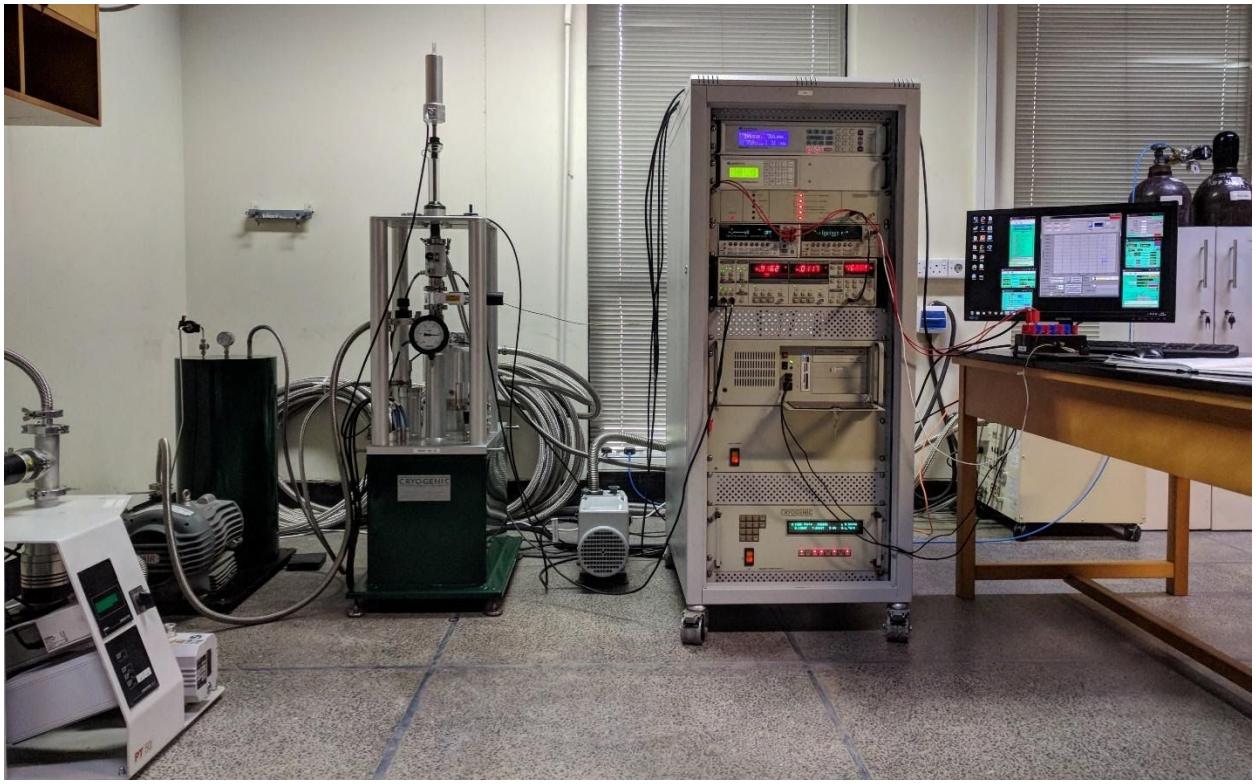
PhysLAB

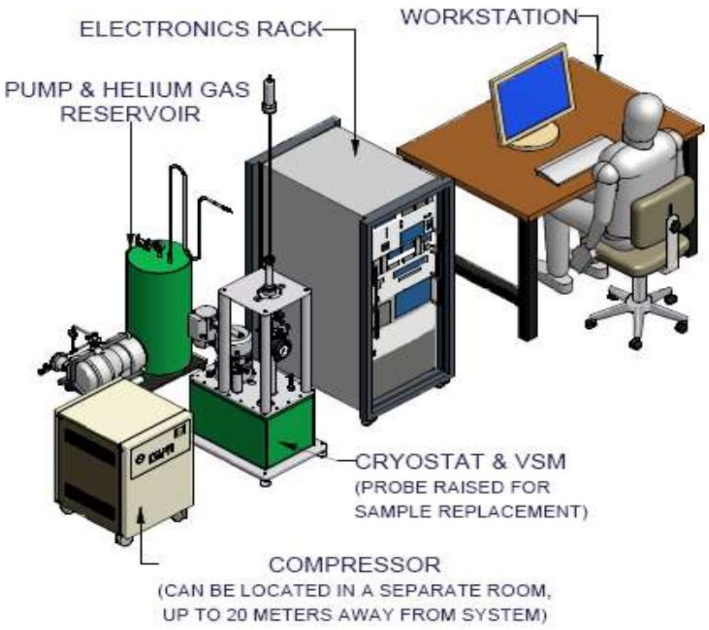
VSM Service Manual : Evacuating and Recharging the System

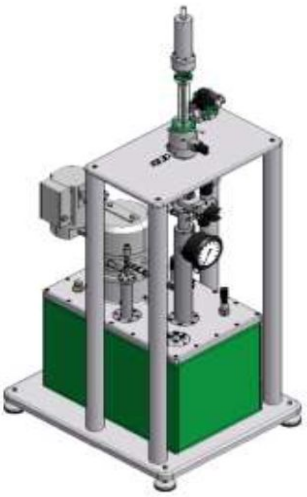
(Version: VSM-2020-I)

18 July, 2020

Ghulam Sarwar, Dr. Ali Akbar, Dr. Murtaza Saleem, Dr. Sabieh Anwar







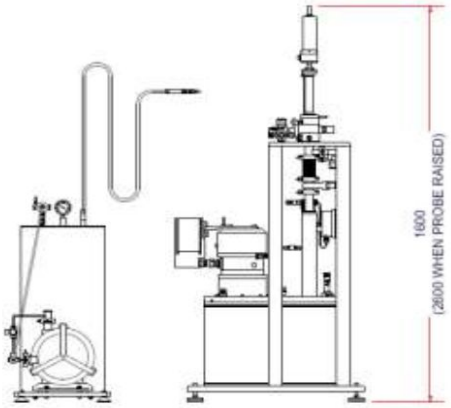


Table of Contents

Evacuating and Recharging the System 1
Switching off the system 1
Pumping out the vacuum chamber of Cryostat 2
Evacuating the VTI Circuit 4
Evacuating and Recharging DUMP 6
Starting the system 7
VTI checks 7
Compressor and Coldhead 8
Starting the Cooldown 8
Once the system is cold 9

Evacuating and Recharging the System

Different types of the problem may arise during the use of the VTI. Common problems are

- The VTI may not reach base temperature.
- The flow rate may not respond to the needle valve and/ or be limited.
- The flow rate may decrease at high temperatures or collapse completely at low temperatures.

If VTI is contaminated and is either partially or fully blocked then it is required to evacuate the VTI and hoses and recharge the dump. This process is best done when the Measurement System is at room temperature, but can be done while the system is above 77K. **If a sample is loaded it should be removed.**

The first step is to warm up the system as described below.

Switching off the system

There are a few steps that must be taken to ensure that the system can be safely restored to room temperature.

1. Turn off the magnetic field to 0 from the magnetic power supply window.
2. Remove the probe by using auto “Sample Removal” steps from “Main Menue” window.
3. **Fully open the VTI needle valve. Leave the V11 & V12 open** and switch off the oil-free pump by pressing the red STOP button as in figure 1. It will let the **helium expand back into the DUMP** through the self-sealer at the inlet.
4. Switch off cryocooler compressor by turning the position to 0 from Drive as in figure 2.
5. Wait 15 minutes then switch off the chiller.

The system will begin to warm up immediately. It is recommended that the system be left to warm to room temperature naturally for **36 – 48 hours**. The warm up can be monitored in the same ways as the cooldown. The system can be cooled down again without waiting for the system to return to room temperature.

Figure 1. To turn OFF the the Oil free pump, press stop

Figure 2. To turn OFF the Cryocooler Compressor, press 0 under DRIVE (double tick)

Precautions

- Do not disconnect the hose, from the helium dump to the helium inlet at VTI circuit, unless the system is at room temperature or the helium has been pumped out from the VTI circuit.
- Never disconnect the compressor hoses unless the system is at room temperature.
- Do not be tempted to allow air into the vacuum space in order to warm the cryostat more rapidly. If done carelessly it can cause serious damage.

Pumping out the vacuum chamber of Cryostat

At room temperature, the materials used in the construction of the cryostat desorb gases (“outgas”). This does not indicate a leak but does mean that you should pump out the vacuum chamber each time the system has been warmed to room temperature. Connect the vacuum pumping port Number 8 in figure 3 to Turbo Molecular Pump capable of a pumping speed of 50l/sec and base pressure below 10-5 mbar. Leave the system for overnight.

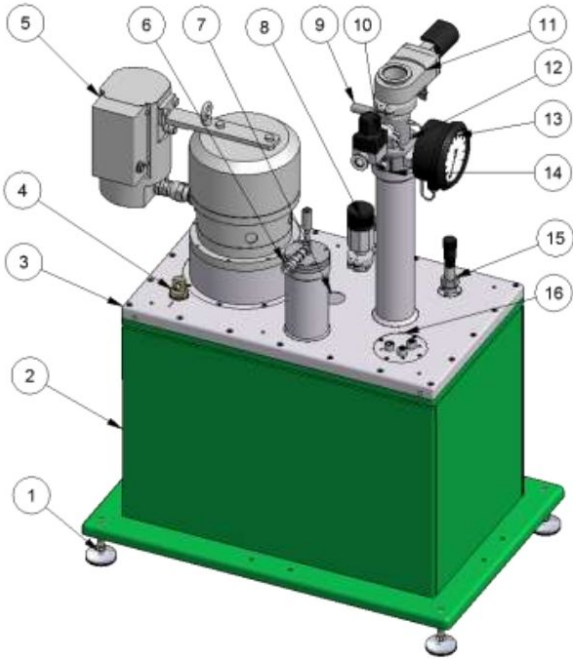


Figure 3.The Cryostat a vacuum insulated Chamber

Operational Steps

1. Close the cryostat pump out valve at port number 8 in figure 3.
2. Switch ON the rotary pump by turning the knob at the position of RP from PT 50 for 15 minutes or until the pressure reaches less than 10^{-2} mbar as in figure 4.
3. Open the cryostat pump out valve at port number 8 in figure 3.
4. When the pressure is maintained at 10^{-2} mbar, switch ON the turbo molecular pump (TMP) by turning the knob at the position of TMP from PT 50. The pressure should reach in the range of 10^{-6} mbar. It will take about 8 hours. Keep it ON and start evacuation of VTI.
5. When the evacuation and cleaning is completed, close the cryostat pump out valve from port number 8 in figure 3 and turn Turn OFF TMP.
6. After 20 minutes, Turn OFF RP from PT 50.

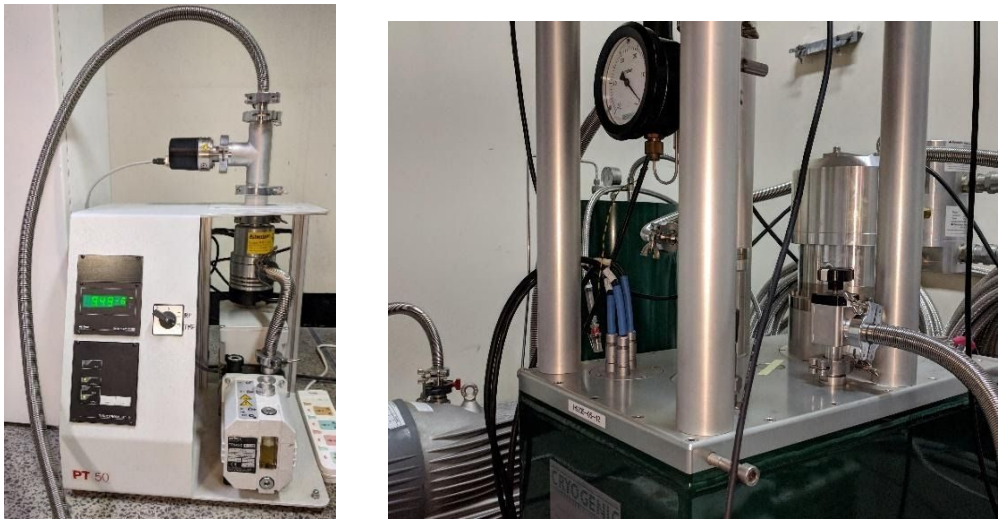
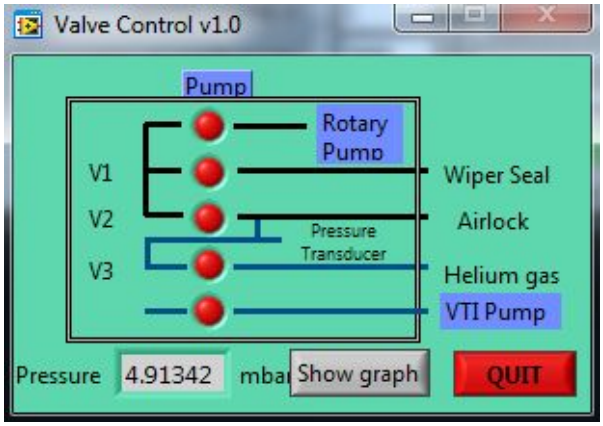


Figure 4. Turbomolecular Pump (TMP) PT 50 and the port8 at cryogenic

Evacuating the VTI Circuit

1. Switch off the oil free pump manually.
2. Switch off the rotary pump from the "valve control VI". V1, V2 and V3 should be closed.



3. Close the valves V11, V12. V13 and V16 were already closed if not close them.
4. Disconnect the helium inlet hose and protect it with a cover as in figure 5. To disconnect, pull back the outer sleeve whilst withdrawing the stem.

Figure 5. Protecting helium inlet hose with cover

5. Fully open the needle valve.
6. Disconnect the Airlock vacuum hose from its usual position located at the back of the gate valve.
7. Attach the adapter to this hose as shown in figure 6.

Figure 6. The adapter to be attached the hose.

8. Attach this adapter to the **helium gas inlet** port (position a in figure 7). Now the rotary pump from the valve block within the electronic rack is connected to the helium inlet port.

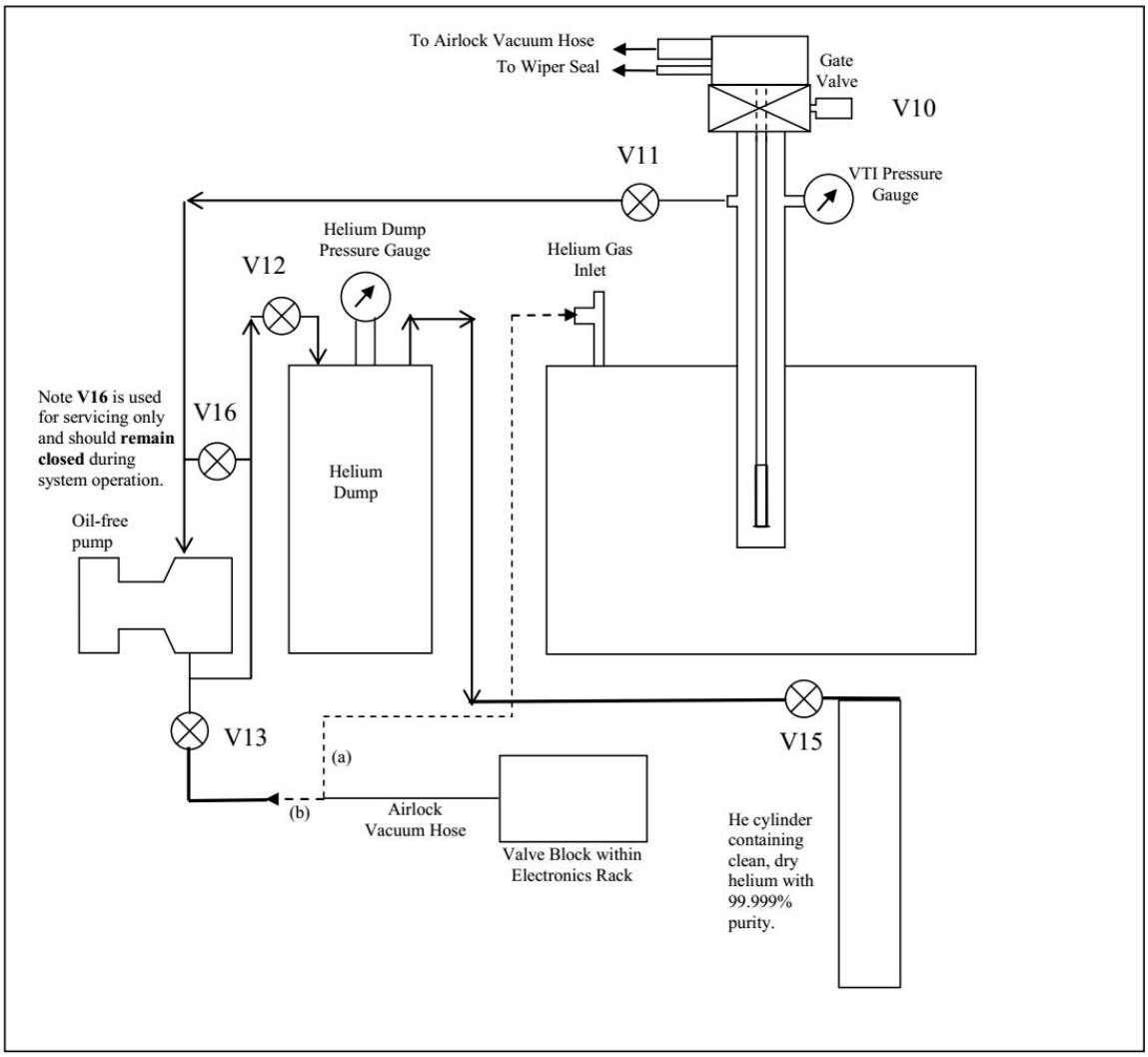


Figure 7. Block diagram for evacuation and recharging.

9. Start the rotary pump and open the airlock from the "valve control VI" window as shown in figure 8. Evacuation starts from the helium inlet port of VTI Circuit.

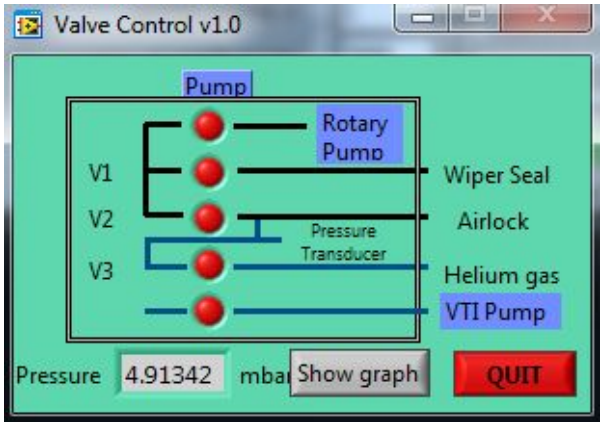
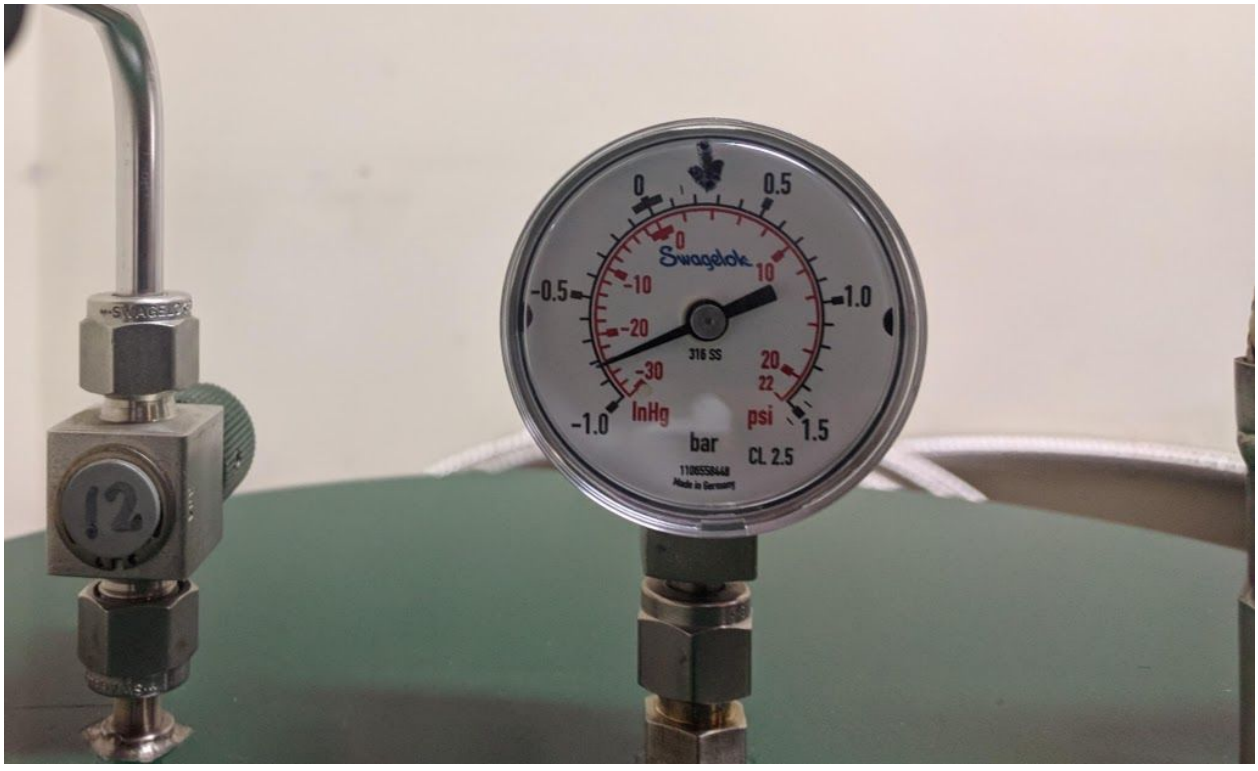


Figure 8. Valve control VI.

10. Open V13, turn ON the ‘Oil-free pump’ manually from a switch located on its body by pressing the green button as shown in figure 1, and then open V11. Evacuation starts from the top of VTI circuit through V11 by the scroll pump. At the start the VTI gauge will be above 50 but after a few minutes it should move towards 0 mbar.
11. Leave both the rotary and oil-free pumps running for at least 6 hours (6 hrs are recommended but should be more than 12 hours). This will ensure that the contamination is removed from the **VTI pipework and charcoal**.

Evacuating and Recharging DUMP

1. Close V11 and switch off the oil free pump. Close air lock V2 and turn off the rotary pump from the valve control window.
2. Disconnect the Airlock vacuum hose from the Helium inlet port. Remove its red adapter as shown in figure 6 and connect it at its usual location at the back of the gate valve.
3. Close the cryostat pump out valve at port number 8 in figure 3.
4. Disconnect the vacuum hose from this cryostat pump out port number 8 and connect it to the port V13 (position b in the block diagram of figure 7).
5. Keep the oil free pump switched off and rotate the knob to the RP position to start the rotary pump from the Turbo Molecular Pump PT 50 as shown in figure 4. **Don’t rotate the knob to the TMP position at this stage.**
6. Open V13, it allows the pump PT 50 to be used for evacuating the DUMP.
7. Open V12 very slowly and observe the helium dump pressure gauge. It should begin to decrease in pressure. Evacuation will take about 60 minutes.
8. When the dump has been fully evacuated and its gauge shows a reading of -1.0 bar, then close V12 and V13.



9. Close V15 and disconnect the helium hose from the helium port at the rear of the valve block in the instrument rack.
10. Attach an adapter to this helium hose coming from the helium cylinder as in figure 9. Connect this Helium hose to the DUMP. Keep V15 closed.

Figure 9. Parts to connect the adapter coming from the helium cylinder

11. Gradually adjust the helium cylinder regulator to a pressure above 1.25 Atmosphere.
12. Flush the helium dump with helium gas by opening V15 and allowing the helium dump pressure gauge to rise to at least -0.5 bar then close V15.
13. Fully evacuate the helium dump again by following steps from 5 to 8 and then move to step 14.
14. Finally close V12 and V13. Open V15 and fill the dump to operating pressure, typically +0.25 bar. Close V15 when helium DUMP reaches the pressure +0.25 bar below the black arrow marker.
15. Disconnect the helium inlet hose of the DUMP from the helium cylinder. Remove its protective cover.
16. Close V13 and switch off the rotary pump from the turbo molecular pump.
17. Reconnect the helium hose from the helium gas cylinder to the helium port in the valve block of the instruments rack. Adjust the helium pressure. Now the system can be started as usual.

    When finished the V11, V12, V13, V16 should be closed and the needle valve should be fully opened.

Starting the system

Before the system is cooled down a few checks need to be done as described in chapter 6 on page 57 of the mini crygen-free user manual. However, VTI check is explained here.

VTI checks

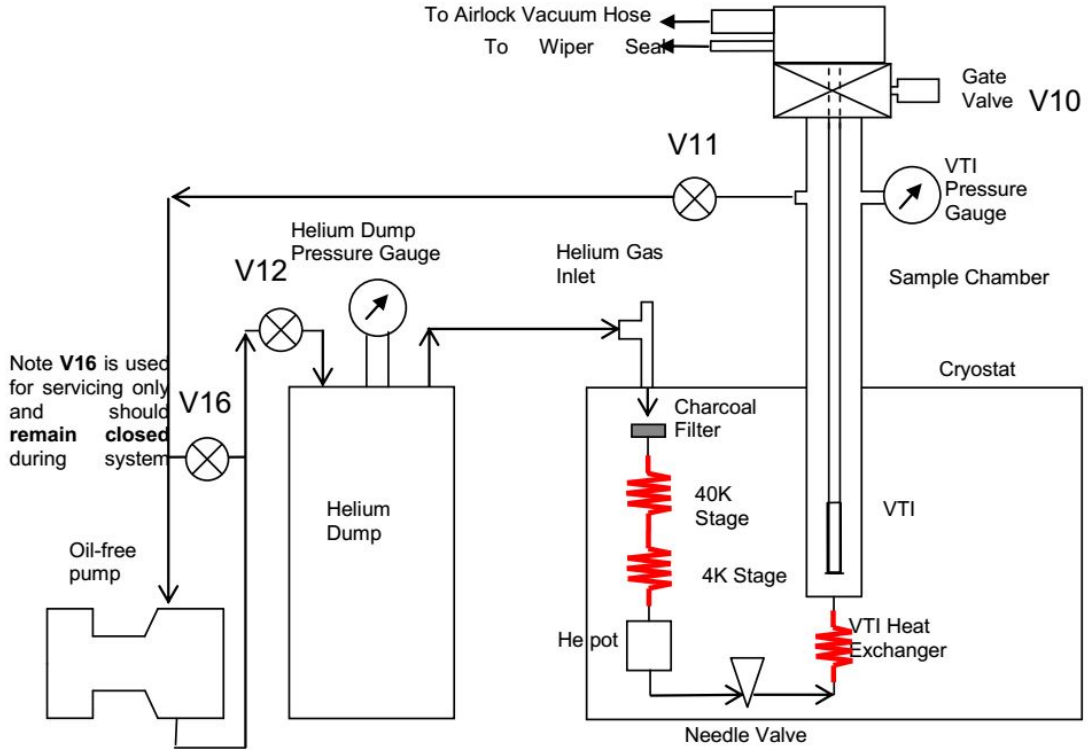


Figure 10. Schematic diagram of the VTI cooling circuit.

- Check that the set-up matches the arrangement depicted in Figure 10.
- Check that the helium dump is properly connected to the cryostat using the small diameter reinforced stainless steel hose. The helium inlet uses a self-sealing connector, **to connect**, push the stem into the helium gas inlet until it clicks. **To disconnect**, pull back the outer sleeve whilst withdrawing the stem.
- Check that the by-pass (valve 16) on the oil-free pump (identified in the figure 10) is closed.
- Switch on the oil-free pump and check that it is possible to control the pressure (on the VTI pressure gauge) using the needle valve. With the needle valve fully open it should be possible to achieve a pressure in excess of 30 mbar while the gas is circulating. This test checks that the flow impedance is correct and there are no obstructions in the circuit.
- Leave the needle valve open, switch off the pump and close V11. This ensures that the helium from the external gas reservoir condenses in the helium pot during cooldown and that the helium only circulates in the one direction.
- Check that the external helium gas reservoir (”dump”) is charged to the correct pressure, approximately 1.25 bar absolute (0.25 bar on the gauge) with clean dry helium gas (99.99% purity recommended). The pressure gauge on the helium reservoir reads the pressure relative to atmospheric pressure rather than absolute pressure relative to vacuum. Hence, atmospheric pressure is 0 bar and vacuum is -1 bar.

Compressor and Coldhead

Never disconnect the compressor hoses unless the system is at room temperature. Check that the helium gas pressure in the Cryo-cooler compressor is at 1.7 Mpa (figure 11).

Figure 11. helium gas pressure in the Cryo-cooler compressor gauge.

Starting the Cooldown

The software provides a convenient way to log the temperature of various parts of the system during the cool down process, confirming normal operation. There are two ways of recording the cooldown, the background temperature logger or to run a sequence. After these checks have been made and any problems identified and corrected the system may be started. To do so:

1. **Ensure that V11 at the top of the VTI and the V12 are closed.** (While shutting down VSM V11 and V12 must be opened)
2. Leave the **needle valve fully opened**.
3. Ensure that the **scroll pump is off**. (V12 must be opened if the scroll pump is ON otherwise the scroll pump may damage. If scroll pump is to start then first open V12 then open the scroll pump)
4. Ensure that chillar is started and adequate cooling water is being supplied to the compressor. Check the water level.
5. After 20 minutes switch on the compressor. Temperature reading should show less than 14C^{o}.
6. Indications of normal cooldown are as follows:
    a. The cryocooler will make a “chirping” sound.
    b. Temperatures within the system fall steadily. To view a plot of the temperatures click on the View data option in the main menu (see section 6.4.6). You will be supplied with a typical cooldown plot for reference.

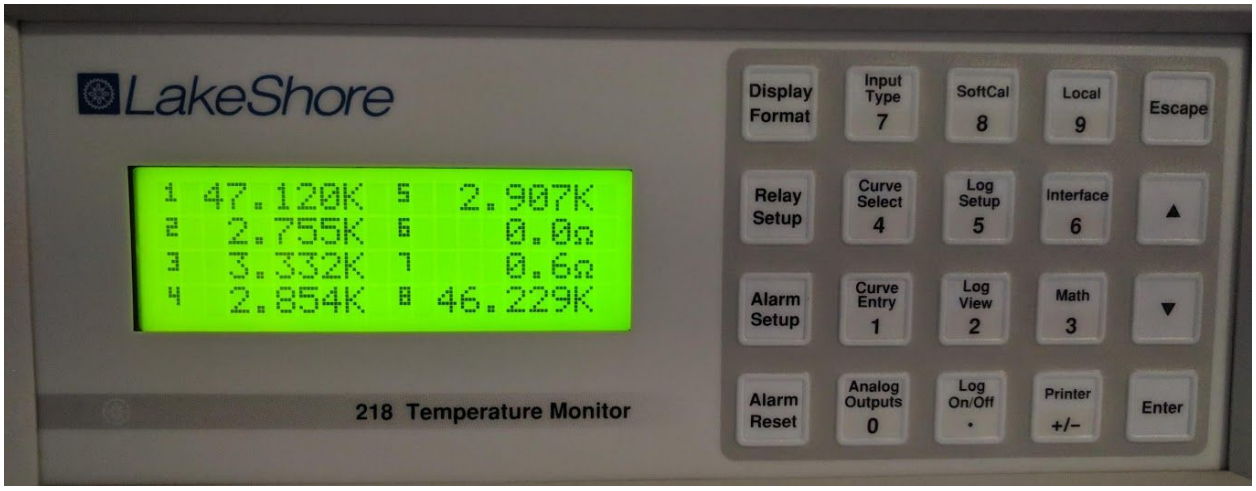
As the second stage reaches 4K, the gas pressure in the helium dump will fall towards its base value (about 0.2bar) as the helium condenses in the helium pot.

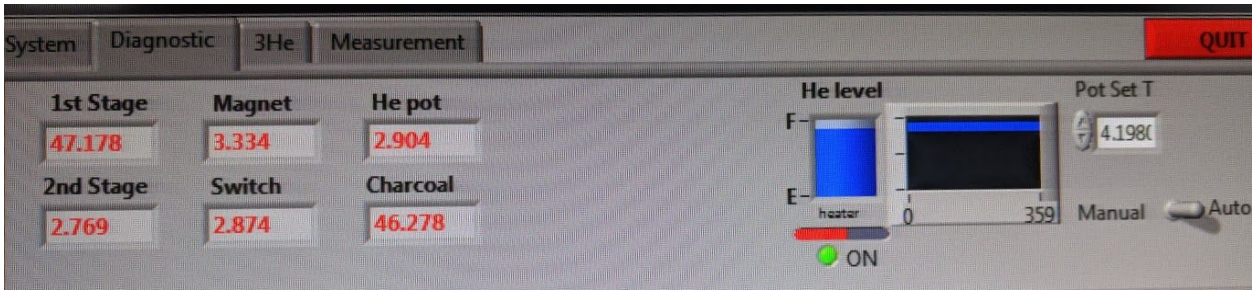
Once the system is cold

Once the system has reached operating temperature as shown in Table below then the VTI circulation may be started. The helium pot will be the last part of the system to reach operating temperature. As the pot reaches 4.2K the helium will condense and form liquid, as this happens the pressure in the

helium reservoir will drop during normal use the pressure in the reservoir will vary according to the VTI temperature as explained in 4.4.2 on page 19.

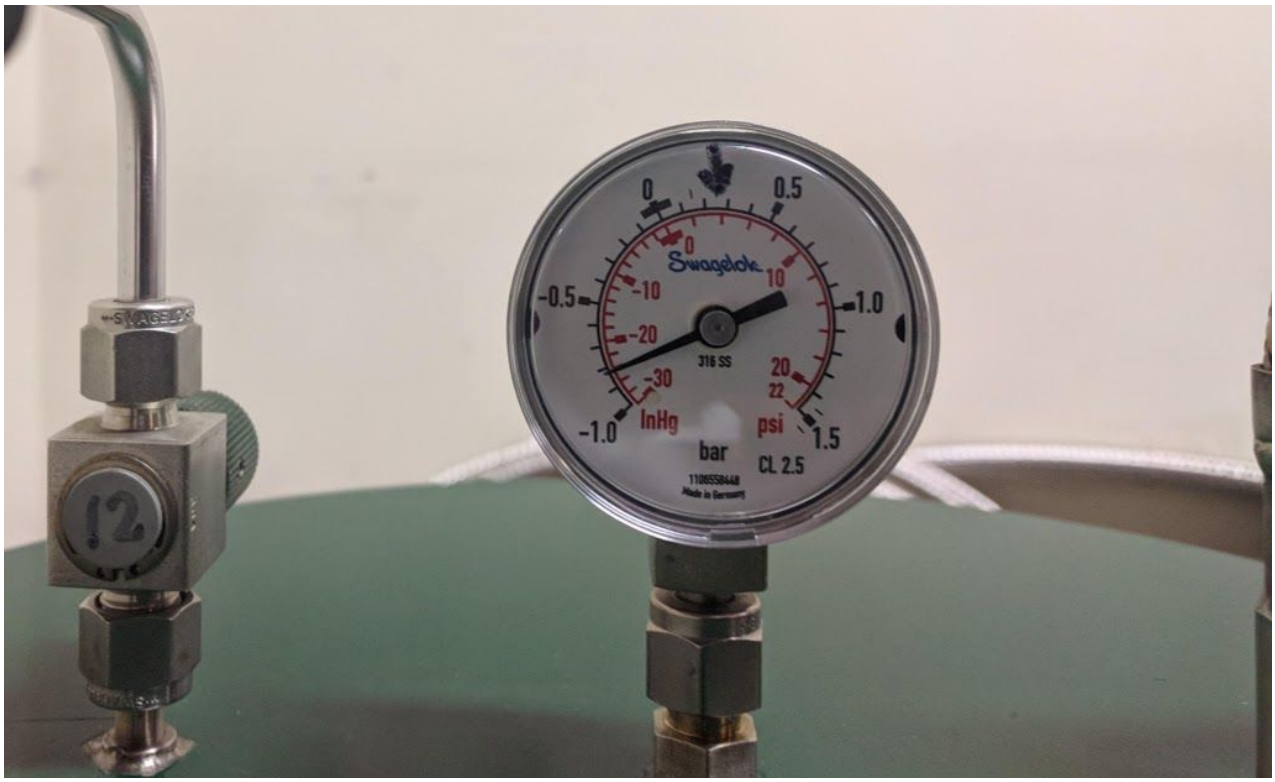
Once the cooldown is achieved, the following will be the status of temperature monitor, temperature controller, VTI gauge and the dump gauge. Helium post will be full.






LakeShore
Input A
298.021 K
Input B
281.014 K
SETPOINT
P
I
D
ALL OFF
Model 350 Temperature Controller
LakeShore
218 Temperature Monitor
Position
Move
Connection
Limit 1
Limit 2
Centre
VALVE CONTROL
Rotary Pump
V1
Wiper Seal
V2
Airlock
Pressure Transducer
V3
Helium Cylinder
Oil - Free Pump


VII
30
20
mba
40
10
50
0
CRYOGENIC



To start the VTI circulation or Start the Scroll Pump:

1. Make sure that valves 11 and 12 are open. V12 is located at the exhaust of the VTI and the top of the helium reservoir or Dump. **If valve 12 is left closed when starting the pump, the pump will be damaged** by the high pressure at the exhaust of the pump.
2. Ensure the bypass valve 16 and V13 are also closed.
3. Switch on the oil free scroll pump.
4. Use the needle valve to set an appropriate flow rate, typically about 8-15 mbar.
    a. If the helium flow rate is too low you will not reach the base temperature of the VTI.
    b. If the flow rate is too high the cryo-cooler 2nd stage, magnet and helium pot temperatures will rise. This limits the maximum ramp rate of the magnet and result in unstable performance of the VTI.

Once the flow is established and stable the system can start to be used. There is no need to wait for the VTI to reach base temperature. The following should be the achieved values of the helium pot, temperature controller, temperature monitor, VTI pressure gauge and the dump gauge.

To STOP Scroll pump temporarily

1. Close V11
2. Turn OFF Scroll Pump
3. Close V12
4. Fully open needle valve

Sometimes it is required to temporarily stop the scroll pump due to electricity failure for a short interval. If the temperature loss of different stages is minor and we and we want to recover it without cleaning then the scroll pump may be turned OFF until the required temperature is not attained. The bypass valve 16 and V13 must always be closed until cleaning is required.

To START Scroll pump when cooling is attained

1. Open V12
2. Turn ON Scroll Pump
3. Open V11
4. Open the needle valve and adjust the He flow

To remove the Scroll Pump

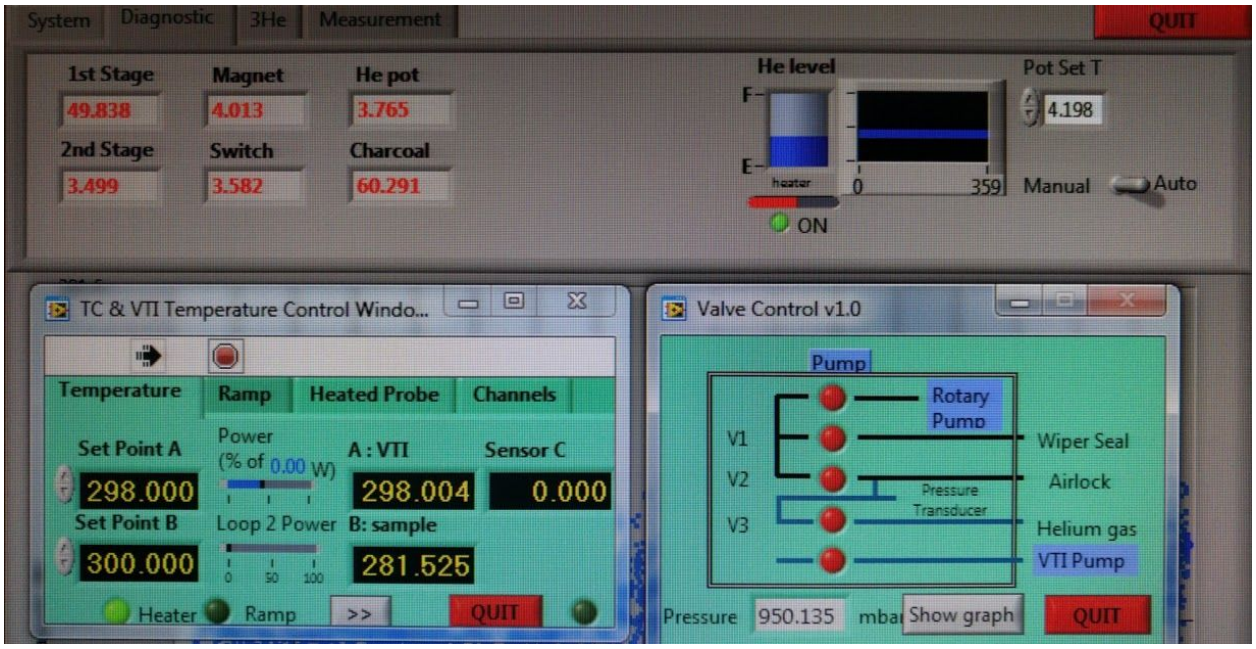
We need to shut down the VSM and follow the protocol from Switching off the system. Remove, repair and then attach the pump. Follow the steps from Pumping out the vacuum chamber of Cryostat to Once the system is cold. We have to follow these steps to maintain the pressure of He within the DUMP at 0.25 bar, the arrow marker on the DUMP gauge.

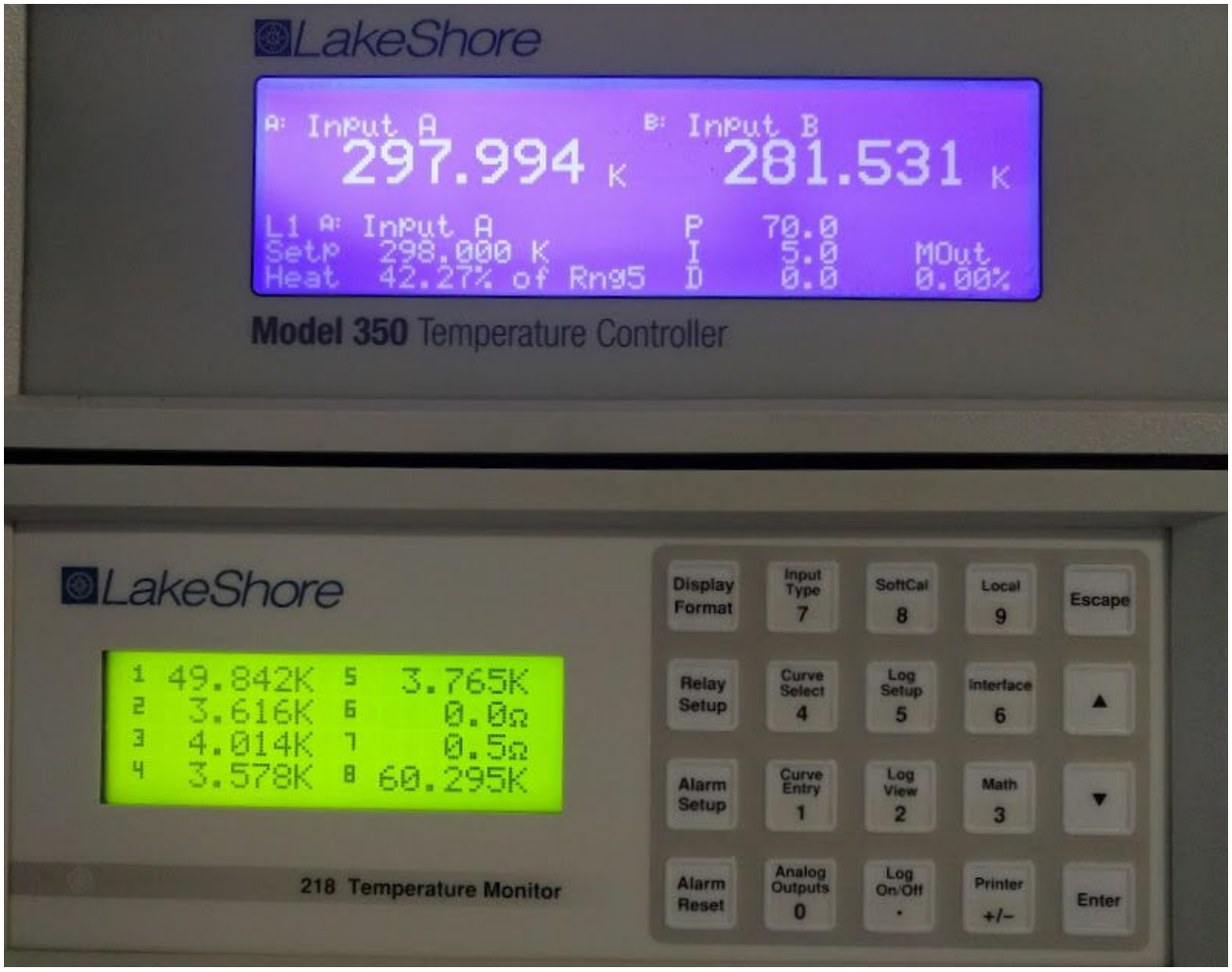
Alternate

If it is required to remove the scroll pump while keeping the whole setup operational, follow these steps.

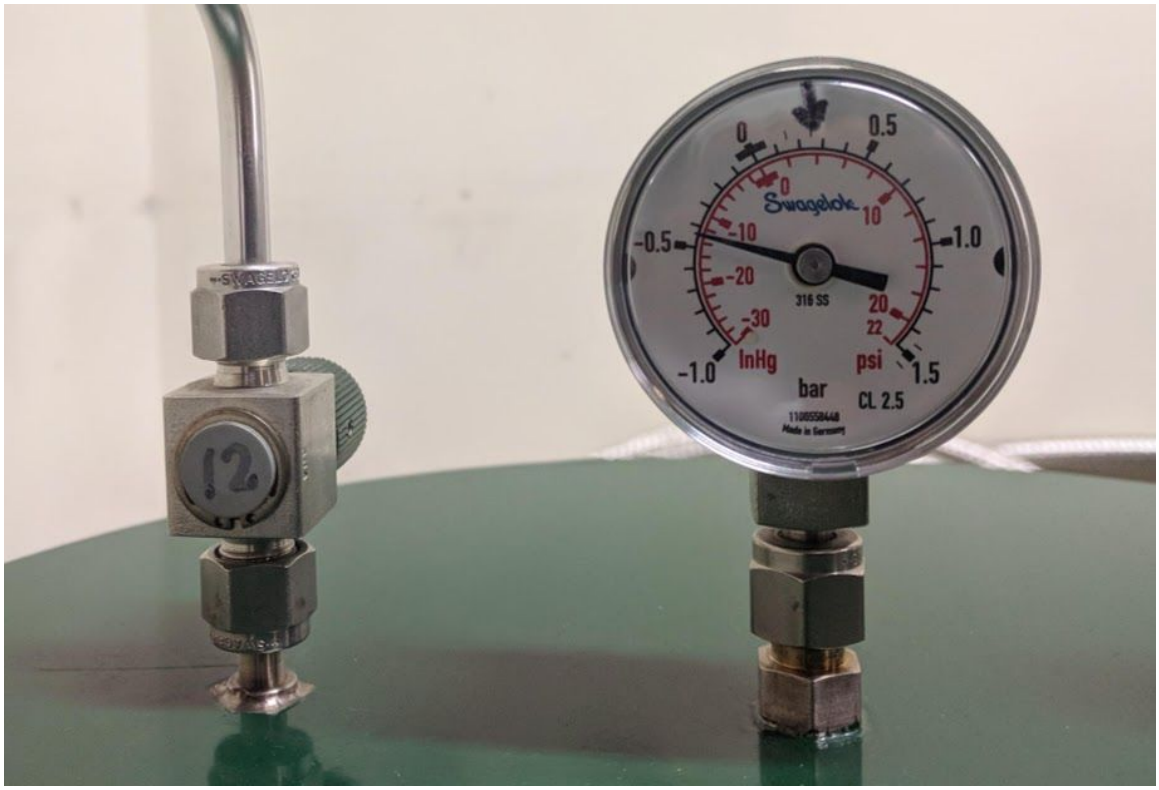
1. Close V11
2. Turn OFF Scroll Pump
3. Close V12
4. Fully open needle valve
5. Wait till the He pot is completely filled
6. Open V12
7. Turn ON the scroll pump for 10 minutes. It will shift the He gas within pipeworks and scroll pump to the DUMP.
8. Close V12
9. Turn OFF scroll pump and remove it for repair.
10. Attach the pump.
11. Open V12
12. Turn ON Scroll Pump
13. Open V11
14. Open the needle valve and adjust the He flow

When system is operational and normal the following should be the readings



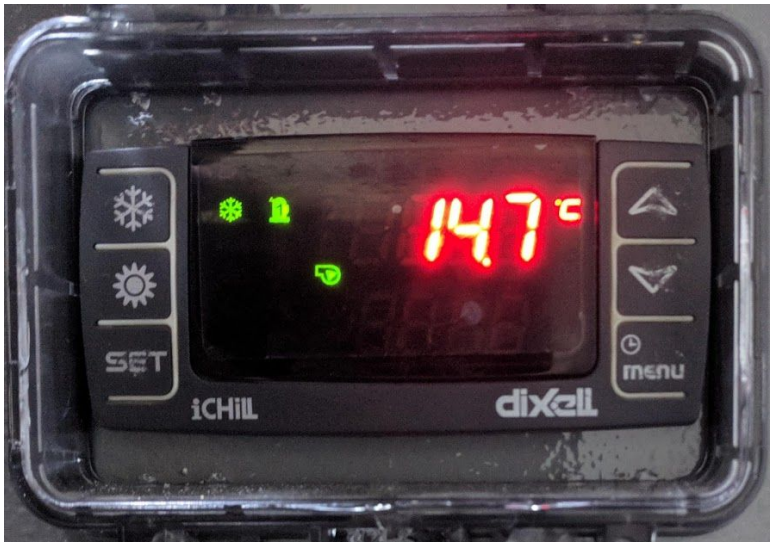

LakeShore
A: Input A
297.994 K
B: Input B
281.531 K
L1 A: Input A
Setp 298.000 K
Heat 42.27% of Rng5
P 70.0
I 5.0
D 0.0
MOut
0.00%
Model 350 Temperature Controller
LakeShore
1 49.842K
2 3.616K
3 4.014K
4 3.578K
5 3.765K
6 0.0Ω
7 0.5Ω
8 60.295K
218 Temperature Monitor
Display Format
Input Type 7
SoftCal 8
Local 9
Escape
Relay Setup
Curve Select 4
Log Setup 5
Interface 6
▲
Alarm Setup
Curve Entry 1
Log View 2
Math 3
▼
Alarm Reset
Analog Outputs 0
Log On/Off .
Printer +/-
Enter


VII
30
20
mbar
40
10
50
0
CRYOGENIC
JEWELLED MOVEMENT


0
0.5
Swagelok
-0.5
1.0
-10
10
-20
316 SS
20
-30
22
-1.0
inHg
psi
1.5
bar
CL 2.5
12

The F-50 Cryogenic Sumitomo Helium pressure should show these readings of helium supply pressure and the return pressure.

The temperature controller of the Chiller iC220 ICS Cool energy should show the reading about 15C.



When HVAC started the the temperature controller of the Chiller iC220 ICS Cool energy attained this value, which is the best possible value ever achieved.

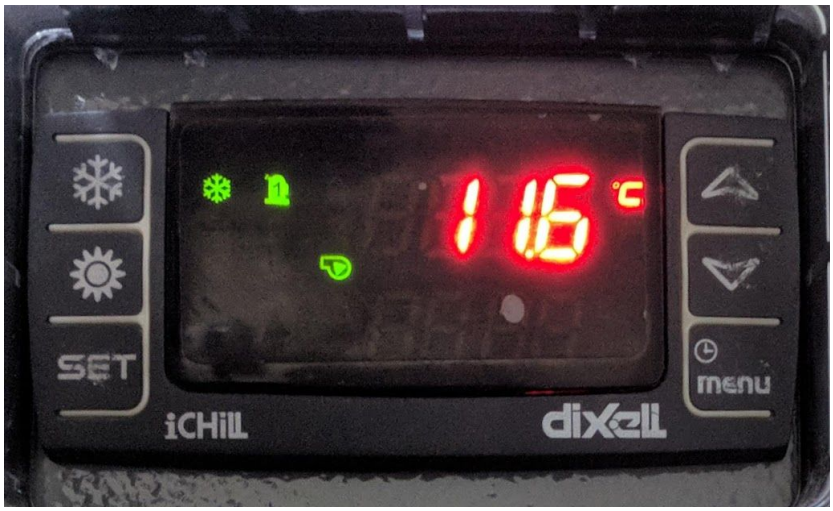


Table. Typical temperature locations

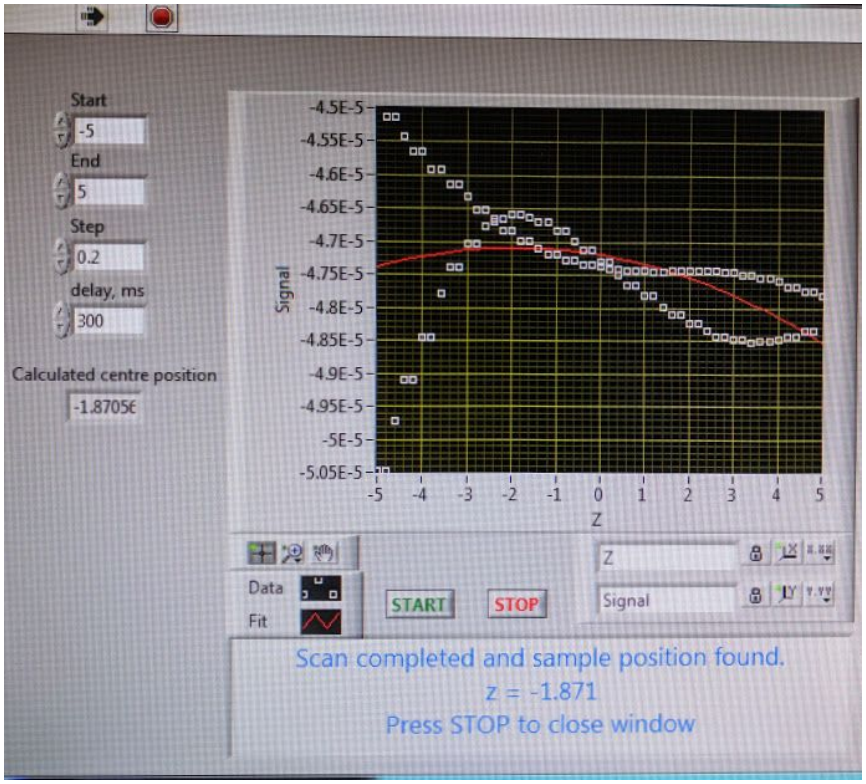
Location	Typical Temperature	Measured by	Type
Cryo-cooler 1st stage	45-50 K	Temperature Monitor channel 1	CCS
Cryo-cooler 2nd stage	3.0-4.5 K	Temperature Monitor channel 2	CCS
Magnet winding	3.0-4.5 K	Temperature Monitor channel 3	CCS
Persistent mode switch	3.0-15 K	Temperature Monitor channel 4	CCS
Helium pot	3.0-4.2 K	Temperature Monitor channel 5	CCS
Cryo-pump	3.0-4.5K (0.2 Ω)	Temperature Monitor channel 7	PT100
Charcoal filter	50-90 K	Temperature Monitor channel 8	CCS
VTI Heat exchanger	1.6-325 K	Temperature controller channel A	Cernox
Sample	1.6-325 K	Temperature controller channel B	Cernox

1 atm = 760 torr = 1.01325 bar = 101325 pascal = 14.695 pound-force per square inch

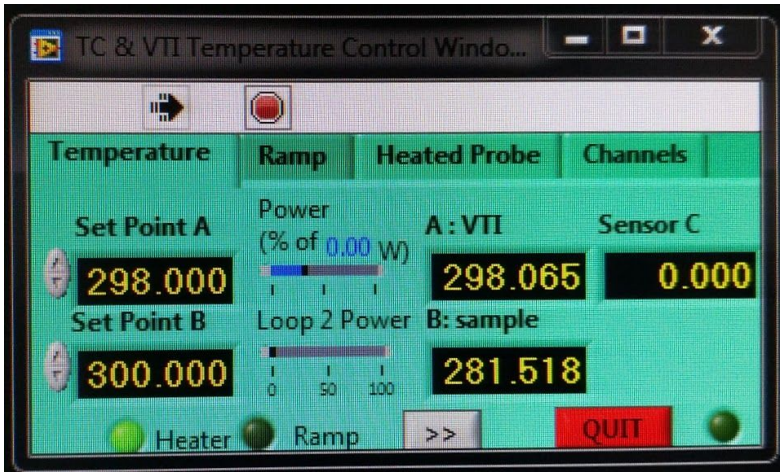
Status of temperature monitor after loading the Ni Standard Sample.

Report

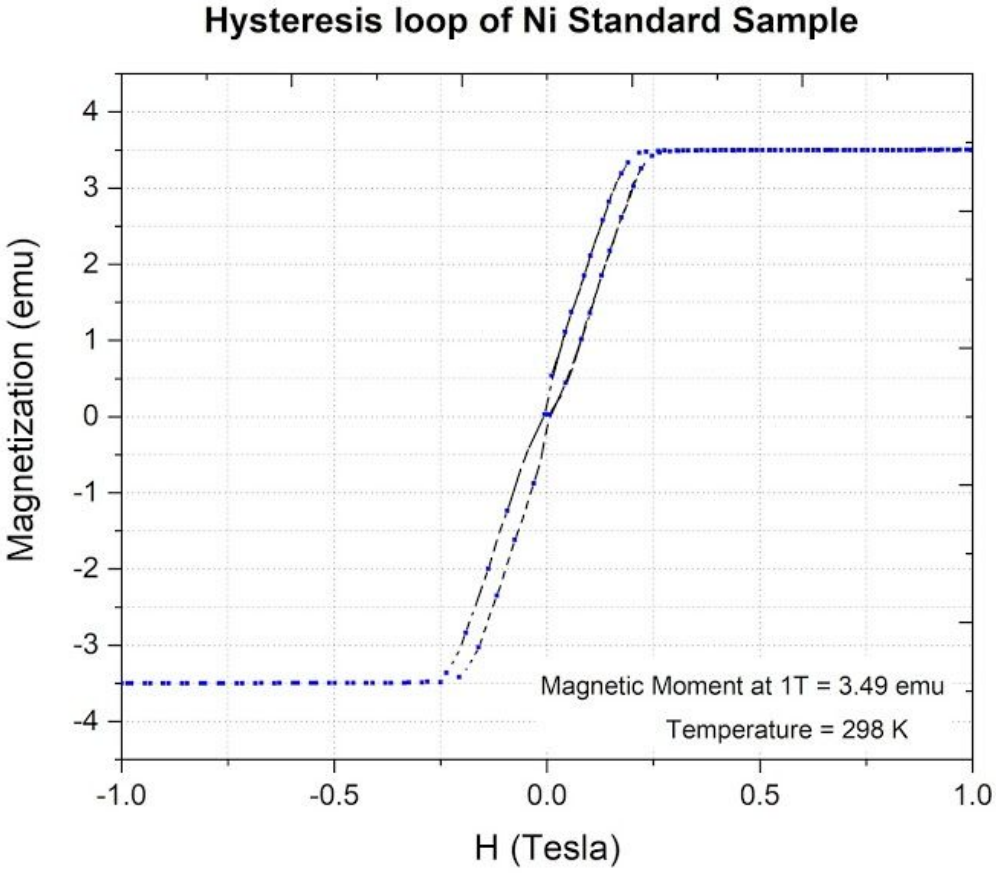
1. Previous Calibration file is loaded. Before this calibration the manual centring was performed to detect the sample at the maximum x-Value of LIA. The auto centring is performed successfully. The calibration is performed. The measured emu value is 3.48 at 1T. Now this file is saved and reloaded after the restart of VSM.
2. The Ni standard sample is loaded again and the manual centring is performed at 0.1T. The measured emu value is 3.437 at 1T and 298C. The phase angle is 168.11
3. Auto centring is performed successfully at **0T with the first attempt** at the same location of step-2

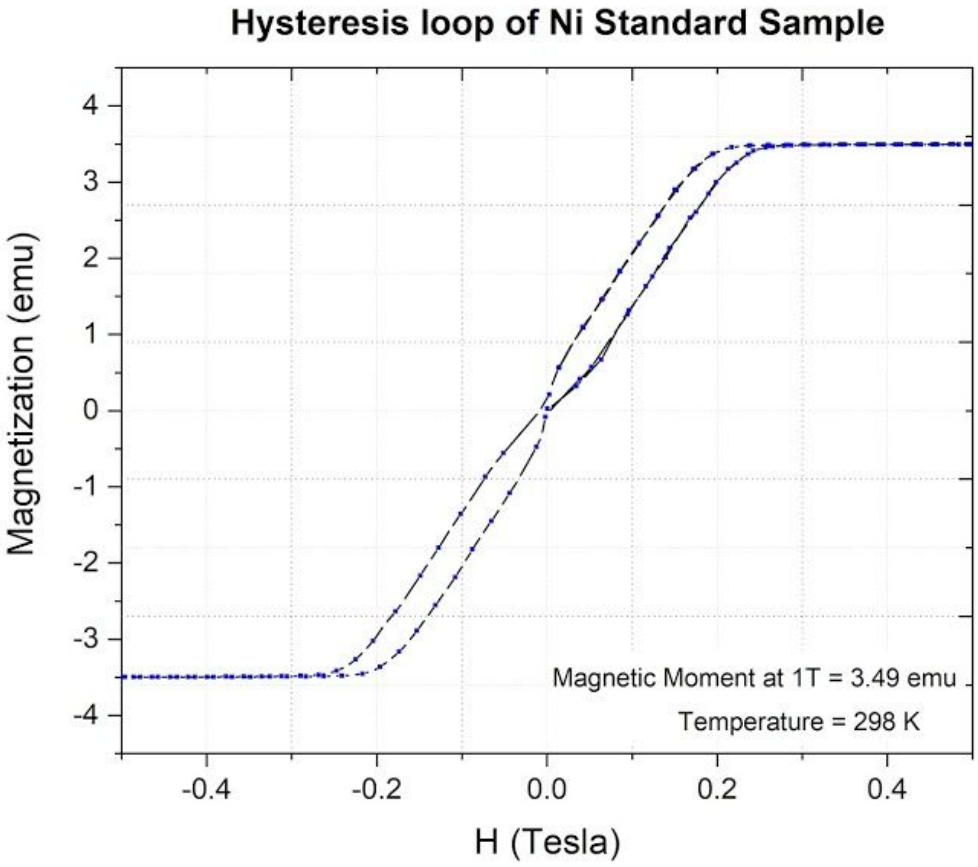


4. The new emu value is 3.339 which is lower than the previous value of manual centring.
5. Now the calibration performed at 1T and these temperature conditions.



6. The measured emu value is 3.49 at 1T and 298C. The phase angle is 168.51. This phase angle again takes the value of 168.11 and the emu value becomes 3.60 at 1T and 298C. No change was made in anything during this change in emu value. emu value changed in two hours.
7. The calibration is performed again and emu value is again at 3.49 and the phase angle at this moment is 168.11 and sample movement amplitude is 4.03mm.
8. The hysteresis loop of the standard Ni sample at 1 Tesla and the 0.5 Tesla.
9. A detailed report about centring and calibration is also available.


Hysteresis loop of Ni Standard Sample
Magnetization (emu)
H (Tesla)
Magnetic Moment at 1T = 3.49 emu
Temperature = 298 K
4
3
2
1
0
-1
-2
-3
-4
-1.0
-0.5
0.0
0.5
1.0


Hysteresis loop of Ni Standard Sample
Magnetization (emu)
4
3
2
1
0
-1
-2
-3
-4
-0.4
-0.2
0.0
0.2
0.4
H (Tesla)
Magnetic Moment at 1T = 3.49 emu
Temperature = 298 K