

Instructions for using the sample cryostat at XAS

Contents:

- 1: Safety Precautions
- 2: Changing the sample
- 3: Normal operation conditions and Troubleshooting
- 4: Figures
- 5: Temperature sensor calibration data



1 Safety precautions

(a) Personal Safety

- When standing on the ladder, always stand on the top platform only, and always close the safety gate behind you to prevent falling off. Only ONE (1) person is allowed on the ladder at any time (load limit 130 kg).
- Whilst changing a sample, any person in the hutch or near the hutch entrance must wear safety glasses (risk of liquid air dropping off a cold sample rod).

(b) System Safety

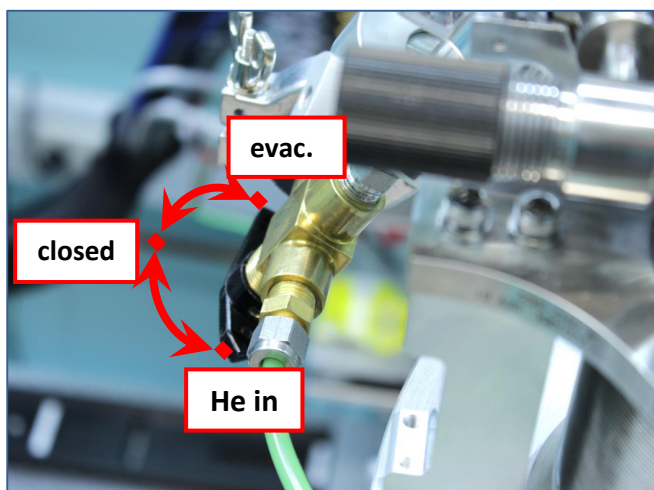
- **Sample rods are inserted into the sample space at room temperature** (the sample holder at the end of the sample rod may be cooled in liquid nitrogen; keep cooling periods as brief as possible). **Do not insert the sample rod if condensation or frost/ice is visible on the copper drum of the sample rod.**
- **Please inserted sample rods carefully into the system.**
- The sample space is purged at least three (3) times with He gas after sample loading. A safe purging pressure is pre-dialled via a wall-mounted pressure regulator. Do not change this pressure.

3 Changing the sample

Samples are mounted on one of the available sample rods using either PMMA or Al sample tails. **Avoid changing the sample tails by yourself; ask Beamline Staff instead.**

(a) Changing the sample rod

- 1) Before changing the sample rod, have the new one ready to go. Either “park” it in one of the holders available on the cross-bar near the top of the ladder, or ask a co-worker to hold it at the ready for you.
- 2) Turn the 3-way valve anti-clockwise (to the door) to flow in He gas (i.e., He flow ON).
- 3) Undo the KF clamp that holds the sample rod (or the blank) in place.
- 4) Carefully lift out the sample rod. **Take care not to bend the rod upon pulling it out.** The lower section is made of pure Cu and **bends and deforms easily.**
- 5) Leave He flow ON; replace the blind flange (without doing up the clamp). Hand the sample rod to your assistant or “park” it in the brackets. Take the new sample rod, remove the blank flange, and **carefully** introduce the rod into the cryostat. **You will experience some level of mechanical resistance when inserting a sample rod; this is normal. Avoid pushing the rod in via the black flange; use the top end of the rod instead to apply gentle downwards pressure.**
- 6) Fit the KF clamp and seal ring to secure the rod into place. Make sure the seal ring is in place properly when you do up the clamp.
- 7) Evacuate the sample space by turning the 3-way valve clockwise (away from the door). Monitor the pressure using the pressure gauges provided.
- 8) Purge the sample space three (3) times using these steps:
 - a) Quickly evacuate sample space to
 - $p < \sim 5$ mbar (1st purge)
 - $p < \sim 2$ mbar (2nd / 3rd purge).
 - b) Fill sample space with He (turn 3-way valve anti-clockwise to “He in” and wait for 5-10 seconds).
 - c) Repeat 3 times.
- 9) Finally, backfill the sample space with He, wait 5 seconds, and then leave the 3-way valve in the middle (“closed”) position.



3-way valve with three positions for user operation: (1) He in, (2) Evacuating (vacuum), (3) Closed.

It will take 15-20 minutes to cool the sample down to 5-10 K.

(b) Removing the sample rod without introducing a new one

If a new sample cannot be loaded immediately or if there is no sample to be loaded (e.g., beamtime finished), please:

- 1) Follow above steps (a).2-(a).4 to unload the sample rod.
- 2) Fit on the blind flange and do up the clamp.
- 3) Follow above step (a).8 to purge the system.
- 4) Turn the three-way valve to “evacuate” and leave it there.

3 Normal operation conditions and Troubleshooting

(a) Normal and abnormal conditions

Under normal operating conditions the cryostat will show certain behaviour and characteristics. Deviation from these characteristics may indicate problems; please inform Beamline Staff or, if after hours, the Operators on -4123 if you notice any abnormalities.

NORMAL conditions	Signs of ABNORMAL conditions
Some sliding resistance is felt when inserting and removing sample rod. Sample rod can be rotated against some small friction.	Sample rod feels stuck when removing or inserting. Sample rod does not rotate freely. May indicate ice formation and sample rod frozen stuck. Call for assistance.
Outside surface of the cryostat vessel is at room temperature.	Cold outside surface; condensation or frost; may indicate insulation vacuum problems. Call for assistance.
Temperature at cold head is ~5K or less.	Temperature does not drop much below 15K. Compressor cold head is very warm to the touch. May indicate insulation vacuum problems. Call for assistance.
Evacuating sample space to p<2-5 mbar takes ~30 seconds (depending on gas and temperature in sample space).	Sample space does not evacuate to 2-5 mbar. May indicate dirty KF sealing ring, missing sealing ring, or improperly aligned sealing ring. Check ring, clean, align. Try again. If problem persists, may indicate internal

	vacuum leak; call for assistance.
After purging and backfilling the sample space with He, the “Javac” vacuum gauge reads 1013mbar.	After purging and backfilling the sample space, pressure is <<1013mbar. May indicate internal vacuum leak. May also indicate that sample space was not backfilled with He; backfill now unless low He pressure is desired. If problem persists, call for support.
With cold-head at 4K, cool-down of a sample rod from room temperature takes about 15-20 minutes.	Cool-down times >>>30 minutes. May indicate vacuum leaks or drop in He pressure in pulse tubes (cooling performance). Call for assistance.

(b) Further troubleshooting

Problem: Sample is stuck (frozen in place)

Introducing moisture and/or air into the sample space can lead to forming ice from air, nitrogen, water vapour, oxygen, CO₂, etc.

If the sample is completely frozen into place, it may take up to 12-24h for the system to arrive back at room temperature and ice (especially from water) to be cleared away.

To **minimise the risk of freezing** / sample holders getting stuck:

- **Do not cool the sample rod in liquid nitrogen.** If necessary, the sample holder at the the end of the sample rod (and only the holder) may briefly be flash-frozen in liquid nitrogen. **If you notice any condensation or frost on the copper drum of the sample rod, do not use this rod until it is completely warm and dry.**
- Avoid keeping the sample space (top flange) open for long periods of time (>30 seconds).
- Check that the seal ring of the sample rod’s flange is clean, not damaged, and centred in place. Securely fasten the sample rod’s flange before evacuating the sample space.
- Carefully seal liquids in sample holders.

If ice has formed: Call for assistance.

4 Figures

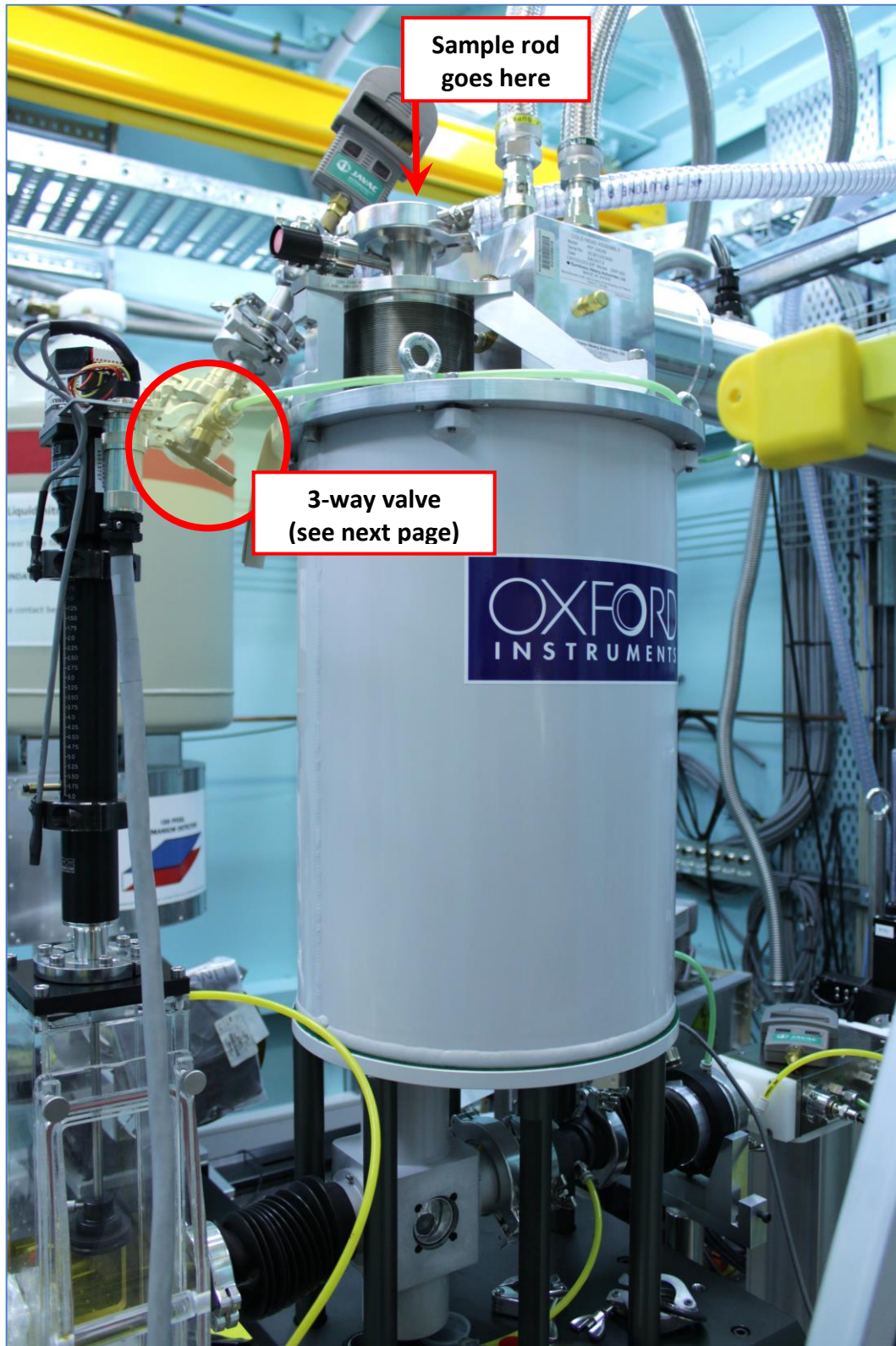


Figure 1: Cryostat overview. Sample rod position and 3-way valve are highlighted.

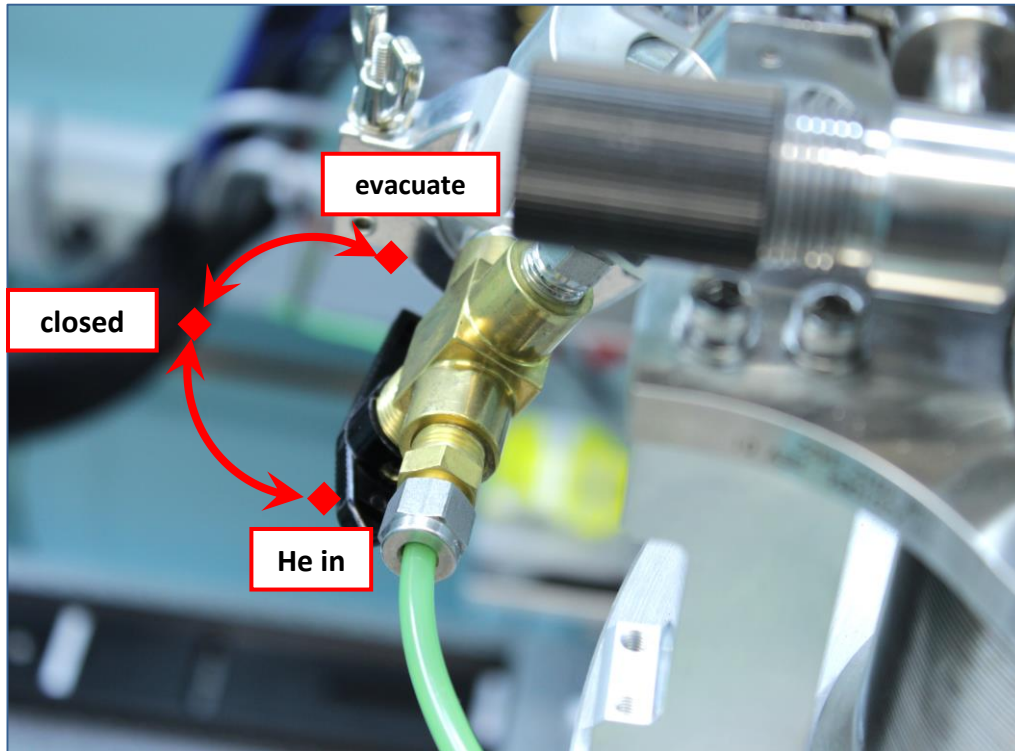


Figure 2: 3-way valve with three positions for user operation:
(1) He in, (2) Evacuating (vacuum), (3) Closed

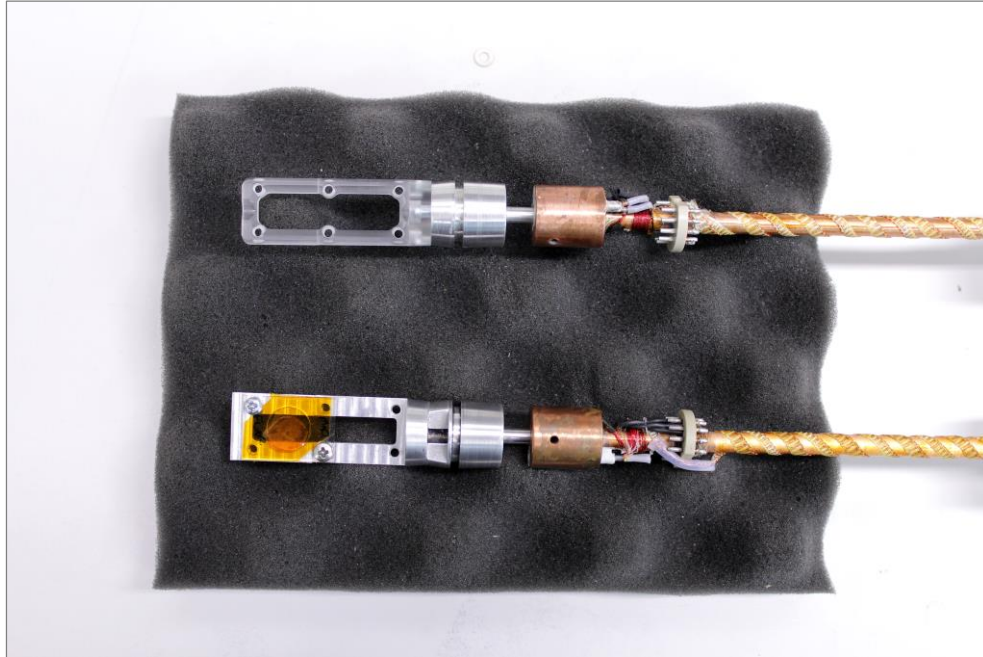
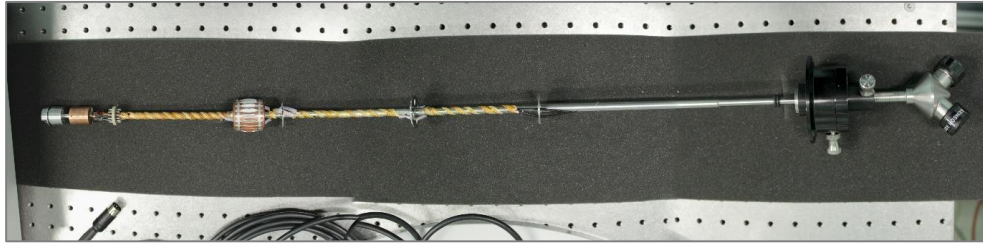


Figure 3: Sample rod (top) and sample mounts (bottom).

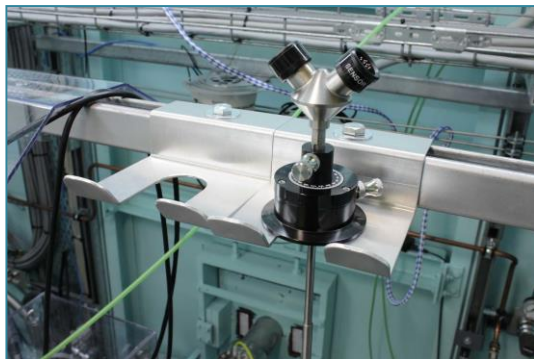
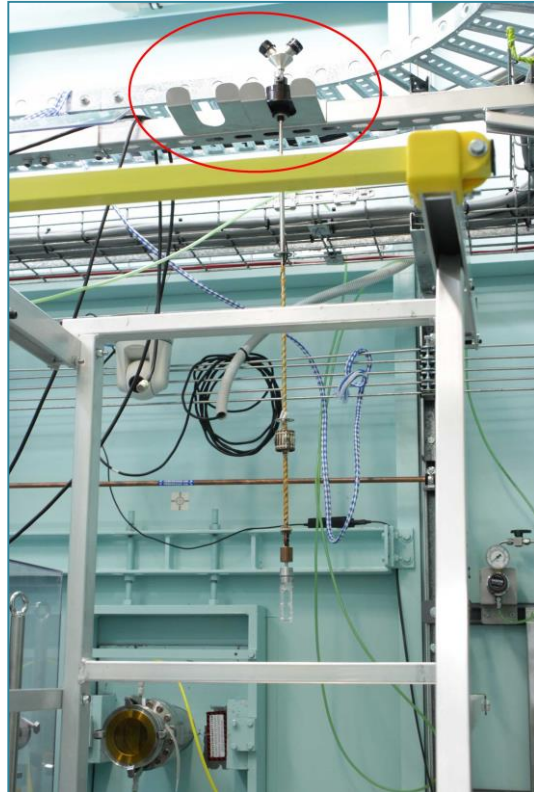


Figure 4: Clamp/park for sample rod; for use during loading a sample rod into the cryostat.

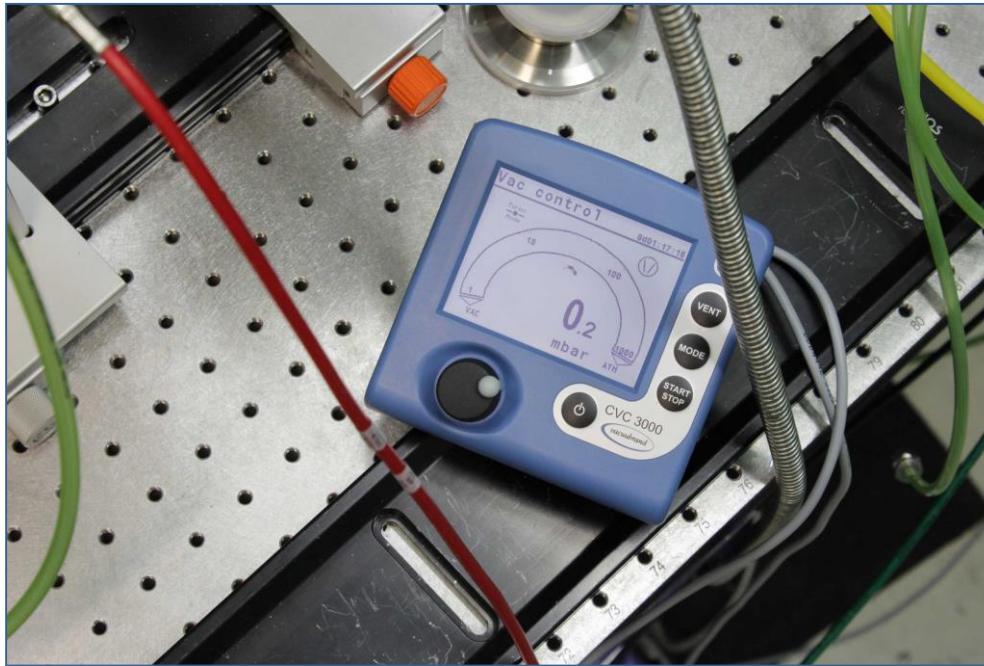


Figure 5: Pressure gauge for monitoring vacuum during evacuating the sample space. A pressure <0.5 mbar is an acceptable target during purging.

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Following sections are under development

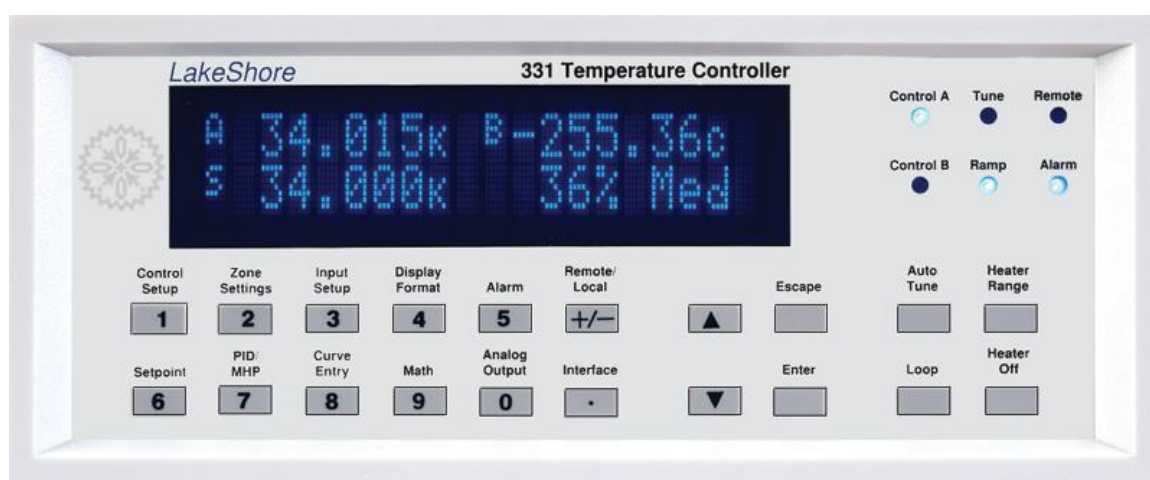
5 Whole system cool-down (Staff only)

Refer to the system and compressor operating instructions provided with the whole system. Cool-down procedures and requirements (insulation vacuum, cooling water flow, etc) are described there in detail. Here only a basic guide for system cool-down:

- 1) Turn the 3 way valve away from the door to pump on the sample space.
- 2) Pump on the vacuum insulating space using the turbo pump. When this vacuum is about 10^{-6} mbar, turn on the compressor (vacuum will increase when it gets cold).
- 3) Once $T_{\text{cold-head}} < 5$ K, close insulation vacuum valve at top of cryostat (next to cold head).
- 4) The cooldown process to base temperature (4 K) will take about 3.5 h. It may take 30 minutes until cooling starts to accelerate.

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).

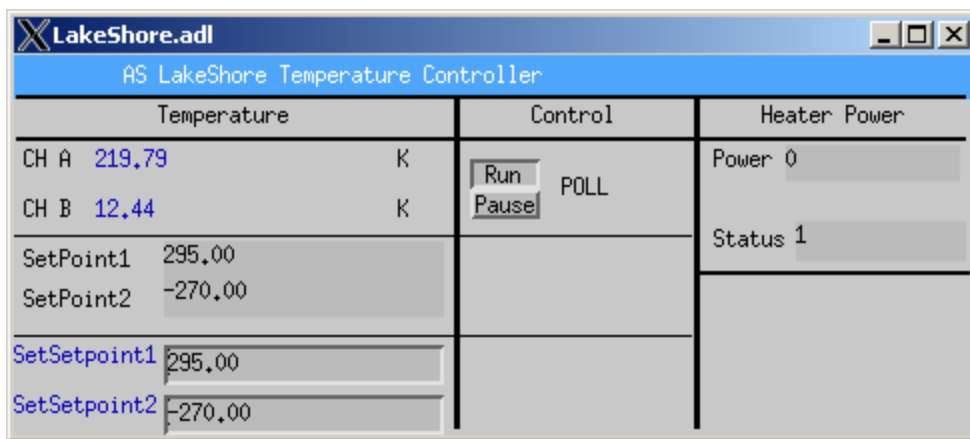
If not using the heater function you do not need to worry. If you wish to use the heater function the following procedure must be followed:

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..

Ie Press the “Pause” button



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

Ie. 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”

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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).