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# QUICK REFERENCES

GPD –  $\mu$ E1 AREA

Oxford Sorption Pumped  $^3\text{He}$  Cryostat type  
Heliox/Variox

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$\mu$ SR FACILITY 2017

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# 1 Oxford Sorption Pumped $^3\text{He}$ Cryostat type Heliox/Variox – QUICK REFERENCE

The main components of Heliox/Variox cryostat are: the sorption pump, the condenser, and the  $^3\text{He}$ -pot (See Fig. 1 for clarity). These three components are connected to form a closed  $^3\text{He}$ -system.  $^3\text{He}$ -pot is inclosed in a separate vacuum chamber which also contains the sample mounted on the pot. The sorption pump and the condenser are cooled through the  $^4\text{He}$  contact gas by the Variox variable temperature insert (VTI).

Low temperature mode (condensed  $^3\text{He}$ ) ( $0.27\text{ K} \rightarrow 1.6\text{ K}$ ):

1. **Precooling.** If the experiment requires measuring in the low temperature mode immediately after inserting the sample, the following procedure for pre-cooling should be followed. Otherwise, this step can be skipped.
  - Using DELTAT program, set low temperature (Modify devices→Heliox→Heliox setpoint = 3K and Variox setpoint = 1 K)
  - Set manual flow mode in the control window [jpsw412:8081/SC/heliox0j](#). Set the needle valve to a large value (80-99 %). The flow should be monitored and the value is expected to be 20-30 l/min. If the flow is smaller, it will take longer to cool down.
  - Using DELTAT program, Turn off the autoHeater (in Modify devices→Heliox).
  - It takes about 2-2.5 hours until the temperature of the "Variox-1.5 K-pot" is below 2 K (variable name "HELIOX He4 variox Loop 1"). Once this is reached, condensation can be started.

## 2. Condensing

- Make sure that the "Variox-1.5 K-pot" is below 2 K, the autoHeater is off and the flow control is set to automatic.
- Using DELTAT program, set the base temperature (Modify devices→Heliox→Heliox setpoint = 0.25K and Variox setpoint = 1 K)
- For condensation, 8-10 l/min flow is needed. In principle this is set automatically, but it should be checked.
- It takes about 30 mins to condense and cool down the system )

## 3. Temperatures can also be changed in the autorun sequence:

- A typical line in an Autorun sequence file should look like, e.g.

```
SET tempvariox 1 0.5 0 0.80 0.5 60  
wait 240
```

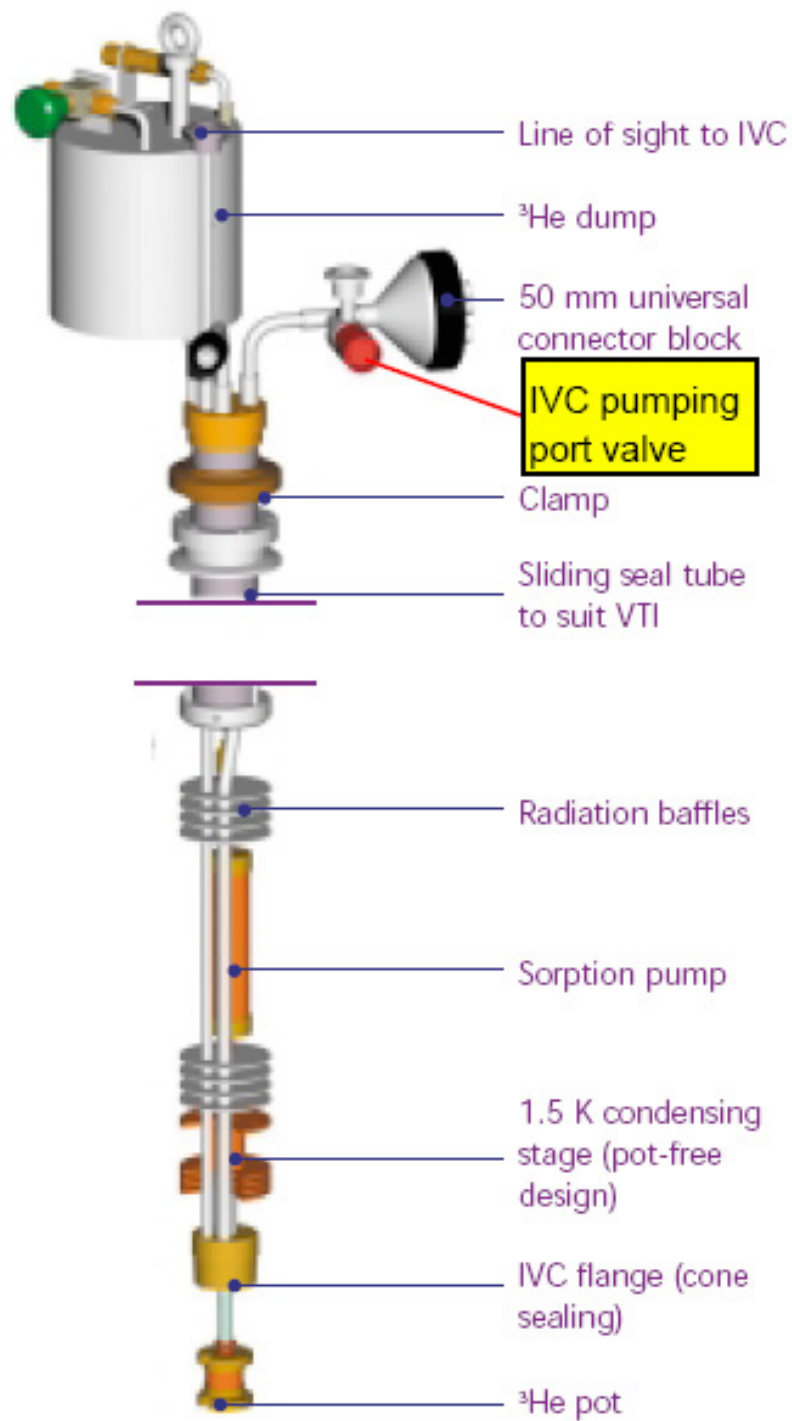
## START HISTOGRAM Back 7000000

First value in the sequence is Variox temperature and the second one is Heliox. A value well below the limit is set on Variox in low temperature mode. In the above sequence the Variox is first set to 1 K, after checking the temperature to be in a tolerance band of 0.5 - 1.5 K it is waiting for 0s and then the temperature of 0.8 K is set on the Heliox. After reaching a temperature of 0.3 -1.3 K it will wait for 60s before an additional waiting period of 240s is started. Finally, a run is started which will stop after 7000000 counts are accumulated in histogram 'Back'.

In principle, the flow is adjusted automatically. However, it can also be set manually by introducing additional parameters in a command in the sequence file (One can use the "show hints" button in the sequence editor for more information). For low temperature regime, the flow is typically around 3 - 4 l/min. The corresponding needle valve settings should be checked individually for each experiment. The linear correlation between the He flow values and the needle valve settings can be considered.

## High temperature regime above 1.6 K

1. For temperatures below 10 K keep the AutoHeater off.
2. Always perform the temperature scans from low temperature to high temperature, because cooling down is very slow.
3. If it is needed to go back to low temperature again, it is better to condense and go up in temperatures. The condensation can be stopped midway to save time.
4. Normally, the temperature is changed from the data acquisition program 'DELTAT' using the 'AUTORUN' as described above.



## 2 SAMPLE CHANGE

- **For safety reasons, a sample change should only be performed in the “High Temperature regime” with a sample temperature  $T > 100$  K. Close the needle valve on Variox and heat the Variox VTI to 100 K. When the Heliox sorb sensor (channel 1 on the Heliox ITC503) has reached 100 K follow the procedure below:**
- Pressurize slightly the Variox sample chamber with He-gas by connecting it to the He-gas pressure cylinder via the three-way valve on the Variox.
- Remove the insert by means of the little crane in the area (open the KF50 flange just above the rotation feedthrough) and mount the insert on the test rack in the area. Close the VTI by a blank flange as soon as possible. Please, avoid any mechanical damage when moving around with the insert or working on it and keep the insert always upright.
- Break the vacuum in the insert so that the sample stick warms up.
- Use a cylindrical a heat gun to warm up the insert.
- Additionally, a hand-held heat gun can be used to speed up the process.
- The IVC may be removed by means of the special tool used to break the cone seal. If the experiment took a short time, occasionally the seal can be broken by hand.
- **The protective equipment should be used when dealing with pressure cells. Before taking of the cone, make sure that all people in the area are protected..**
- Remove the sample (pressure cell).
- Remove the silicone paste used for the cone seal and clean both surfaces carefully. This should be done as soon as possible. Please, avoid scratching the surfaces of the cone seal.
- Mount the new sample (pressure cell).
- Check carefully whether there would be any thermal bridging due to dirt like residual sealing paste.
- Put some Silicon paste on the cone flange. Attach the brass IVC chamber to the cone and start pumping the IVC through the IVC pumping port by means of the turbo pump. The cone must be held by hand until the vacuum is high enough. This will take 0.3 - 1 h.
- Close the sample chamber in the insert, then switch off the pump and remove the pumping tube from the insert.

- Insert carefully the Heliox into the Variox by means of the little crane (Please, do not forget to pressurize the Variox chamber before putting the insert).
- Add exchange gas to the sample chamber using the bladder.

### 3 He-DEWAR CHANGE

Should the He-Dewar levelmeter read below  $\sim 15\%$  (in the case of a 100-liters dewar) the He-Dewar needs to be changed.

- Loosen the O-rings on the cryostat side.
- Take out the transfer line from the cryostat. Plug the cryostat entry.
- Remove completely the transfer line on the He-dewar side.  
Replace the plug of the He-dewar to prevent freezing of the He-dewar.
- **Carefully** warm up the transfer line using the available heat-gun.
- Be sure that the sintered end-part is free of any ice.
- Change the He-dewar.
- **Check that the He-recovery line is connected to the dewar and that the corresponding valves are open. Be sure that the recovery line is not bent and that the He-gas can flow freely.**
- Insert slowly the transfer line in the new He-dewar.
- Wait until the helium gas flow from the transfer line slows down - otherwise you will push out the liquid helium from the cryostat.
- Insert the transfer line into the cryostat and tighten the nut.
- Do not forget to tightly close the empty He-dewar and to connect it to the He-recovery line



## 4 SETTING THE EXPERIMENT MAGNETS

The main magnetic field of 0-0.66T can be powered by two different power supplies:

- the GOSSEN power supply, recommended for low fields, 0-250G.
- or the HELMHOLTZ beam line power supply for high fields, 0-6660G.

Only positive values in Gauss are allowed for both power supplies.

The respective power supplies can be set from DELTAT by

- SET Magnet HELM 150 30  
SET Magnet GOSSEN 150 30

Note:

- A 16-bit DAC is used to set the 6.6kG-Helmholtz power supply, i.e. the resolution is about 0.1G. An ADC with only 12-bit resolution (1.6G resolution) is used for read back. This causes a discrepancy between the values displayed in 'Demand' and 'Measured' in 'Deltat/Exp. Magnets/'). The value set by the power supply always corresponds to the 'Demand' value within the DAC-resolution of 0.1G. This value will also be stored in the header of the data file.
- Like all other magnet power supplies HELMH2 (650A/250V) is located on the second floor above the Studios but it is placed at the end of the front row above Studio E.
- Setting HELM to zero causes the Helmholtz power supply to switch off physically. You can also see the settings of the beam line power supply on the beam line PC in the windows for TCP/IP COMBI CONTROL or TCP/IP SETTINGS. Note: HELMH2 is the corresponding name in the device list on the beam line PC for the new GPD, HELMH was used for the old instrument.
- The GOSSEN can be switched off only manually. The GOSSEN power supply is located in the bottom part of rack No. 2.
- There is no auxiliary field available, i.e. the coils have to be rotated for calibration measurements when working in LF-mode.

## 5 More things to check

- Is the Helium gas cylinder full? It is used for auto-refilling and creating overpressure in the sample chamber. If not, replace it. Usually there is a spare full one right next to the current one.
- Is there enough LN2? If the reading shows less than 1/8, it is time to refill it. The refilling station is next to HAL instrument
- Is there enough liquid helium for the future? If not - order some. The order needs to be placed 3 days in advance.