Quantum Design PPMS DynaCool: Electrical Transport Option (ETO)

(sort of) Quick Start Guide

- Start your session in iLab: this powers up the monitor on the PPMS and connects it to internet (so it shows up on Teamviewer).
- In the PPMS log sheet binder, start a new sheet with your info.
- The PPMS computer lock screen password is written in beginning of the MSDS binder in the lab, • if you forget it.
- PPMS should be at 300 K, zero field, and purged chamber. If it is NOT, please tell Neil and note this in your log sheet.
- Make all cable connections:
 - if you are using the puck, plug in ETO grey Lemo cable into the PPMS port.
 - If you are using Rotator or MFP probes, first plug the Rotator/MFP Experiment cable (it's a box with a cable coming out of it) into the PPMS, and the ETO grey Lemo cable into this box (more info in ETO manual).
 - You should hear a click when ETO cable engages in the PPMS or the rotator/MFP box, and it will lock in place. DO NOT FORCE ANYTHING, adjust slightly and retry if it doesn't engage.
 - IMPORTANT: THE ONLY CABLES YOU WILL PLUG/UNPLUG WILL BE TO THE GREY LEMO PORT OR THE BNC CABLES TO THE PICOSCOPE. There is another cable running power and digital logic that will always be connected, do not mess with this. It has a big yellow sticker on it saying this also.
- Quit and restart the MultiVu application (red "D" icon in the launcher tray): this helps to clear error states for ETO that tend to slow things down.
- Activate Electrical Transport under *Utilities > Activate Option* in MultiVu.
- Getting your new puck ready for the chamber: •
 - Put on gloves when handling pucks.
 - To ensure your electrical connections to the sample are good, plug the puck into the P150 Sample Wiring Test Station (pic on right) and test connections using a multimeter.
- - If the sample cannot handle a multimeter's voltage drop or currents, or if you want more detailed diagnostics on the sample now, you can take the P150 box to the PPMS and plug the ETO grey Lemo cable into the side of it and use the ETO software to test the sample before you put it in the chamber of PPMS.
 - Make sure no wires are going over the edge of the puck where the extraction tool grabs the puck, and that any debris has been removed from puck.
 - Remove from the P150 and make sure bottom and edges of puck are clean. 0
- Under *Instrument > Chamber...* or by double-clicking chamber area at bottom of screen, press the Vent/Seal button and wait for chamber to get above 760 torr.

- Release the KF clamp at the top of the chamber, remove the baffle set, and put the baffle set in one of the clear vertical storage tubes on the side of the PPMS.
- Ensure there is no sample currently in the chamber: step up and look into chamber. If you can't tell whether the chamber is empty, get the extraction tool and try to remove a puck.
- Using extraction tool to <u>remove</u> a sample in the chamber:
 - Put on gloves in preparation for handling the puck.
 - The storage tool is a sensitive instrument, please do not bump or drop it, especially the thin walled stainless steel tube at the bottom, as this can break it.
 - Remove the extraction tool carefully from the storage tube, open the lever at the top (lever in vertical position), and lower the tool down into the chamber until it comes to the bottom. Rotate it a bit to make sure it's at the bottom.
 - Close the lever (horizontal), and if there's a puck in the chamber you will feel resistance when you try to pull up.
 - Pull out the extraction tool from the chamber, which will be carrying the puck, and DO NOT MESS WITH THE LEVER at top of extraction tool in this step. It's critical that we fully remove the puck and not drop it.
 - Once extraction tool is out of chamber, secure it with your other hand and take to the lab bench where you will cup that hand under the bottom end of the tool while you release the lever (put it in vertical position), which releases the puck.
 - Put the extraction tool back in the storage tube to protect it.
- Go back to PPMS, look at the bottom of the baffle set to make sure that the contact baffle (see diagram at right, and Chap. 2 of <u>TTO user's</u> <u>manual</u>) is NOT present, since it has charcoal which will cryopump He gas when T<15 K. Then put the baffle set back into chamber, close the KF plastic clamp, and press *Purge/Seal* in chamber commands dialog. This ensures that the chamber stays free of air and moisture.



- Open chamber as before: press Vent/Seal, remove baffle set and put it in the storage tube.
- Using extraction tool to insert your sample into the chamber:
 - Wear gloves when handling pucks.
 - With one hand, hold top of extraction tool and open the lever (vertical position).
 - With other hand, cup the bottom of extraction tool and hold puck between thumb and first finger.
 - Insert puck into extraction tool and gently rotate it to ensure it is seated evenly at the stop.
 - Close the lever at top of tool and see that the puck is held firmly.
 - Note the position of the key on the puck which will face to the front of the chamber when fully inserted.
 - Take the tool to the PPMS chamber, insert fully into the chamber, and rotate around until you feel the key slip into the notch. Once you feel this, push down firmly (a few kg of force) another ~5mm.
 - Release the lever (vertical position) at top of the extraction tool and you should be able to lift it without resistance from the engaged puck.
 - If you feel the puck is still engaged, close the lever again, remove tool/puck from the chamber, and repeat the process of inserting the puck.

- If it still is not working, then remove tool/puck, put in baffles, purge/seal, and call Neil or Mike.
- Put baffle set back in chamber, close the KF clamp, and press *Purge/Seal* in chamber commands.
- In ETO software, click *Datafile* tab and press *change* to set up a new measurement data file for ETO.
- Click on *Setup* tab in ETO Console and click *Sample Properties* if you wish to have your sample information (dimensions, composition, mounting method, etc.) recorded in the header of your measurement data (.DAT) file. Note that this is only for your information since sample geometry is not used in calculating any reported quantities: ETO reports voltages, currents and resulting resistances (in ohms) instead of resistivity (in ohm-cm).
- It is HIGHLY recommended to check your sample with ETO before launching any measurement sequence.
- <u>Resistance</u>: click on *Measurement* tab, select *Resistance*, and click *Launch Measurement*.
- At the bottom left of the Resistance measurement dialog, click "Save Results to file" if you want these test measurements written to your current .DAT file (I recommend this!).
- There are four regimes of sample resistance to consider initially:
 - $\mathbf{R} = \mathbf{1} \ \mathbf{m} \mathbf{\Omega} \ \mathbf{to} \ \mathbf{100} \ \mathbf{k} \mathbf{\Omega}$: "low resistance" requires higher frequency for best amplifier performance, enough current to get us out of nV noise of amplifier
 - Amplitude = .001 mA (larger if R even lower, but max. R will decrease)
 - Freq = 18 21 Hz
 - Wiring/Range = 4-wire, Autorange
 - Averaging = 1 sec., Number of Measurements = 5
 - **R** = 40 k Ω to 3 M Ω : "high resistance" requires lower frequency to avoid RC attenuation
 - Amplitude = .001 mA
 - Freq = 0.4 0.5 Hz
 - Wiring/Range = 4-wire, Autorange
 - Averaging = 2-3 sec., Number of Measurements = 5
 - o (these 2-wire measurements that follow are more rare)
 - **R = 1 MΩ to 300 MΩ :** "huge resistance" employs the special 2-wire mode (see sample mounting section) with voltage drive instead of current drive, low frequency
 - Amplitude = 0.3 V (this is 2-wire mode now, with voltage drive)
 - Freq = 0.4 0.5 Hz
 - Wiring/Range = **2-wire**, Autorange
 - Averaging = 2-3 sec., Number of Measurements = 5
 - R = 30 MΩ to 5 GΩ : "colossal resistance" employs the special 2-wire mode (see sample mounting section) with voltage drive instead of current drive, low frequency
 - Amplitude = 10 V (this is 2-wire mode now, with voltage drive)
 - Freq = 0.4 0.5 Hz
 - Wiring/Range = **2-wire**, Autorange
 - Averaging = 2-3 sec., Number of Measurements = 5
 - You should see some resistance and phase angle reported in this dialog, or you can see data in your data file under *Datafile > View*.
 - If no data or it looks bad (large phase angle, very noisy...), try repeating measurement.

- If still issues, use the Grey Lemo Breakout Box (see section in sample mounting training) to test out sample connections with the multimeter while sample is inside chamber.
- If still no data, then deactivate ETO (you can keep your sample in and all cables connected), close MultiVu, and reopen MultiVu and reactivate ETO, then retry.
- Inspecting the quality of the data:
 - Phase angle should always be small <2 degrees since we are trying to measure resistance (not reactance) here. If it is larger, it can be from sample resistance being too large for the selected regime (see 4 regimes above). Another reason for large phase is non-ohmic contacts which can be checked by making an I-V measurement (see ETO manual for I-V guidance). If the I-V is not linear then usually barriers (Schottky) at the sample-contact interface are to blame and more careful sample contact prep is needed.
 - 2nd and 3rd harmonic: these are a measure of non-ohmic response as well, and are a way of knowing from a resistance measurement that you will not be getting a linear I-V curve. They are stated in dB (decibels) which is logarithm (base 10) scale and the value is "nth harmonic"=20*log₁₀[V(n*f)/V(f)] where n=2 or 3 in our case, *f* is the ETO measurement frequency, and V(n*f) and V(f) are the lock-in voltage magnitude at the nth harmonic and the fundamental frequency, respectively. Things to look for:
 - in cases where the signal is well above the 1-10 nV noise floor, you should see 2nd and 3rd harmonics in the -60 to -80 dB range, i.e., they are 1000x to 10,000x smaller than the fundamental.
 - If 2nd harmonic rises above -40 dB, it indicates a rectifying contact: more current passes in one direction. A diode is the textbook example of this; Schottky barrier at contacts to a sample can bring this about.
 - If 3rd harmonic rises above -40 dB, it indicates nonlinearity in I-V which is symmetric around V=0; one example is "clipping" where the readback signal looks like a square wave instead of a sine wave. The other case is where there are barriers in both directions. Make sure autoranging is working (try on a higher range manually), improve contacts to sample.
- Using PicoScope USB oscilloscope on the system: this can be a great visualization of the signal waveform from ETO. See Neil for guidance on using it.
- There are example sequences that use the above resistance ranges in: C:\QdDynacool\Sequence\Example sequences to get you started\ETO
- For I-V and dV/dI measurements or more guidance, consult the ETO User Manual
- Mental checklist ANY time you step away from the machine:
 - Is the cap on the chamber?
 - Is the chamber purged AND the pressure <10 torr (MPMS) or <50 torr (PPMS)?
 - \circ Is the system going to be left at 300 K and zero field at conclusion of my run?
 - Is MultiVu software running?
 - Are all tools put away and the benchtop clean for the next user?