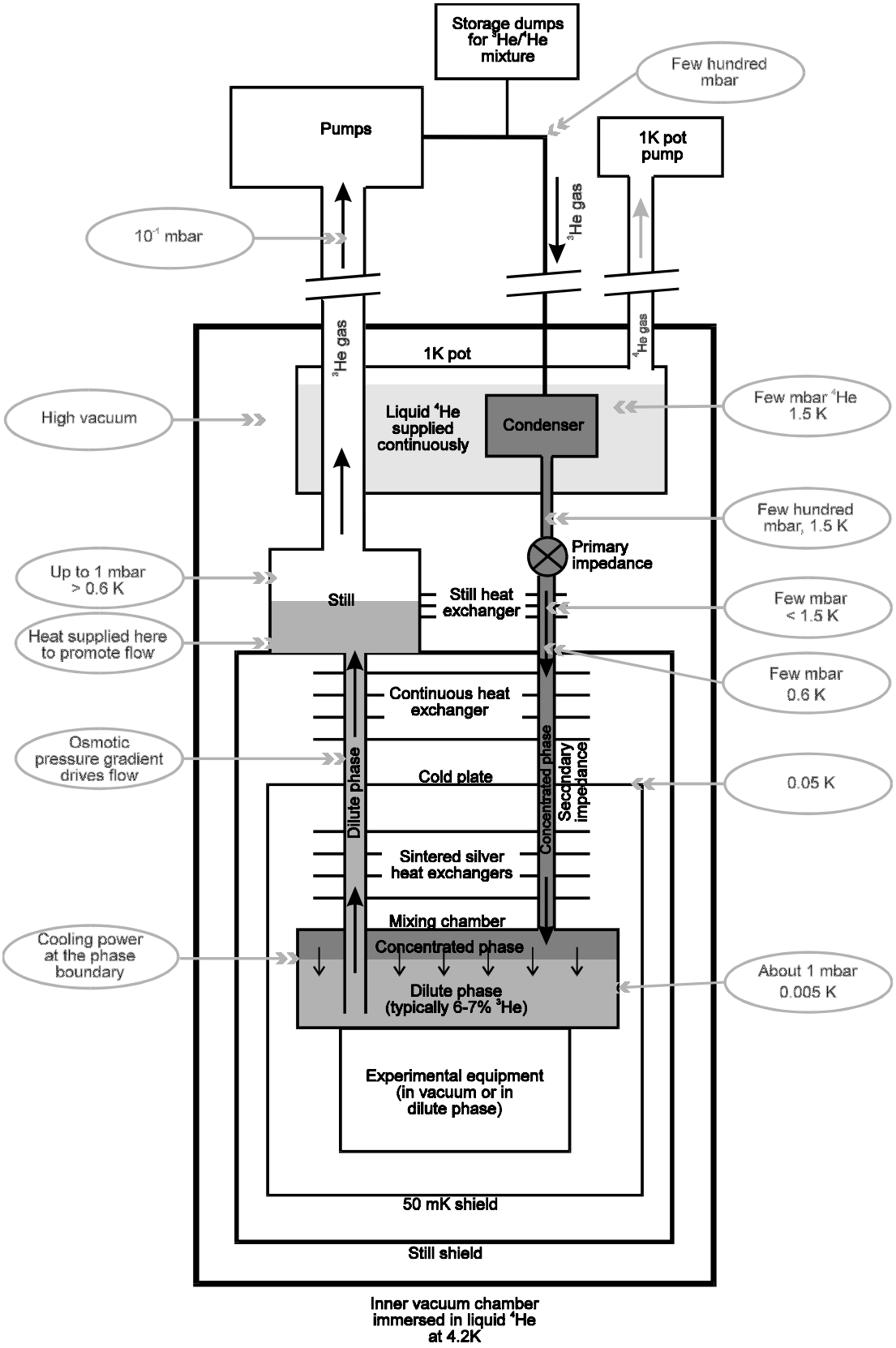


Figure 2.2: Schematic diagram of a dilution refrigerator

is in good thermal contact with the dilute phase. 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 chamber and give the lowest possible base temperature. If the experiment is to be carried out at higher temperatures, the mixing chamber can be warmed by applying heat to it directly, and a temperature controller can be used to give good stability.

As Our *Triton 300* system is a "dry" system therefore the 1K pot does not exist in our system. Instead the ^{3} gas are cooled by the pulse tube refrigerator.

2.2 Nuclear Orientation Thermometry

For accurate temperature measurement below $\sim$ 100mK.

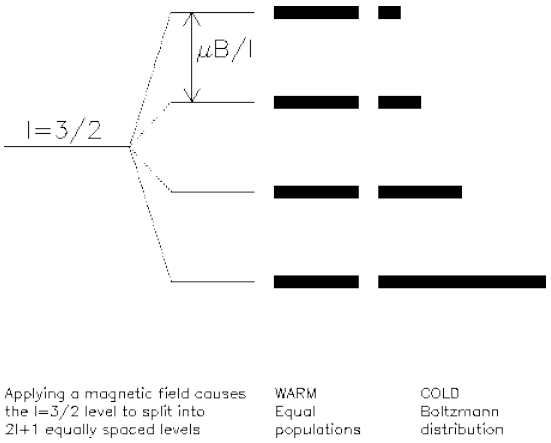


Figure 2.3: ...

2.3 Outgassing

The release of gas that was absorbed by materials. The gradual increase in pressure of a high vacuum container can be primarily explained by outgassing.

Chapter 3

The basic of operating *Triton 300*

The operation of the dilution refrigerator can be summarised into 3 main parts as follows:-

- Preparation before cooling
- Full cooldown sequences
- Warming-up sequences

To begin this tutorial of operating the *Triton 300* dilution refrigerator, we will begin with the situation in figure 3.1 where the Cryostat is without its shielding.

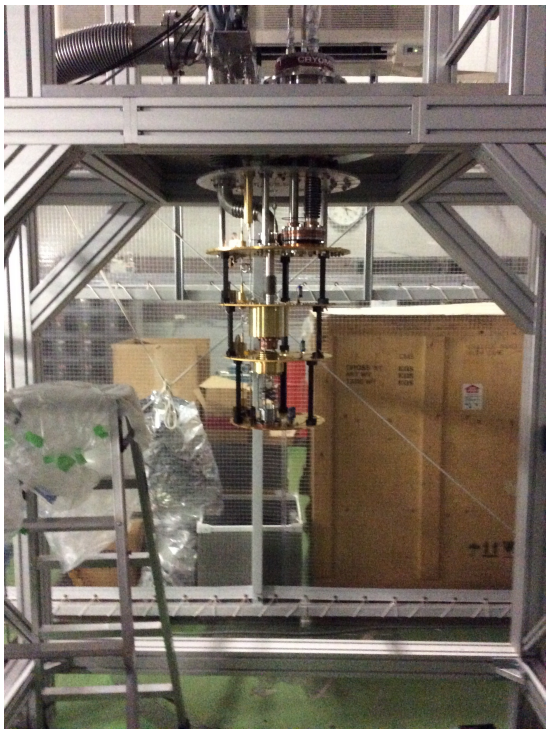


Figure 3.1: Main cryostat

3.1 Preparation before cooling

This procedure contain 3 main steps:-

- Assembly of the OVC

- Leak check of the OVC
- Throughput test

3.1.1 Assembling of OVC

The OVC comprises of 4 separate components:- still copper can, PT2 aluminium can, PT1 aluminium can and Stainless steel OVC can. It is important to note that the OVC, radiation shields, stainless steel washer and bolts are made of different materials. **As the washer and bolts are harder than the copper and aluminium cans, if they are overly tighten it can damage the copper and aluminium cans permanently!** Initial gentle tighten to make sure all bolts and washer are in place. Then tighten the bolts with washer alternatively in opposite directions as indicated in Figure 3.2 to ensure the radiation shields and OVC is properly tighten.

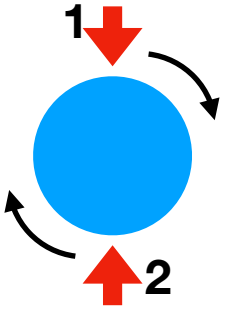


Figure 3.2: Red arrow indicate bolts position

Ensuring good vacuum seal in OVC

For the final stainless steel OVC, we need to ensure that there is a good vacuum seal therefore this step is of utmost importance in making sure of achieving base temperature of Triton.

Wearing a pair of gloves and clean the O-ring drain and the O-ring thoroughly using tissue and ethanol. Inspect both of them under bright light source and check visually there there is no dust is visible. Apply a moderate amount of vacuum grease onto the O-ring. Place the O-ring gently on the O-ring drain of the OVC. Please ensure that the O-ring is properly placed in the O-ring drain.

Place the OVC onto a lift table and using a lift table to gently raise the OVC into place. Please be careful to not damage the Al can when lifting OVC into place. Please ensure that the OVC is evenly raise into position.

Tighten the bolts with washer same as before but this time have to be as tight as possible. After cooldown has started, tighten it once again to ensure it is vacuum tight. **The O-ring is a soft material therefore be careful as not to damage it.**

notes - OVC valve

Do not open the OVC valve fully as the valve can be detach by just simply pulling the cap off when the valve is fully open. Leak from OVC valve cannot be detected.

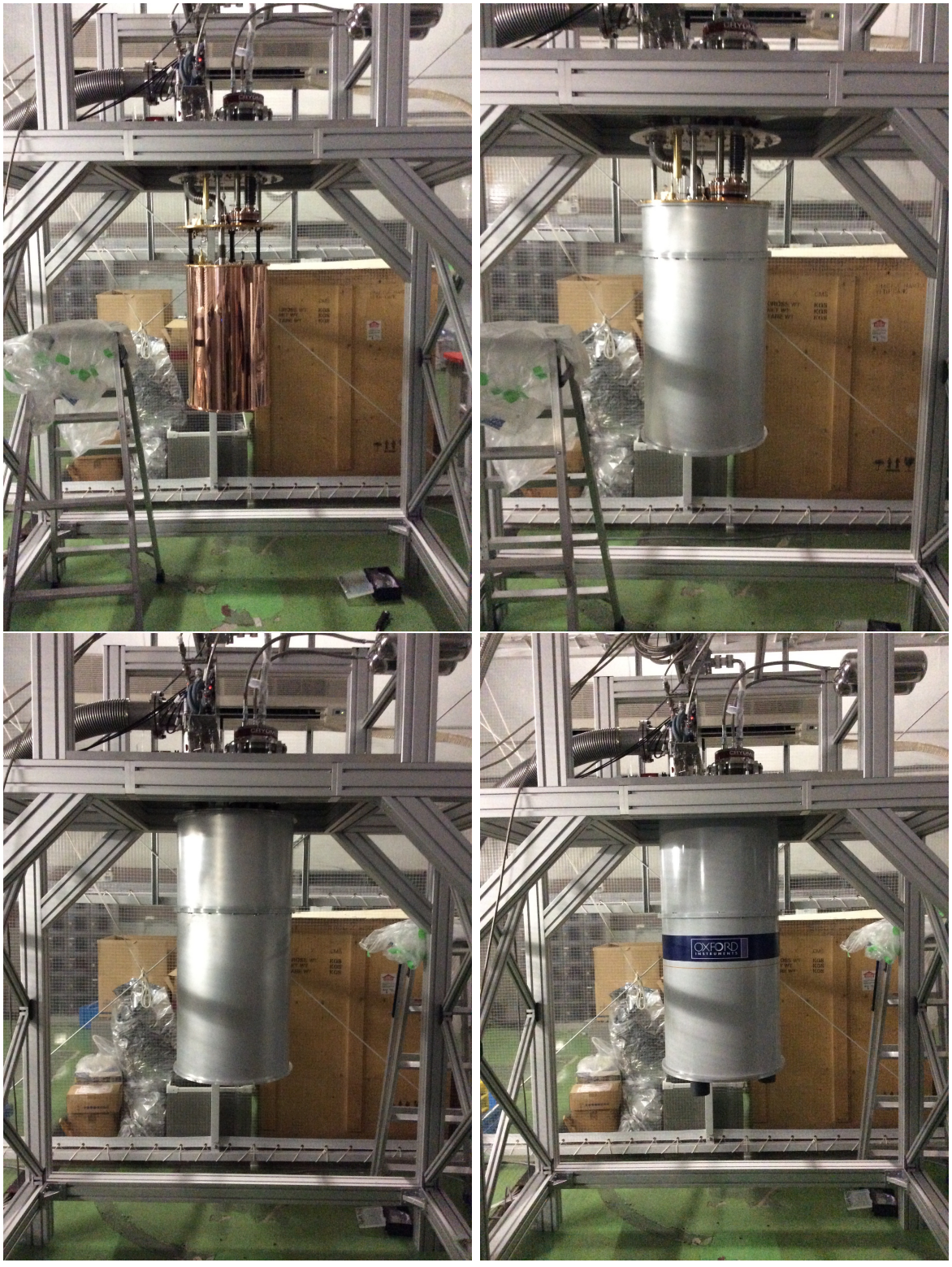


Figure 3.3: The radiation shields and OVC. From Top right:- still can, PT2 Al can, PT1 Al can, OVC

3.1.2 Leak Check of OVC

For the Triton to cool properly to base temperature of 10mK, it is important that we have a good vacuum seal of the OVC. To check this, the best way would be to do a helium leak check.

From normal atmospheric pressure, attach a rotary pump to the OVC valve located on the Top plate. This preliminary step is to first reduce the pressure of the OVC before using a more powerful TMP that is in the Helium Leak Detector and Turbo Molecular Pump Unit. **Using helium leak detector and TMP unit from normal atmospheric pressure directly may damage the TMP within**. Using the rotary pump to reduce the pressure of OVC down to ∼10Pa. Then you may switch it to the HLD/TMP for further pressure reduction. **When switching pumps, please turn off the OVC valve first before turning off the pump.**

For a good helium leak check, we need the OVC pressure to be around $\sim 10^{-2}$Pa/$10^{-3}mbar$ with a suitable background rate of around $10^{-9} \sim 10^{-10}$mbar cm^{3}s^{-1}. A sharp increase of the helium leak rate indicates that there is a leakage as shown in Figure 3.5. Random fluctuation or spikes in the leak rate maybe observed where this should not be interpreted as leakage due to outgassing and uneven distribution of helium gas at microscopic level.

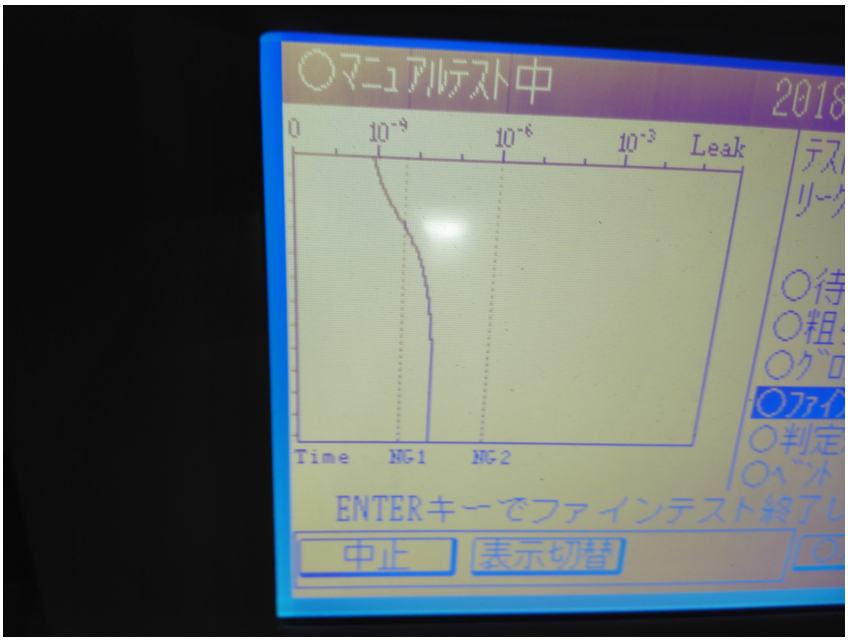


Figure 3.4: An example of a Helium Leak

Figure 3.5: From top right to bottom left:- OVC valve, Helium Leak Detector, Rotary pump, TMP unit

3.1.3 Throughput test at 300K

From the Operator's handbook, the factory data sheets contains information of the throughput test results of our Triton system tested at the factory at Tubney Woods, Oxfordshire, UK. We should expect a similar results of the throughput test when we test the system ourself at room temperature and at 10K. This is a test to ensure that there is no blockages in the mixture gas components of the dilution unit and cold trap.

For this test, we open the manual valve V11 but leave V12 close. As shown in Figure 3.6, the digitally controlled valve, V5/V1 are open with pump F/C also switched on. Open close V9 rapidly to inject tiny amount of mixture gas into the system. While doing so, it is important to not let P5 exceed 1.1bar. the initial goal is to inject gas into the system until P2 reaches 3bar. When P2 is stabilise at 3bar, observe P3 by moving the cursor onto it to observe the change in pressure for the past minute. We should observe Δ0.3mbar in P3 which indicates this throughput test a success.

Failure to observe Δ0.3mbar in P3 might indicate that there is a blockage in the mixture loop. This means that we need to carry out routine cleaning of of the cold trap and dilution unit which is explained in the later chapter 4. *P2 should not exceed 5 bar.*

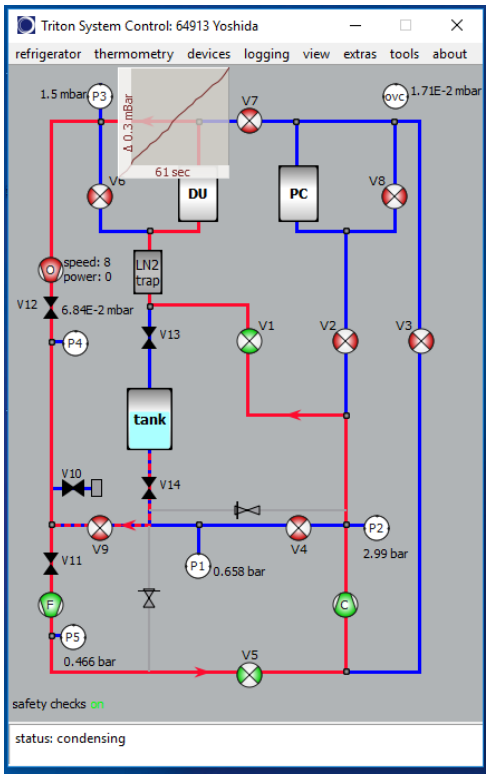


Figure 3.6: Throughput test at 300K

3.2 Full Cool Down Sequences

When all the necessary preparation are done, we can begin the full cool down sequences through the Triton System Control. This entire process is fully automated and it is as simple as a click of a button away. In Triton System Control, we can begin the full cool

down sequences from "refrigerator/full cool down" menu. The software itself will start the pulse tube cooler and begin the pre-cooling cycle.

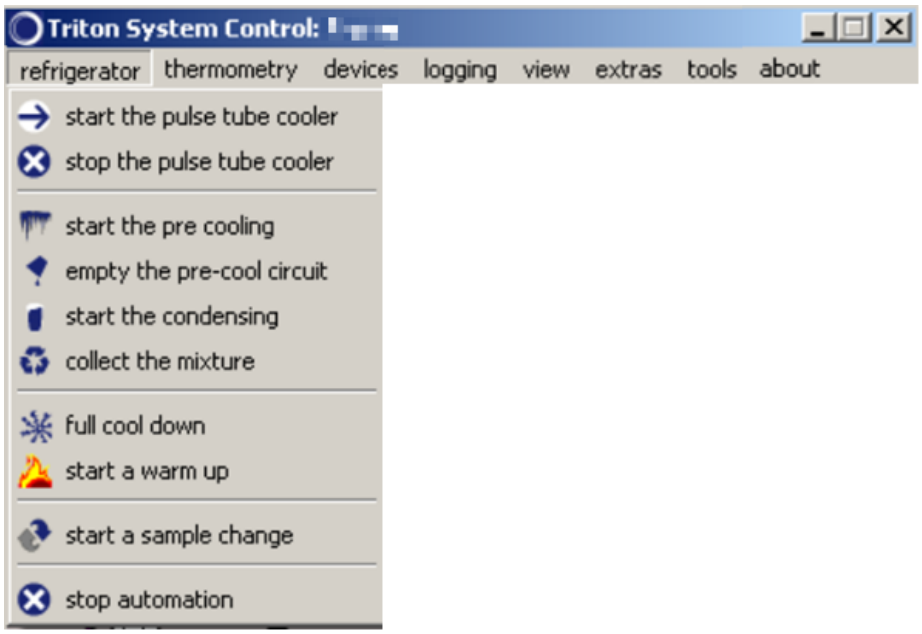


Figure 3.7: The drop down menu of Triton System Control

As simple as it is, we need to understand what each automated steps are so that proper diagnostic can be carried out in the event the dilution refrigerator unable to cool down to base temperature.

3.2.1 Cool Down to Base Temperature - full cool down

This command, it is the combination of "start the pulse tube cooler" + "start the pre cooling" + "empty the pre-cool circuit" + "start the condensing" sequences.

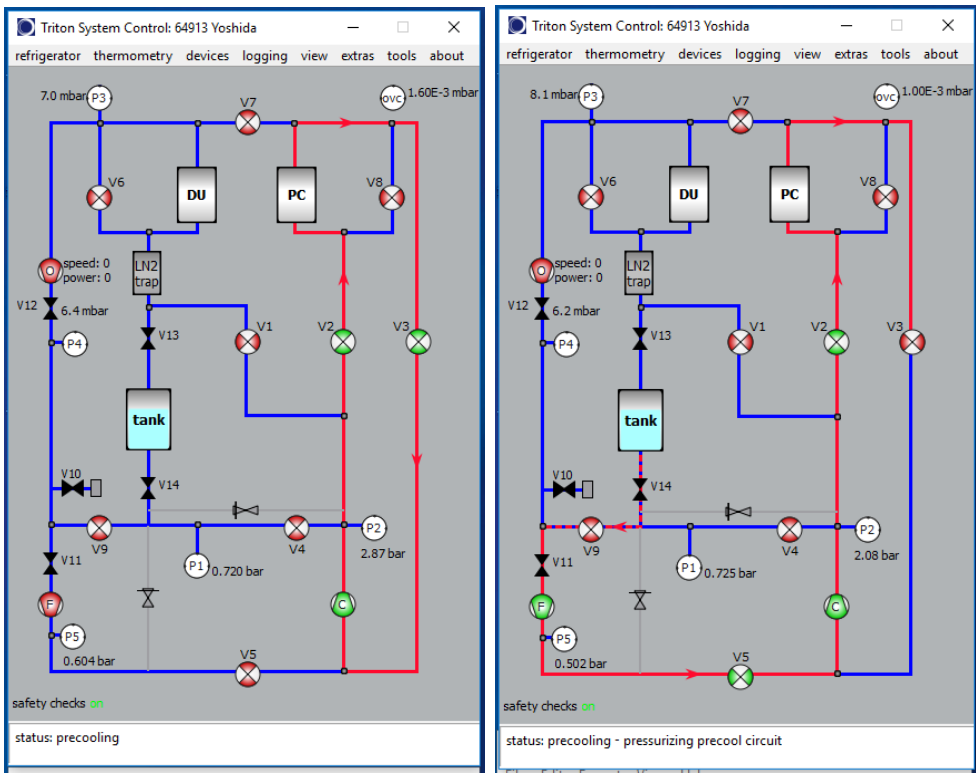


Figure 3.8: Right : circulation of mixture gas in pre-cooling line, Left : the moment during pre-cooling during the adjustment of P2 pressure at 290K of mixing chamber

Cooling The Cryostat - start the pulse tube cooler

The main cooling system of the dilution refrigerator is done by the pulse tube cooler where most of the cooling from r.t. to $\sim$ 4K is done by the PTR. *Please ensure that there is sufficient cooling water flow through the PTR compressor* as the compressor can generate a lot of heat. If the compressor is not sufficiently cooled, the cooling process of the refrigerator can be compromised or even damage the compressor itself.

Pre-cooling to 10K - start the pre-cooling

From root temperature to 10K, the system uses a small portion of the mixture gas for heat exchange via the pre-cool line in the system. This is done by adjusting the appropriate pressure in the pre-cool line at the various MC temperature level. In general, the amount of mixture gas required for the pre-cooling procedure decreases as the MC temperature decreases.

Cooling from 10K to Base Temperature - start the condensing

When the MC temperatures reaches 10K, the cooling of the MC will switch from using pre-cool unit to dilution unit. The system control will recollect mixture from the pre-cool line for about 90 minutes. After that it will begin the condensation of the mixture gas through the dilution unit. Although the actual condensation will start at 4K but using the gas mixture to exchange heat in the system as in the pre-cooling procedure.

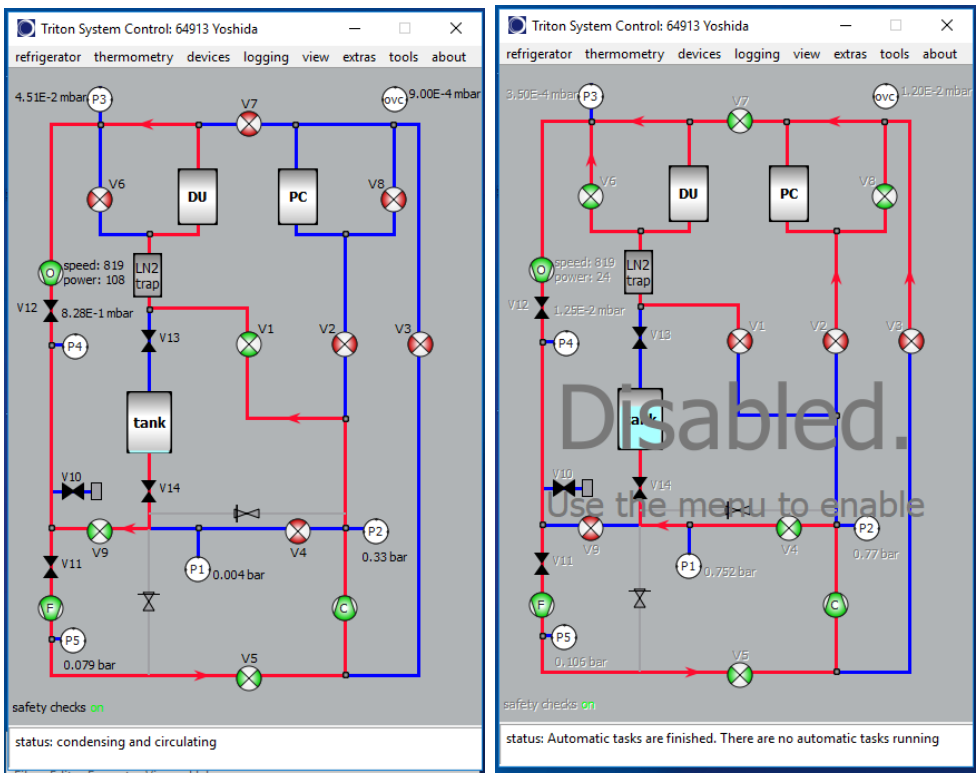


Figure 3.9: Right : dilution unit circulation at base temperature, Left : collecting mixture gas sequences

3.3 Warming up Sequences

The warming up sequences is also as simple through the drop-down menu of Triton System Control but there is a few things to take care to ensure the future functionality of the dilution refrigerator itself. The go to command for this procedure is "refrigerator/start a warm up"

3.3.1 Warming up to Room Temperature - start a warm up

This procedure is sufficient to effective warm the system back up. However the alternative way would be "stop the pulse tube cooler" → "collect the mixture". Although it is recommended to run "collect the mixture" again when the system is fully warm up. **Remember to turn off the heater after the system is sufficiently warm up, usually after a day**. This is to prevent overheating of the heating element.

Stop Cooling - stop the pulse tube cooler

This command will stop the cooling process of the pulse tube refrigerator thus effectively warm up the surrounding environment of the mixing chamber.

Recollecting ^{3}He/^{4}He - collect the mixture

This command will recollect the mixture gas, ^{3}He/^{4}He from the dilution unit into the storage tank. It is recommended to run this command again when the system is fully

warm up to ensure most of the mixture gas is recollected into the storage tank.

Turning off heating element

After approximately a day initiating warm up procedure, turn off the heater in the mixing chamber and still plate. This is to prevent overheating of the heating element.

3.3.2 Rapid warm-up - Urgent situations only

Only for Extreme situations when the only inevitable solution requires to open the OVC!

Turn on both heaters to 25mW to warm up the system. When the system reaches above 72K, inject N^2 gas into the OVC via the OVC valve on the top plate. Inject the N^2 gas slowly into the OVC as the pressure differential is quite big, as sudden rush of gas may damage the component within the cryostat. You should aim OVC pressure of around 0.3bar, this is to allow for gas expansion as it warms up. *As this procedure may cause the OVC to over-pressurised, it is important to keep an eye on the OVC pressure to not exceed 0.9bar.* Turn off the heater when it is near room temperature.

This is an extremely dangerous procedure therefore I caution anyone to not attempt this as it is an extremely complicated manoeuvre. NEVER use Helium gas as buffer gas!!! Helium molecules can be absorbed the cryostat material, it may cause outgassing when cryostat is under vacuum which renders any leak test as useless.

3.3.3 Extra: Temperature Control

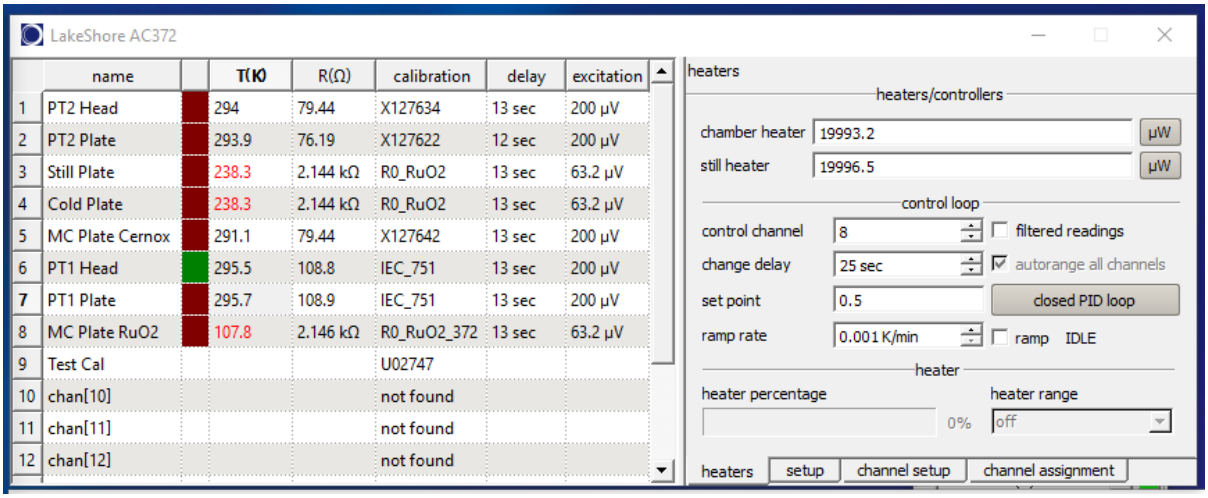


Figure 3.10: Thermometry panel, It is through this panel where temperature of MC is controlled. The heater in MC and still can also be manipulate from here.

The temperature of the mixing chamber can be controlled via proportional-integral-derivative(PID) controller. This is done by input the desire temperature in K in "set point" and closed PID loop to begin the temperature process. The PID will adjust the heaters accordingly to obtain the "set point" temperature.

3.3.4 Extra: Throughput test at 10K

The procedure is the same as at 300K. This throughput test can be carry out after the pre-cooling sequences during non-automation cooling procedure. This is just before "start the condensing" procedure.

3.3.5 Extra: Collecting mixture gas, ^{3}He/^{4}He

This is probably this most important sequences of all. It is recommended to execute this sequence just before the the warming sequences and once again when the cryostat is fully warm up. Execute this command at your own discretion such as after throughput test and mixture gas transfer. **It is important that the mixture gas is recollected into the storage tank before cleaning the cold trap or dilution unit.**

Chapter 4

Triton 300 DR Maintainances

The maintenance of Triton dilution refrigerator is an integral part of ensuring the continuous operation and longevity of this dilution refrigerator. *Please take note the status of the various component of the dilution refrigerator from time to time.*

4.1 During Triton Active Operations

This section will focus on the maintenance aspect of Triton dilution refrigerator when the dilution refrigerator is running.

4.1.1 Refilling Cold Trap

Please do not overfill the liquid nitrogen dewar for cold trap(Figure 4.1). It is recommended to refill the dewar every 1∼2 weeks via the the ventilation port of the cold trap. When refilling the cold trap dewar with liquid nitrogen, it is important to ensure that there is sufficient ventilation in the experimental room. **There is danger of asphyxiation!**

4.1.2 Cooling of TMP and PTR compressor

The cooling water flow rate for the PTR compressor (Figure 4.2) needs to be at least 10L per minute. This is because the PTR compressor produce lots of heat and the compressor itself needs to be maintained at room temperature for cooling of the PTR head to be effective.

This section is incomplete(future update/upgrade).

4.2 Downtime Maintainance

During the non-operation of the Triton dilution refrigerator, there is a few maintenance needed to be routinely carried out.

4.2.1 Pump Cycle Maintainance

The pumps and compressor in Triton requires regular maintenance which is carried out by the engineers from Oxford Instruments. It is possible to carry out the maintenance

Figure 4.1: Top left: Liquid nitrogen dewar and cold trap. Red-circled indicates the position of ventilation port that is used for refilling; Top right: Liquid nitrogen dewar used for transport and refilling; Bottom: liquid nitrogen transfer tube used together with the dewar on the top right.

by ourself but it is recommended that it is done by the experienced engineers from OI. The maintenance intervals for the compressor and pumps are listed in Table **??**. The cumulative operation time of the fore pump and PTR compressor can be obtained as described in Figure 4.4.

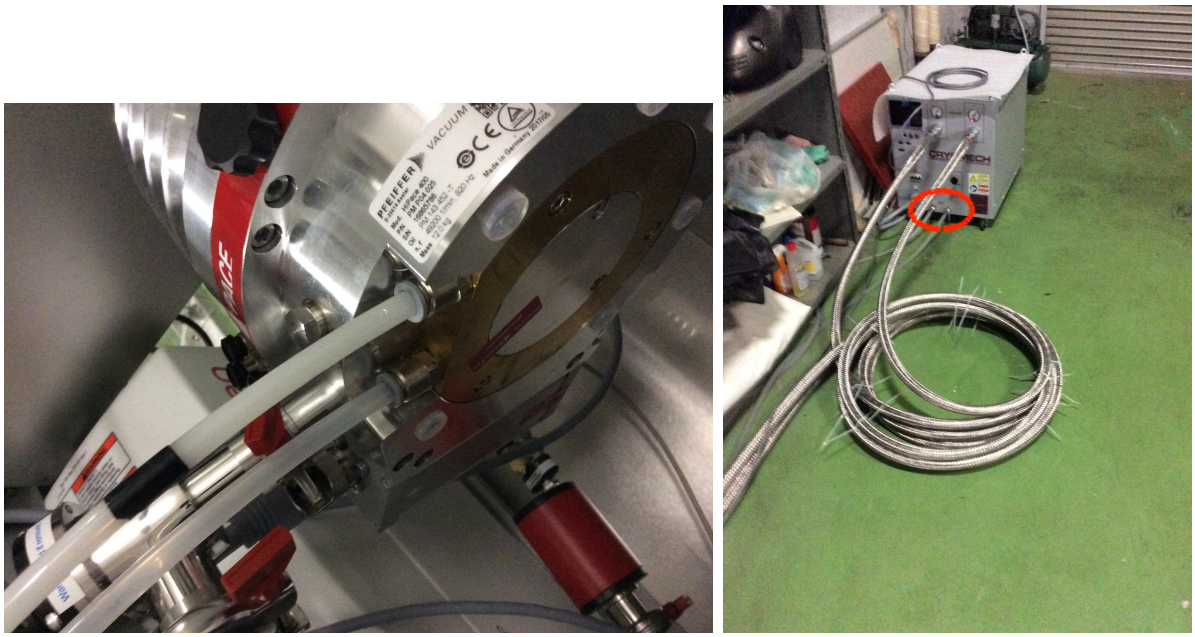


Figure 4.2: Left: cooling water inlet and outlet of TMP pump. (black taped tubing indicate outlet); Right: cooling water inlet and outlet of PTR compressor.

Component	Required Maintenance	Who Can Do This?	Frequency
PTR Compressor	Change Adsorber	User/ Service Engineer	20,000 hours
Pfeiffer Turbo pump	Change operating fluid reservoir and bearings	Oxford Instruments / Pfeiffer	4 years
Adixen Pump	Oil draining, bearing and seal replacement	Oxford Instruments / Adixen	22,000 hours or 4 years
KNF Pump	Change diaphragms	Oxford Instruments / KNF	8,000 hours

Figure 4.3: Maintenance interval for the various essential pump and compressor in the Triton system.*From Triton manual

Pump maintenance history

- No pump maintenance yet as of October 30, 2018.

4.2.2 Cleaning of Cold Trap* will update in future

When throughput put test has lower flow rate than expected, the most probably solution is to clean the cold trap or dilution unit. It is recommended that we clean the cold trap every $3 \sim 6$ months.

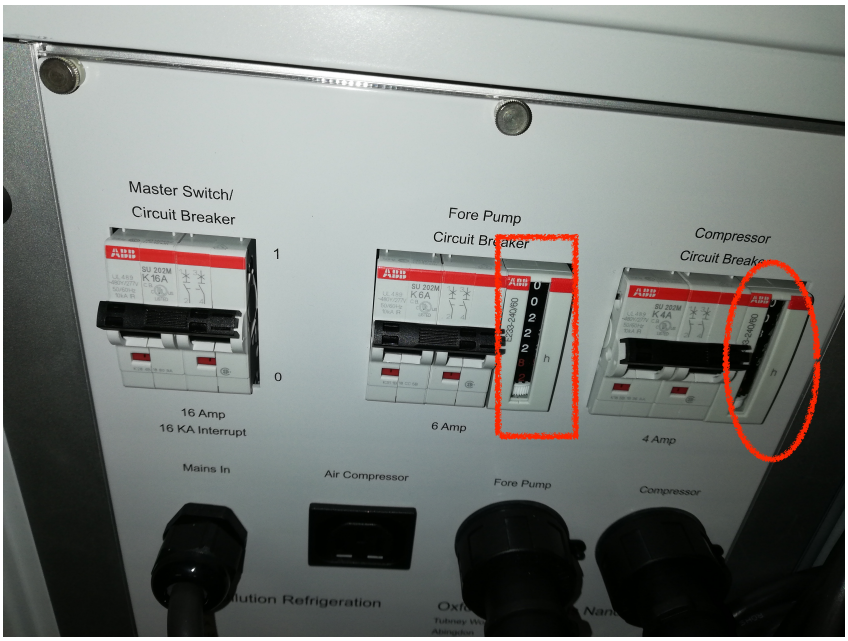


Figure 4.4: The fore pump and compressor cumulative operation time are indicate at the red circle/square. The number are in hours.

After ensuring mixture gas are all collected back in the storage tank, close all the valve of the Triton system. Detach the inlet and outlet from the cold trap and connect them together bypassing the cold trap.

Connect the cold trap cleaning tools to the cold trap and connect it to an external TMP pump. The cold trap is clean by vacuum the cold trap itself.

Figure 4.5: The tools and parts required for cleaning the cold trap.

4.2.3 Cleaning of Dilution Unit* will update in future

The cleaning principle for dilution unit is similar to the cleaning of cold trap.

The clean of dilution unit is done via V10. Before starting this procedure, please ensure

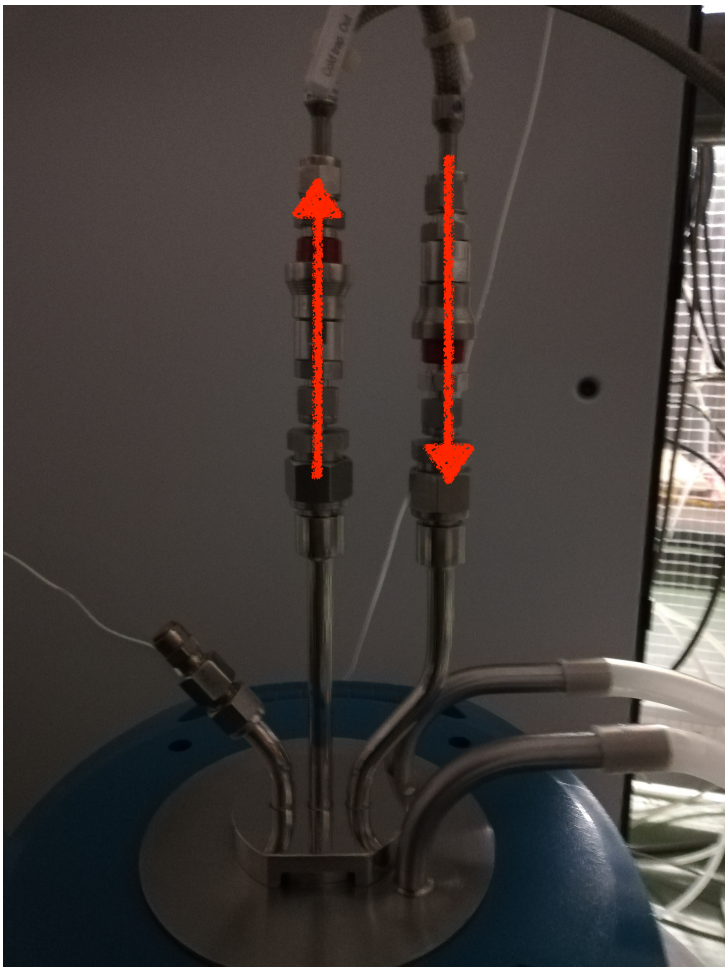


Figure 4.6: The tools and parts required for cleaning the cold trap.

that the mixture gas are recollected back into the storage tank. After confirming all the mixture gas is back in the storage tank, we can begin the procedure of cleaning the dilution unit. Close V14 valve before begin the process. This is to ensure the mixture gas is safely stored in the storage tank and not mistakenly removed.

This procedure requires us to connect an external TMP pump(Figure ..) to V10. First, ensure that V10 and V12 manual valve are open. Then proceed to ensure V9, V1 and V7 are closed. Leave V6 open. In this set-up, the components of the dilution unit are open to be vacuum by the TMP pump. By vacuuming the dilution unit over an extended period of time, the contamination can be remove from the system.

4.2.4 Changing of Water Filter Cartridge* will update in future

This section will be updated in future(after finer water filter is installed).

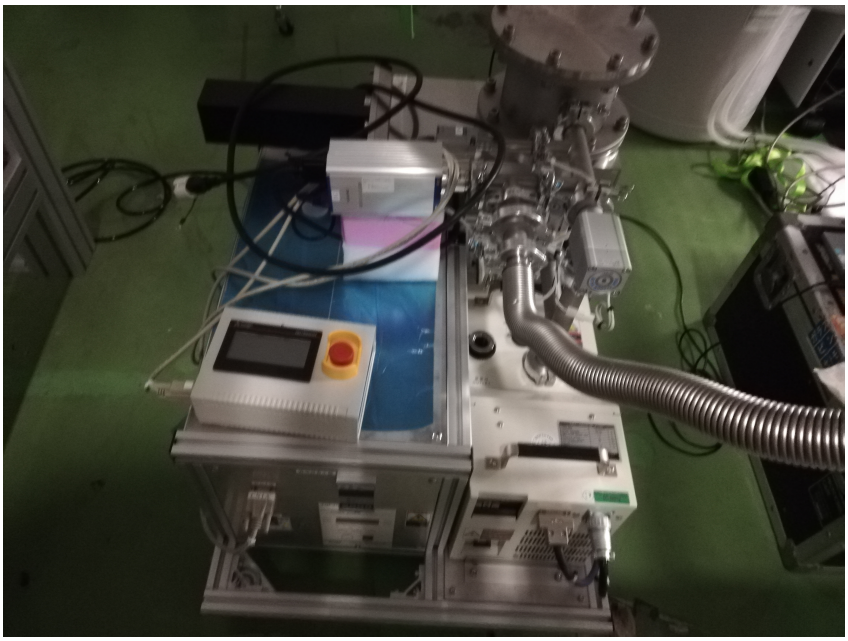


Figure 4.7: External turbo-molecular pump.

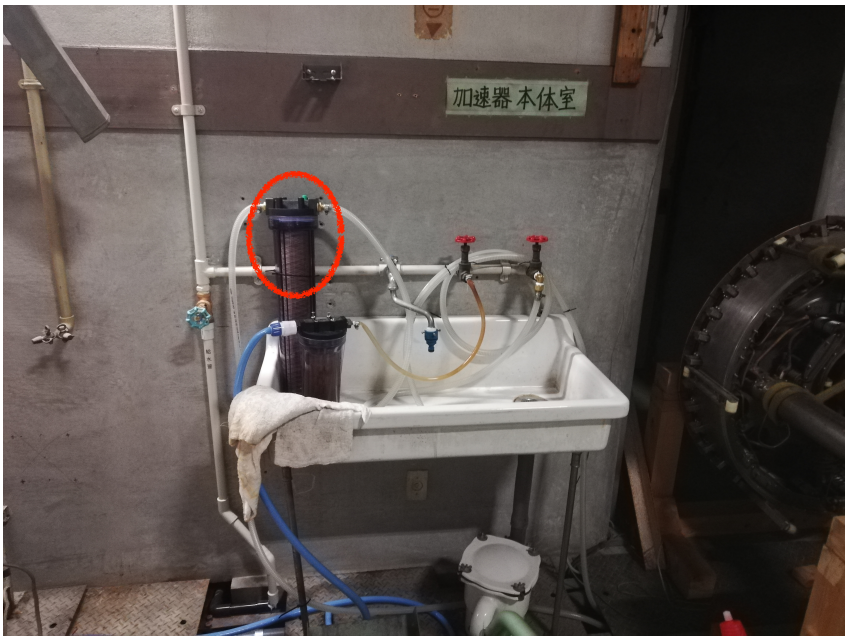


Figure 4.8: Water filter and water supply for cooling TMP pump and compressor.

4.3 In the Event of a Power Cuts* will update in future

This system currently does not have UPS(uninterruptible power supply) for the Triton's computer. Therefore for planned power cut, shut down the system appropriately.

Figure 4.9: Air compressor for the external TMP pump.

Figure 4.10: Lift trolley