Instructions for using the Helium closed cycle cryostat

A rapid transfer, top loading closed-cycle helium cryostat is available at the XAS beamline. The sample is maintained under Helium carrier gas for thermal conduction, which enables rapid sample change and quick re-equilibration at the desired cryogenic temperature. The entire cryostat’s position is motor controlled, such that multiple sample holder can be loaded.
The sample temperature can be automatically set and maintained in the range 20 – 300 K (accuracy estimated as 1 - 2 K) by supplying electrical heat to the heater installed on the sample mount. Both the sample and cold head have diode temperature sensors installed.

The system can be functionally divided into three separate flow systems: the sample space, the vacuum space, and the refrigerator connected to the compressor.

The sample space consists of a tube down the center of the cryostat body. The sample is inserted into this sample space. The space is filled with He during measurements. The sample space is not connected in any way to either the vacuum space or the He flow system. The exchange gas in the sample tube is an adjustable thermal link to the refrigerator. Higher pressure increases thermal coupling. As the temperature becomes lower, the pressure of gas in the sample tube will decrease. You can vary the helium exchange pressure from vacuum to atmosphere. At lower pressure you will have less cooling power.

The vacuum space surrounds the sample space and consists of the area between it and the outer shroud. Under normal operation the vacuum space is dynamically pumped to assure thermal insulation of the sample space.
COOLDOWN

This is normally done by the beamline scientists before the experiment begin.

1) Turn the 3 way valve anti-clockwise to pump on the sample space.

2) Pump on the vacuum insulating space. When this vacuum is about $10^{-5}$, turn on the compressor. (vacum will increase when it gets cold).

3) The cooldown process to base temperature (10 K) will take about 1 hour.
CHANGING THE SAMPLE

1) Turn the 3-way valve clockwise to flow in He gas (to assure that the sample zone is at atmospheric pressure before changing the sample)

2) Pre-cool the new sample stick in the small liquid nitrogen dewar.

3) Remove the KF clamp on the cryostat holding the instered sample holder (or blank).

4) Once fully cooled in #2 (ie the LN2 stops boiling) , quickly remove the sample rod, give it a thorough shake to remove any excess LN2 and immediately remove the existing sample stick and insert the new one. Reconnect the KF clamp. Care should be taken not to force in or out the sample sticks. Any sticking indicated freezing and the cryostat should be warmed up.

5) Using the 3 way valve do 3 separate pump purge cycles. Ie Pump the He out and flow He in againWait about 30 seconds each time.

6) Leave the 3 way valve in the middle ‘upright’ position.
HEATING IN XAS CRYOSTAT

When changing a sample or inserting a new sample the heater circuit in the Lakeshore 331 temperature controller will automatically turn off.

The controller is setup such that

CHANNEL A = Sample (usually minimum temp is ~ 20 K)
CHANNEL B = Cryostat Cold Head (typically 10 – 12 K)

All temperatures are in Kelvin (K).

If not using the heater function you do not need to worry. If you wish to use the heater function the following procedure must be followed:

1. Connect the sample stick sensor / heater cable.

Ie Connect the MIL connector to the terminals labeled “SEN/HTR” on the top of the sample stick
2. Turn off remote computer control in the Lakeshore.adl control screen on the endstation control computer.

   Press the “Pause” button

3. Put the Lakeshore 331 controller in “Local” mode

   On the Lakeshore 331 control panel press the “Remote / Local” button. The remote light will be unlit.

4. Turn on the heater circuit

   Press the “Heater Range” button once, until “Select for Loop 1” is displayed.

   Use the Up/Down arrows on the Lakeshore 331 to select “High”.

   Press “Enter”

5. Restart remote communication with the Lakeshore 331

   Ensure / set “SetPoint 1” on Lakeshore.adl screen to “10”
Press “Run” on the control section

Observe that the correct temperatures are reported for CH A (sample) and CH B (Cold Head).
TROUBLESHOOTING

Sample probe is stuck or ice is formed

If the sample probe will not come out easily when pulling it up, DO NOT USE FORCE. It most likely means that there is frozen N$_2$ and O$_2$ around the sample. This needs to be removed by heating the cryostat. Use the following procedure.

1) Turn off the compressor; do not turn off the pump for the insulation vacuum.

2) Put the sample in and connect the heater to the sample holder

3) Heat the sample until 300 K following the previous instructions

4) Wait until the cryostat cold head is at 120 K

5) Turn off heater

6) Disconnect heater plug

7) Remove carefully the sample holder and cover with the blank cap.

8) Check that ice is effectively removed

9) Pump out the He

10) Pump in the He

11) Turn on the compressor