Setting up Electrical Transport Measurements in PPMS

These instructions apply to anyone who wants to measure electrical properties in the DynaCool, whether using the Quantum Design ETO or external electronics.

There are four aspects to be considered here:

- 1) Choose the sample stage: puck, rotator, or "MFP" (multi-function probe)
- 2) Choose the measurement electronics
- 3) Mount sample and Test contacts
- 4) Set up measurement electronics

1) Choose the sample stage

This table should guide users to the best configuration for their measurements:

| name / illustration | # of | Sample | ESD/ | Notes |
|---------------------------|-----------------|--------------------------|---------|---|
| | contacts | Rotation | gating | |
| PUCK | 12 | No | No/Yes | Sample < 10x10 mm Most sensitive (~ 1 nV) Uses PPMS wiring B 1 sample |
| ROTATOR | 8 | Automated | No/Yes | Sample < 9x9 mm Uses PPMS wiring Can also rotate B in sample plane |
| Multifunction Probe (MFP) | 16 (+1 Coax) | Manually at the bench | Yes/Yes | Sample < 5x5 mm Uses MFP wiring User supplies own 16-pin carrier Coax up to 200 V |

Notes about the table notes:

- ESD "yes" means that ESD-sensitive samples can be protected during insertion into PPMS
- PPMS wiring: 6 twisted pairs of low resistance Cu wires (<1 ohm each), see "<u>PPMS sample</u> chamber properties" on DynaCool Wiki for more details and important limitations
- Rotation can take B field from perpendicular to in-plane (default), or rotate the field in the plane of the sample ("shark fin"); sampe mounts are supplied by Spin Lab
- MFP wiring: 8 twisted pairs of Constantan wires, 90 ohms each, see section on MFP at end of this document
- The 16-pin DIP carriers are CSB01655: <u>https://www.spectrum-semi.com/products/sidebraze</u>

2) Choose the measurement electronics

possibilities include:

- 1) Electrical Transport Option (ETO) in DynaCool sidecar
 - a. ETO module is a lock-in based resistance measurement covering micro-ohm to 5 gigaohms using sine wave excitation (current when R<10 mega-ohm, voltage source above that) and an exquisite ~1 nV/rt-Hz amplifier at low frequency (0.4 – 200 Hz); I-V and dV/dI measurements also possible
- 2) Resistivity Option in DynaCool sidecar
 - a. uses the BRT module to make 10 Hz commutated (+/-) DC resistance measurements using a current source (10nA-5mA) and voltmeter (~10 nV/rt-Hz)
- 3) LakeShore FastHall M91 on the rolling rack by DynaCool
 - a. commutated DC resistance with capability to fully permute all the source and measure contacts for van der Pauw and "FastHall" permuted Hall effect; it does all contact checks (2-probe I-V verifying linearity) and can calculate materials properties of mobility and carrier density from measurements
- 4) Various source/meters on the rack
 - a. Typical setup is the Kiethley 6221 DC/AC source and a SR830 lock-in but many SMUs are available, see table on BNC Wiki page for Electrical and Magnetic properties

3) Mounting samples, testing contacts

- *Mounting samples to the puck*: configurations shown below.
- Testing contacts on the benchtop:
 - put puck into test box and use Fluke meter at banana plugs or ETO/Resistivity using grey Lemo to test box.
 - Ensure sample is not shorted to metal parts of puck
 - If you need to patch a contact, Ag paint may be used.
- Designing a measurement sequence for your sample
 - See example sequence folder
 C:\QdDynaCool\Sequence\Example sequences to get you started\ETO
 - We show example R(T) scans for four regimes
 - 4-wire low resistance : $1 \text{ m}\Omega$ to $100 \text{ k}\Omega$
 - 4-wire high resistance: 40 kΩ to 3 MΩ
 - 2-wire low resistance: 1 MΩ to 300 MΩ
 - 2-wire high resistance: 30 MΩ to 5 GΩ
 - Resistivity option sequences are much simpler but limited in what you can do

Examples of sample mounting on the puck

More info in seminar here.

Rule 1: electrically insulate your sample from the metal of the puck! If it is a film on insulating substrate, you're fine. Otherwise use Kapton tape or cigarette paper/grease.

| | Option | | |
|--------|--------|-------------|--|
| socket | ETO | Resistivity | |
| 3 | 1 I+ | 1 I+ | |
| 4 | 1 I- | 1 I- | |
| 5 | 1 V+ | 1 V+ | |
| 6 | 1 V- | 1 V- | |
| 7 | 2 I+ | 2 I+ | |
| 8 | 2 - | 2 I- | |
| 9 | 2 V+ | 2 V+ | |
| 10 | 2 V- | 2 V- | |
| 11 | | 3 I+ | |
| 12 | | 3 I- | |
| 13 | | 3 V+ | |
| 14 | | 3 V- | |



(LEFT) wiring diagram relating the sockets on the puck to the functions in the measurement electronics. Note that pins 3-4, 5-6, etc. are paired. (RIGHT) Puck for ETO and Resistivity measurements. Sample, shown in blue, is mounted for typical 4-wire measurements on Channel 1; wires in red.



Van der Pauw

More examples of sample mounting



(To learn about this technique to determine the intrinsic resistivity and mobility from uniformly thick samples, read his paper posted on BNC Wiki.)

Samples are rectangular (shown in blue on RIGHT figure) with square shape being ideal, leads are attached to the corners at small contacts, and you will use the grey Lemo breakout box (see next section of this document) to permute the leads on the sample. I highly recommend making a diagram (LEFT) in your notebook to tell you how the sample corners connect to sockets of the puck, as you will reassign the V's and I's.

Very resistive samples (1 mega-ohm < R < 5 giga-ohm): 2-wire ETO



If sample resistance gets above 1 mega-ohm, there is another mode in ETO which allows us to get to 5 giga-ohms. We use an AC voltage and measure the AC current. The requirement is that **ONLY the I+ and V- leads are allowed to go back to ETO**, otherwise the method will not work. This means you can mount the sample as shown on left, or mount it for 4-wire and use the grey Lemo breakout box (see next section) to break the I- and V+ leads when you want to go to 2-wire.

<u>Note about 2-wire measurements:</u> this should be done on one sample at a time – don't try to measure two samples simultaneously.

Using the Grey Lemo Breakout Box



This box provides a way for users to manipulate the electrical signals going to/from your device under test.

There is the "Q" side with a panel of 12 banana sockets which connects to QD electronics using the grey Lemo plug for that option (ETO, Resistivity,...). The numbers by each socket go from 3...14 which is the numbering convention for pins on the puck, and the mapping of these numbers to their functions is given below, or you can look on the plastic overlays for the Puck Wiring Test Station P150.

Then there is the "D" side, also having a 12 socket panel, which goes to your device in the sample chamber. When measuring low level signals or running higher currents, pay special attention to the "PAIR" labels which indicate the twisted pairs on the chamber (3/4, 5/6, 7/8...) and run your signals in/out these pairs. Also, twist the banana jumper cables as well as you can to reduce cross-talk between drive and readback pairs and generally reduce inductive pickup in the wiring. In the photo above, the jumpers are NOT twisted so this is an example of how NOT to do it!

The short colored banana plug jumper cables provide the only connection between the "Q" and "D" panels of the box. A useful convention is to use a colored cable for the "+" side of a pair, and a black cable for the "-" side (half of the cables are black).

Use the Grey Lemo Breakout Box as follows:

- As a patch box between QD electronics and your device (in PPMS), for example to:
 - AC couple the ETO voltmeter by putting in a passive RC circuit in-line: see Ch.1 V and Ch.2 I leads in diagram below for the Mod Coil example
 - to permute leads for van der Pauw method
 - check contacts by making 2- or 3-probe measurements
 - test multiple devices on the puck by making use of all 12 contacts on the puck
- this is how you'll hook it up:
 - Plug in QD grey Lemo into the Q panel, and the cable from this box into the PPMS.
 - On Q panel, see table below for mapping from socket number to its function in the electronics.
 - Route banana cable jumpers as twisted pairs from Q panel to D panel

- For D panel, these socket numbers correspond to pads on the puck, and you can consult the puck photos below to help you keep things straight.
- \circ $\;$ Write down in your notebook which PADS your sample connects to.
- IMPORTANT NOTE: if using the MFP or Rotator probes, be sure to plug in the Rotator/MFP Experiment cable into PPMS, and the Breakout Box cable into this, and finally the ETO cable into the Breakout Box. See Chap. 6 of <u>ETO Manual</u> for more info.
- <u>To bring outside bias sources or measurement electronics into PPMS sample chamber.</u>
 - Basic rules for chamber wiring when bringing in outside sources: DC voltage < 50 V; current < 1 amp. Round trip resistance is ~1 ohm.
 - External electronics will connect to the "D" panel of 12 banana sockets.
 - Mixing external electronics and QD electronics : due to risk of damage to ETO electronics (\$20,000!), there is only one case we allow and is described below ("gating samples in ETO"). For any other application, please contact Neil.

Using the press contact assembly for PPMS transport measurements

There are currently two different press contact boards available in the lab:

- 3 sample, in-line 4-probe (PC-RES-FINE)
- 1 sample, van der Pauw (PC-RES-VDP)

Both sets are stored in the ETO User Kit.

Procedure:

With the green press contact board removed from the puck, place the sample on the puck surface so it will be contacted by the pins shown in the diagrams below. **SAMPLE LENGTH:**

PC-RES-FINE : from 4mm to 10mm.

PC-RES-VDP : 3x3mm to 10x10mm (depending on pin placement). Lower the press contact board over the threaded rods, hold in position and secure using the two thumbscrews. You may want to adjust the tension on the thumbscrews to obtain ideal contact resistance to sample.











A film sample is placed under the Channel 1 pins and the press contact assembly is tightened down. Above: side view of sample under the middle 4 pins.

Puck pin assignment (3-14) for each of the contacts on the PC-RES-FINE board. Consult these if you wish to make van der Pauw or other modifications of the usual in-line 4-point measurement. To get material resistivity from this in-line "4-point probe" system, see: Smits, F. M. "Measurement of sheet resistivities with the four-point probe." *Bell System Technical Journal* 37.3 (1958): 711-718. A typical sample (10x3mm) will show (Neil's calc.): rho[ohm-cm] = 2.27*R[ohms]*thickness[cm]



Right: diagram for PC-RES-VDP board showing the pins at 2x2, 4x4, 6x6, or 8x8mm. Each corner (connections T1,T2,T3,T4) maps to multiple pads on the 3-channel Resistivity puck such that channels 1 and 2 measure van der Pauw resistivity while channel 3 measure var der Pauw Hall. NOTE! the Hall V+/V- leads are reversed so you will get the WRONG type (n/p) for carriers. Referencing the puck pins that each corner maps to:

T1: 3, 8, 11 / T2: 5, 7, 14 / T3: 6, 9, 12 / T4: 4, 10, 13

| Probe material | Full-hard beryllium copper, gold-plated over nickel |
|------------------------|---|
| Probe type | Needle point (standard) Pointed, Concave, Serrated (options) |
| Contact resistance | < 100 mΩ |
| Current rating | 3 A |
| Probe travel | 2.65 mm |
| Spring force | 0.9 N at 2 mm travel |
| Durability | 100,000 cycles |
| Functional temperature | 2 – 400 K |



The T1...T4 are labeled like that since there is a FastHall triax-PPMS adapter box specially wired for this van der Pauw assembly such that the triax 1-4 wire up to these corners.

See Neil if you wish to move the pins to different spring sockets.

Custom applications: using the Low Field Coil

There is a long 2-layer superconducting (Nb-Ti) solenoid wound against the inner bore of the main magnet for the Dynacool. Its purpose is to 1) generate small (milli-Oersted up to 20 Oe) fields using current supplied by the user, or 2) to compensate the main magnet's remanent field if used within the Low Field option of Dynacool. We do not currently have the Low Field option (which involves a fluxgate magnetometer to measure the absolute magetic field in the sample space), but we can still provide the info for users to supply small fields if they wish and describing that is the aim of this section.

There is a cable in the "cables" drawer of Dynacool drawer unit which is called Mod Coil Drive Cable. It has a DB-15 at one end and two banana plugs at the other. If plugged into JQR-8 "MOD COIL IN" DB-15 receptacle on back of the Dynacool electronics rack, one can supply current at the banana plugs from a current source. As stated on the cable's bag, the field/current ratio is **67.1 Oe/amp** but we recommend you stay below ~200 mA as I am not familiar with this low field coil ever going much above 20 Oe.

Use Case: making ultra small field scans

First, we assume you are not using the main magnet since the noise and fluctuations in that field are ~0.3 Oe. So, ramp the main field to zero and then turn it off (or close contactor). This part requires Neil to come in, so please arrange to have him come work with you. Connect the Mod Coil cable to your current source and program it in LabVIEW or WinWrap scripting (both examples are available in our lab or on QD website <u>www.qdusa.com</u>).

Custom ETO: AC-coupling the voltmeter

If doing differential resistance dV/dI measurements in ETO, the DC-coupled voltmeter of ETO is not wellsuited: large DC voltage offsets will exist while we try to resolve small AC modulation atop them. Putting a simple RC high pass filter in-line using the breakout box will AC-couple the voltmeter and enable high resolution voltage measurements, see diagram below:

