

Equipment

- list of valves: V1-V12 on the gas handling panel, V13-V16 on the cryostat, V17&V18 at the still pump, V19-V23 at the vacuum pump, V24 at the pot pump, V25&V26 at the He gas cylinder; direct outlet valve and overpressure outlet valve on the He dewar ("handle" valves); black main valve and two small T-valves on the heat exchange gas (He-4) dispenser
- vacuum gauges:
 - analog gauges on the gas handling panel: mixture tank 1 (not in use), mixture tank 2, still pump back, condensing line
 - digital gauges: vacuum pump (sensor 1 of the left unit), still line (sensor 1 of the right unit), pot line (sensor 2 of the right unit)
- pumps:
 - scroll pump (Agilent?) and turbomolecular pump (Agilent Turbo-V 81-M) for the vacuum pumping system, rotary oil pump(?) (Alcatel?) for the 1 K pot, multi-stage roots pump (Alcatel?) for the still, i.e. circulation
- sensors and heaters:
 - for AVS-48: 1=pot sensor, 2=still heater, 3=mixing chamber heater, 4=mixing chamber sensor
 - for AVS-47: 0=pot sensor, 1=still heater, 2=mixing chamber heater, 3=mixing chamber sensor
 - $R_{\text{pot}}=0.6\text{...}21\text{ kohm}$ (4.2 K \rightarrow 1.4 K), $R_{\text{stillht}}\sim 552\text{ ohm}$ (@ $\sim 200\text{ mK}$), $R_{\text{mxcht}}\sim 414\text{ ohm}$, $R_{\text{mxc}}=1\text{...}6\text{ kohm}$ (300 K \rightarrow 50 mK)
- flow meter: Bronkhorst EL-FLOW F-111C-HBD-33-V, operating voltage $\sim 15\text{ V}$, output voltage proportional to flow rate ($\sim 37\text{ }\mu\text{mol/s/V}$ according to an old calibration)

Basic operation

Preparations

- close vacuum can and attach μ -metal shield
 - Insert sample holder and remove shorting headers. Close vacuum can with In seal and attach μ -metal shield (leave pot siphon outside). Press pot siphon firmly against the shield and wrap it with tape so that it doesn't get stuck when lowering the cryostat to the dewar.
- pump the vacuum can
 - Initially, all valves should be closed (and all pumps running). Connect the heat exchange gas dispenser to the vacuum can and open the black main valve. Connect the vacuum pumping line to the dispenser. Open V21, V20 & V14 and pump to $<1\text{ mbar}$; open V23 and start the turbo. Close V21, open V22 and pump to $<10^{-3}\text{ mbar}$. Close V14, the black valve on the heat exchange gas dispenser, V20 & V22 and switch off the turbo. Close V23 after the turbo has stopped.
- flush and pressurize the pot
 - Close the direct outlet valve on the He dewar (the one that goes to the recovery system), move the plastic hose from the direct outlet to the

overpressure outlet (the one behind the 3-50 psi relief valve) and make sure that the overpressure valve is open. Connect the He gas cylinder to the He dewar's direct outlet (plastic hose), to the vacuum pump (steel tube), and to the pot line (long steel tube). Open V21, V20, V25 & V13 to pump the lines and the pot to <1 mbar. Close V20 and open the direct outlet valve on the He dewar to flush the lines and the pot with He. Close the He dewar valve and open V20 to pump again to <1 mbar. Repeat flushing and pumping a few times. Close V20, open the He dewar valve and V26 to pressurize the He gas cylinder. Check from the He dewar's pressure gauge that there is some overpressure (preferably ~0.3 bar).

Cooling down to 4 K

- precool with LN2
 - Fill a tall LN2 dewar halfway and another LN2 dewar full. Lower the cryostat to the tall dewar and fill it up from the other one. Cover the top with bubble wrap to prevent icing. Open V14 and add 5-10 pulses of He-4 to the vacuum can by opening and closing the small T-valves successively (both valves must never be open at the same time). Connect the resistance bridge and monitor mixing chamber temperature (channel 4 in AVS-48, channel 3 in AVS-47). Let the cryostat precool for 1-3 hours (fill up the LN2 dewar every now and then). You don't have to wait for the cryostat to cool to 77 K, as that could take a long time; 100 K is definitely enough.
- transfer from LN2 to the He dewar
 - Close V25 and the He dewar's direct outlet valve. Move the recovery line hose from the overpressure outlet to the direct outlet, reopen the valve and let the pressure inside the dewar decrease to ambient level (the recovery hose will partially freeze so be careful not to bend it and break it). Set resistance bridge to zero mode (command 'picobridge_zero' for AVS-48) and disconnect the sensor cable. Open the He dewar's top clamps and remove the lid. Lift the cryostat from the LN2 dewar and lower it carefully to the He dewar (wear thick gloves and guide the cryostat all the way down so that the radiation shield flanges do not get stuck at the top of the dewar). Close pot valve V13 after the pot syphon has reached the surface of liquid He. When the cryostat is all the way down, tighten the dewar's top clamps, reconnect the resistance bridge cable and monitor temperature down to 4 K (~1557 ohm). Pour leftover LN2 to the trap.
- connect and evacuate tubes
 - Connect pot, still, condensing and vacuum pumping lines to the cryostat. Open V24 to pump the pot line. Open V19-V21, V1-V4 & V17 to pump everything else. When the still pressure drops below 1 mbar, open V23, start the turbo, close V21 and open V22. (fixme: sometimes it might also be a good idea to open condensing and still valves V15 & V16 to pump leaked air from inside the cryostat)
- pump out heat exchange gas when temperature reaches 4 K
 - Open V14 and the black main valve on the heat exchange gas dispenser. Pump for a few hours (or overnight). Close V14, the black valve on the heat

exchange gas dispenser, V17, V1-V4, V19, V20 & V22. Switch off turbo and close V23 after the turbo has stopped. All valves should now be closed, except for the pot pump valve V24.

Cooling down to base temperature

- cool the 1 K pot
 - Check that all valves are closed, except the pot pump valve V24. Open pot valve V13 and monitor the pot pressure, which should eventually stabilize to ~ 1 mbar. If it settles to a distinctly smaller value, there may be a blockage in the pot syphon.
- fill the LN2 trap (and refill once a week)
- condense the mixture down the still line
 - Open V9 & V9B to precool mixture behind the still pump in the LN2 trap. Open V6, V2-3, V17 & V16 to condense the mixture behind the still pump. The pot pressure should first increase, then decrease and finally return to ~ 1 mbar, and the mixing chamber temperature should eventually decrease to the pot temperature of ~ 1.5 K. Close V17 and open V12, V8 & V11 to let the mixture from the tank to precool in the trap. Reopen V17 just slightly until you see an increase in pot pressure (the still gauge shows over range, "or", because He pressure is >6 mbar). Temperature should jump up and then start to decrease towards the pot temperature. Increase still pressure by slowly opening V17 until completely open. Wait $\sim 1-2$ h until all the mixture has condensed (pressure <0.05 bar on the analog gauges and mixing chamber temperature stable at ~ 1.5 K). Condensing is faster if the pot was cooled for a longer time.
- close the tank and start circulation
 - Prevent mixture from flowing back into the tank by closing V8. Leave V11 & V12 open so that if there's a failure and the circulation pressure increases to >1 bar, the mixture can flow through the overpressure diaphragm valve (parallel to V8) to the tank. Close V2, V3, V16 & V17 and open V4, V15 & V18. The only closed valve in the circulation path is now the still valve V16. Open V16 just slightly, until there's an increase in still pressure on the digital gauge. Adjust V16 continuously so that the still pressure stays below ~ 10 mbar and the still pump back pressure below ~ 0.8 bar (analog gauge). Eventually when the pressures decrease, leave V16 open. Mixing chamber temperature should decrease to <300 mK (~ 2587 ohm) in ~ 15 min with pressures eventually reaching $\sim 6 \cdot 10^{-2}$ mbar in the still and ~ 0.04 bar in the gas panel (flow meter ~ 0.2 V).
- switch on still heater to ~ 1.7 V or ~ 3.0 mA (optimal flow rate $\sim 1.6-2.0$ V)
 - Connect the still heater cable to the battery box or a function generator. If you use a function generator, you need to measure the actual voltage because the internal 50 ohm resistance of the generator will decrease the output voltage from the nominal value. If you use one of the old Agilents, model 33120A, connect the BNC shield to ground with a separate cable to reduce noise drastically. Flick the still switch on the heater breakout box (next to the resistance bridge preamplifier box) from "measure" to "heat".

Set voltage/current to a suitable level and monitor flow rate and temperature. A base temperature <50 mK (>5 kohm) should be achievable.

Warming up

- stop cooling the pot and pump mixture to the tank
 - Close pot valves V13 & V24. Open V8 and close V9 & V9B to pump mixture from still to the tank. Open V17, V2 & V3 to also pump from the condensing side.
- speed up pumping by switching both heaters on
 - Connect mxc and still heaters in parallel to an adjustable voltage source (e.g. a function generator) and make sure that both switches in the heater breakout box are set to "heat". Apply ~ 2 V to the heaters and monitor still pressure on the digital gauge. Adjust heater voltage to keep still pressure below ~ 1 mbar. As the pressure in the tank increases, you can increase heater voltage up to ~ 4 V as long as the still pressure stays below ~ 1 mbar. Monitor mxc temperature and tank pressure to see when pumping has finished ($T > 4$ K and tank pressure ~ 0.5 bar)
- switch off heaters and close all valves when the tank pressure is stable at ~ 0.5 bar
 - When temperature reaches ~ 4 K, continue pumping for ~ 30 min more until the still and tank pressures stabilize to $\sim 10^{-1}$ mbar and ~ 0.5 bar. Switch off heaters. Close V15 & V16, then V6 & V2-4, then V17 & V18, and finally V8 & V11-12. All valves should now be closed.
- connect an overpressure valve to the pot and lift up the cryostat
 - Disconnect all pumping lines. Set resistance bridge to zero mode (command 'picobridge_zero' for AVS-48) and disconnect all cables. Connect an overpressure valve (~ 0.3 bar) to the pot flange on the cryostat and open the pot valve V13. Open the He dewar's top clamps and slowly lift up the cryostat while making sure that the radiation shield flanges don't get stuck. Lower the cryostat on its stand and cover the vacuum can with bubble wrap to prevent icing. Close the dewar's lid. Reconnect resistance bridge sensor cable and restart monitoring.

Moving the cryostat between dewars

- follow the instructions above in section "Warming up" up to the point of lifting the cryostat from the dewar
 - Make sure that there's an overpressure valve (~ 0.3 bar) connected to the pot flange on the cryostat and that the pot valve V13 is open.
- move the cryostat from one dewar to the other
 - Have the full dewar ready for transfer by opening the top clamps and bringing it next to the empty dewar, with both dewars connected to the He recovery system. Slowly lift up the cryostat from the empty dewar. When it's fully above the dewar's neck, pause for a moment to let the liquid He drip out from the μ -metal shield. Move the lid from the full dewar to the empty dewar and position the full dewar directly under the cryostat. Carefully lower the cryostat into the full dewar, making sure the radiation shield flanges do

not get stuck at the top of the dewar. Close pot valve V13 after the pot syphon has reached the surface of liquid He. When the cryostat is all the way down, tighten the top clamps on both dewars.

- connect and evacuate tubes, as instructed in section "Cooling down to 4 K"

Advanced operation

- cooling down to 50 mK without still heater
 - Add a needle valve to the pot pumping line (condensing can't be done by pumping through the needle valve because the pot will remain too warm to get all the mixture out of the tank). Adjust so that flow is ~ 1.5 V at base temperature.
 - using both the needle valve and the still heater in an optimal configuration will enable cooling down to ~ 35 mK
- controlling temperature between 50 mK and 1.4 K (circulation and both heaters)
 - slightly increased flow (2.0-2.5 V) can speed up cooling down to low temperatures
- controlling temperature between 300 mK and 1 K (circulation and mxc heater)
 - slow to cool down
- controlling temperature between 1.4 K and 4.2 K (pot-only cooling and mxc heater)
 - using just the mixture behind the still pump would be enough to cool down from 4 K to ~ 1.4 K
 - getting a stable temperature control requires using more of the mixture (in practice all of it, i.e. tank open all the time)
 - stable operation above 3 K requires increasing the pot temperature by adding a needle valve to the pot pumping line

Possible problems

- 1 K pot partially blocked
- running out of He in the dewar
- gas in the vacuum can (In seal leaking)