"Red" Physical Property Measurement System (PPMS) - General Instructions & Info

All users must receive training for the PPMS and for whichever specific options they wish to use. Reserve all necessary time in FOM and log on and off when starting and finishing your experiment.

This document contains general system information and control instructions. The PPMS Hardware manual and the individual option manuals contain more detailed information. Please refer to the specific option SOPs for information on changing options/samples and making measurements. Please consult the manuals and/or the facility staff for help and troubleshooting information.

Model 6000 Controller

This control unit sits in the PPMS cabinet and is the main controller for general hardware operations. While system controls are generally performed via the MultiVu software, all hardware control operations can also be performed using the Model 6000. This is primarily useful if the computer/software crashes or is unavailable for any reason. In some cases, changing options requires interchanging cable connectors on the back of the Model 6000 (see specific option SOPs).

MultiVu

Main hardware controls

PPMS MultiVu is the main interface system for all hardware and measurement controls. The bottom of the window (see below) contains the main hardware system indicators, which can be used to access their respective controls by clicking the appropriate area. From right to left:



- EverCool Indicates the status of the EverCool II helium compression/circulation system. The normal status is shown above. When compression is required (usually during magnet ramps and briefly during chamber purging), the pressure indicated on the top line will rise and that bar will expand and change from green to yellow. The system will also become significantly louder during this time. Users will generally not need to control the EverCool system. In some cases, it may be desirable to disable the cold head rotation in order to reduce the noise level on high-sensitivity measurements; consult facility staff if you think this will be necessary.
- Chamber Indicates the sample chamber pressure, the gas control state, and the helium level. Normal operating pressure is 10 15 torr (helium). Control buttons include Seal (seal off chamber from pump and gas inlets), Purge/Seal (chamber is purged with helium gas and pumped three times, then sealed), Vent/Seal (chamber is purged with helium gas, then sealed), Pump Cont. (chamber is pumped continuously), and Vent Cont. (chamber is continuously flooded with helium).
- Magnet Indicates the magnetic field and state of the magnet, the field setpoint, field ramp rate and approach mode. Control fields include: Setpoint (-90,000 to 90,000 in Oe where 1 T = 10,000 Oe), Rate (0 to 178 Oe/sec), Approach (Linear/No O'shoot/Oscillate see manual for definitions), and Mode (Persistent/Driven). The normal operating state is Persistent, meaning a current (or no current for 0 field) is flowing through the superconducting electromagnet without any driving voltage. When the field is changed, a switch is activated (the state briefly reads "SW Warm"), a voltage is applied to change the drive current and thus the field (the changing field is indicated along with "Ramping"), then when the field is reached, the switch is opened ("SW Cool") and the field

stabilizes. Under Driven mode, the switch always remains closed. This allows for faster changing between fields, but uses significantly more helium due to ohmic heating of the active switch. Do not apply a magnetic field if the helium level is below 60%.

- Temperature Indicates the system temperature and control state, the temperature setpoint, and the ramp rate and approach mode. Control fields include Setpoint (1.9 to 400 K), Rate (0 to 20 K/min), and Mode (Fast Settle/No O'shoot see manual for definitions). Note that the software will accept ramp rates up to 20 K/min to be input, but the system will likely not actually ramp this fast. Actual maximum ramp rates depend on the option being used and the temperature range. The temperature should not be immediately changed from high temperatures to < 10 K. In such cases, set the temperature to 10 K, wait for 10-30 minutes, then continue to the lower temperature. This practice should also be used within sequences.
- Sequence See below.

Data and sequences

While hardware operations can be controlled directly through the Model 6000, MultiVu is required for saving data and running automated sequences. In general, data from any measurement option can be saved for immediate measurements (these are generally handled through the option control windows – see option SOPs for details) or for measurements as part of a sequence. In general, the user has to specify to have data from immediate measurements saved, and data from measurements in sequences is automatically saved. Data is saved in .dat files, which are commadelimited text files that can be imported into any standard analysis program. Opening a .dat file within MultiVu (this can be done in the main software window, or by clicking the View button within the data file area of the active option control window) will open a graph by default. Right clicking brings up a menu which includes Data Selection for changing the data axes and ranges (multiple Y axes are available) and Table View for viewing the data in table form. Once a file has been opened as a graph, MultiVu also creates an additional file with .gph appended to the file name; this file automatically saves the graph with the most recent data and axes.

Sequences are sets of control and measurement commands which can be used to run automated experiments. They are saved as .seq files, which appear in MultiVu as a simple series of command lines. The right side of the MultiVu screen is the menu of sequence commands. Double-clicking any item will add that command to the sequence above the line in the sequence which is highlighted. Double-clicking on a line within a sequence allows the command parameters to be edited. All the operations listed at the top of this page and many more are available. Commonly-used commands include temperature and field scans, during which measurements can be taken at specified intervals. The list of available measurement commands depends on the active option, and generally needs to be expanded by clicking "+" next to the option name. After writing a sequence, it must be saved, at which point clicking Run on the left side of the screen or the "play" button on the top toolbar will start the sequence. The current sequence status is given in the bottom (see above) and left sections of the screen. Some notes on sequences:

- Individual or multiple (using Ctrl-click or Shift-click) lines can be selected and cut, copied, pasted, or deleted.
- Right-clicking a selected line or set of lines allows it to be disabled, so it can be excluded from a running of the sequence without being permanently deleted from it.
- All temperature and field scans will automatically have an "End scan" line at the end. Any lines within the scan will be indented and will be executed at each point within the scan.
- Within a temperature scan, Sweep is a third Mode in addition to Fast Settle and No Overshoot. In this mode, measurements are made as the specified temperature is reached, and the temperature continues sweeping without ever settling. This is useful for very fast measurements (most electrical measurements), but is not recommended for measurements that take any appreciable amount of time (most magnetic measurements).
- At the end of all sequences, set the system temperature to 300 K and the magnetic field to zero Oe (persistent).

Physical Property Measurement System (PPMS) - AC Measurement System (ACMS)

All users must receive specific training for the ACMS option regardless of training on other options. Reserve all necessary time in FOM and log on and off when starting and finishing your experiment. Keep in mind that the ACMS can take a long time to equilibrate temperature and plan accordingly when making reservations.

Sample Preparation

Refer to Chapter 3 of the ACMS manual for information on sample mounting and installation. Samples must be attached to the end of the sample rod; we have 3 rods, which should be kept in the long plastic tube on the wire shelf when not in use. There are two main



devices for mounting the sample to the rod – a plastic straw and the rod extensions with "bucket" style holders.

- When using a straw, securely place the sample roughly in the middle of the straw. Cut one end (~40% of the length) of the straw off and attach the other end to the black plastic straw adapter (these are kept in the ACMS Options case). Use the Pd reference sample (also kept in the case put it back there when done!) to verify that the distance between the sample and the end of the rod is correct, and adjust if necessary. Attach the straw adapter to the end of the sample rod.
- The bucket can be particularly useful for liquid or powder samples, but can also be used for solid samples. There are two different bucket/rod extension assemblies with different labels on their storage tubes: the "sealed" assembly is better for liquids and is simply fully tightened to seal; the "short" assembly does not seal and can be tightened to a range of heights along the rod extension's threading. In both cases, the sample must be rigidly in place relative to the rod during testing (particularly DC magnetization, where the sample moves), so packing tightly with cotton or some other means should be used to keep it from moving.

Activation

- 1. In the MultiVu software, de-activate any other active option, and physically remove the cables/hardware as necessary according to the respective SOP.
- 2. Vent the system (if necessary), remove the baffle/flange rod, and place it in the cabinet.
- 3. Visually verify that there is not a puck in the system, and if there is, remove it (see the "Measurement Procedure" section of the Resistivity or AC Transport procedures).
- 4. Insert the ACMS coil set, making sure the key engages (this will occur with the white dot forward and slightly right of center). Remove the screw and keep it in a secure place.
- 5. Place the ACMS transfer head on top of the system and secure the flange seal by pushing the wings in.
- 6. Click Utilities Activate Option... and open the ACMS connection diagram and connect all cables accordingly. Note that if the AC Transport option has been used more recently than the ACMS option (check the log book to determine this if necessary), two cables on the back of the Model 6000 controller will need to be changed.
- 7. Select and activate the ACMS option. The ACMS log should appear and should indicate that the option has been successfully activated. Note that the User temperature field on the Model 6000 will now be active and the temperature reading in the MultiVu software should indicate this temperature.

Measurement Procedure

Refer to Chapter 4 of the ACMS manual for information on performing measurements. Once the option is activated, the ACMS Control Center window contains most commands needed to set up and perform a measurement. To insert a sample:

- 1. Use the Sample tab and click the Change button. This will vent the system and home the motor.
- 2. Remove the cap and insert the sample rod.
- 3. Replace the cap and click the Finished button to purge and seal.
- 4. Click the Locate Sample button to locate the sample using either DC or AC measurement with some appropriate applied field and scan parameters. When the locate operation is complete, the expected and actual waveforms will be displayed, as well as the sample location. If the location is more than 0.4 cm off center (positive distance means it is high, negative means it is low), it will need to be removed and adjusted.

When the sample is properly located, measurements can be made immediately or as part of a sequence:

- First, a data file must be selected using the Datafile tab. In the Sample tab, immediate measurements can be made by clicking the AC Measure or DC Measure button and specifying the desired parameters (range plus number of scans to average for DC, frequency and amplitude of oscillation and duration/count for AC). Results of an immediate measurement will be displayed when the measurement is complete and can be written to the specified data file.
- For information on setting up and running sequences, refer to the PPMS general instructions. ACMS measurement commands are listed under "ACMS" (click the + to expand the list of choices) and are similar to immediate measurements.
- For AC measurements, a series of multiple frequencies and/or amplitudes can be specified within one measurement command line.
- All measurement results (plus other data as specified in the PPMS Data tab) will be saved to the specified data file.

To change samples, set the system temperature to 300 K and the magnetic field to 0 Oe (persistent); this should be done at the end of each sequence, but can be done manually if necessary. Use the sample change sequence as described above. When finished, the ACMS option can be left active in the system.

- 1. Set the system temperature to 300 K and the magnetic field to 0 Oe (persistent).
- 2. Vent the system and remove the cap from the ACMS transport head. If there is a sample rod installed, it must be removed <u>before de-activating the ACMS option</u>. Use the sample changing procedure at the bottom of the previous page to remove the sample.
- 3. If a sample has been removed, the system will purge and seal at the end of this procedure, so it must be vented again.
- 4. Click Utilities Activate Option... and select and de-activate the ACMS option.
- 5. Pull the flange seal wings out and lift the transport head off the top of the system. Leaving the cable attached, place the head securely in the holder on the shelf.
- 6. Remove the coil set by placing a screw in one of the tapped holes and lifting the coil set out. Leave the screw in and place the coil set in the cabinet.
- 7. Remove the ACMS amplifier/LEMO connector from the back of the probe and, if necessary, the connectors from the back of the Model 6000 controller. Hang the LEMO connector from one of the hooks on the wall.

Physical Property Measurement System (PPMS) - Resistivity (DC)

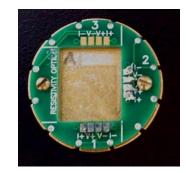
All users must receive specific training for the Resistivity option regardless of training on other options. Reserve all necessary time in FOM and log on and off when starting and finishing your experiment.

The Resistivity option is used for making basic DC resistance measurements by applying a current and measuring the resulting voltage. Resistance measurements can also be made using the AC Transport option. Which option is more appropriate depends on several factors, including whether an AC signal is desirable/acceptable, the resistance of the device(s) to be tested, and the number of devices to be tested. Consult the system documentation and/or facility staff to help determine which option is appropriate. The three "Transport..." PDF documents on the PPMS computer desktop are a particularly useful resource for making electronic transport measurements.

Sample Preparation

The Resistivity option performs a 4-point resistance measurement on up to 3 channels at a time. The standard

Resistivity puck (see right) includes pre-mapped pads for + and - current and voltage for Channels 1-3. The recessed area measures $11.6 \text{ mm} \times 13.6 \text{ mm}$ (with rounded corners), so samples that fit within this area will work best. The base is conducting, so an insulating layer such as Kapton tape will be necessary for samples with conductive back sides. Electrical contact between the sample and the desired channel input pads can be made via wire bonding, soldered or silver-painted wires, spring-loaded press contacts (consult staff on use of these), or other methods. Alternatively, samples can be mounted on the universal puck, which has a blank face and no pre-configured channel set-up. This can be useful for larger samples or if no Resistivity pucks are available. The pin-to-channel



correspondence for the user puck is posted on the bulletin board. The Resistivity or universal puck can be used in combination with the P-150 test box (the red face plate corresponds to the Resistivity option channels) to test sample-to-puck connections in advance of insertion into the PPMS using a hand-held ohm meter or other device.

Activation

- 1. In the MultiVu software, de-activate any other active option, and physically remove the cables/hardware as necessary according to the respective SOP.
- 2. Attach the Resistivity LEMO connector to the back of the probe head.
- 3. Click Utilities Activate Option... and select and activate the Resistivity option. The Resistivity Option control window should appear if the option has been successfully activated.
- 4. If not immediately inserting a sample, insert the baffle/flange rod and clamp it in place, then purge and seal the system. Or, go to step 3 under Measurement Procedure to install a sample.

Measurement Procedure

Refer to Chapter 3 of the Resistivity manual for information on performing measurements. Once the option is activated, the Resistivity Option control window contains most commands needed to set up and perform a measurement. To insert a sample:

- 1. Click the Install/Remove... button and follow the on-screen instructions.
- 2. When the system is vented, remove the baffle/flange rod.
- 3. Visually verify that there is not a puck in the system, and if there is, remove it.
- 4. Place the puck in the puck insertion tool:
 - a. Flip the switch on the handle up to open the ring.
 - b. Put the puck into the bottom of the tool, sample side up.

- c. Hold the puck in place while flipping the switch down to lock the puck in place.
- 5. Using the tool, slowly lower the puck into the probe. When it reaches the bottom, rotate it until the key aligns with the key slot (this happens with the key facing forward), then press down to engage the pins. Do not press too hard if the pins are not engaging properly, there may be damage which could be worsened.
- 6. Flip the switch on the tool up and withdraw the tool.
- 7. Replace the baffle/flange rod and finish the install procedure in the software to purge and seal the system.
- 8. Place the puck insertion tool back on the wire shelving unit.
- 9. Under the Datafile header, click Browse... and set up a file location and name. Once this is defined, you will be prompted to enter sample info for any channels to be used. This includes the length and cross-sectional area to be used in the resistivity calculation; these can be ignored if the sample geometry is not appropriate for a simple bar geometry calculation of resistivity.

When the sample is inserted, real-time resistance readings can be seen via the bridge set-up, which must be defined by clicking the <code>Bridge Setup...</code> button. In the subsequent window, parameters for each of the three channels (plus Channel 4, which is not used) can be specified. See page 2-10 of the Resistivity manual for details. Note that Channel Set dictates whether the excitation current is being applied or not, and that the measurement can be limited by any one of current, power, or voltage depending on the specific limits specified and the sample characteristics (the graph on page 2-12 provides a nice illustration of this). Despite the "DC" designation, all measurement excitations are actually single-pulse measurements at 60 Hz (corresponding to the input line frequency), and Drive Mode dictates whether this is a single positive pulse (DC) or a full-cycle bi-polar pulse (AC, which is the default). The right side of the window gives the "Status" of each channel – the specified parameters are applied whenever the channel is on, and the current and resistance are displayed.

For data to be saved, a "measurement" (as opposed to a simple real-time indication) must be made, either in immediate mode or as part of a sequence.

- Immediate measurements can be made by clicking the Measure... button and using the Resistivity Quick Measure window. The channel(s) to be measured and the number of readings to average are specified. Note that the bridge needs to be defined again within the measurement window by clicking Bridge Setup... Click Measure to begin the measurement. Results are displayed and can be saved to the specified file (see above) by clicking Save.
- For information on setting up and running sequences, refer to the PPMS general instructions. Resistivity measurement commands are listed under "Resistivity" (click the + to expand the list of choices) and are similar to immediate measurements. The Resistivity measurement definition window again allows the bridge set-up to be defined using the Bridge Configuration tab, and the Data Fields tab allows the user to select what parameters and results (from both the measurement and the system hardware) will be saved to the file.

To change samples, set the system temperature to 300 K and the magnetic field to 0 Oe (persistent); this should be done at the end of each sequence, but can be done manually if necessary. Use the sample change sequence as described above (with slight differences for puck removal). When finished, the Resistivity option can be left active in the system.

- 1. Click Utilities Activate Option... and select and de-activate the Resistivity option.
- 2. Remove the Resistivity LEMO connector from the back of the probe and hang the LEMO connector from one of the hooks on the wall.

Physical Property Measurement System (PPMS) - AC Transport (ACT)

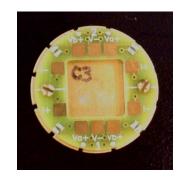
All users must receive specific training for the AC Transport option regardless of training on other options. Reserve all necessary time in FOM and log on and off when starting and finishing your experiment.

The AC transport option can used for making several different measurements with AC excitation: resistivity, I-V curves, Hall Effect, and critical current. Resistance measurements can also be made using the Resistivity option. Which option is more appropriate depends on several factors, including whether an AC signal is desirable/acceptable, the resistance of the device(s) to be tested, and the number of devices to be tested. Consult the system documentation and/or facility staff to help determine which option is appropriate. The three "transport..." PDF documents on the PPMS computer desktop are a particularly useful resource for making electronic transport measurements.

Sample Preparation

The AC Transport option performs measurements using 4 or 5 contact points on up to 2 channels at a time. The

standard ACT puck (see right) includes pre-mapped pads I+, I-, V_A +, V_B +, and V_B - on both Channels 1 & 2. The recessed area measures 10.8 mm square (with rounded corners), so samples that fit within this area will work best. The base is conducting, so an insulating layer such as Kapton tape will be necessary for samples with conductive back sides. Electrical contact between the sample and the desired channel input pads can be made via wire bonding, soldered or silver-painted wires, spring-loaded press contacts (consult staff on use of these), or other methods. Alternatively, samples can be mounted on the user puck, which has a blank face and no pre-configured channel set-up. This can be useful for larger samples or if no ACT pucks are available. The pin-to-channel correspondence for the user puck is



posted on the bulletin board. The ACT or user puck can be used in combination with the User Bridge (the blue face plate corresponds to the ACT option channels) to test sample-to-puck connections in advance of insertion into the PPMS using a hand-held ohm meter or other device.

<u>Activation</u>

- 1. In the MultiVu software, de-activate any other active option, and physically remove the cables/hardware as necessary according to the respective SOP.
- 2. Click Utilities Activate Option... and open the ACT connection diagram and connect all cables accordingly. Note that if the ACMS option has been used more recently than the AC Transport option (check the log book to determine this if necessary), two cables on the back of the Model 6000 controller will need to be changed.
- 3. Select and activate the AC Transport option. ActStatus.log will pop up to indicate the activation status.
- 4. If not immediately inserting a sample, insert the baffle/flange rod and clamp it in place, then purge and seal the system (note this is not necessary if switching from the Resistivity option). Or, go to step 3 under Measurement Procedure to install a sample.

Measurement Procedure

Refer to Chapter 5 of the AC Transport manual for information on performing measurements. Once the option is activated, the tabbed AC Transport Control Center contains most commands needed to set up and perform a measurement. To insert a sample:

- 1. On the Install tab, click the Vent button and follow the on-screen instructions.
- 2. When the system is vented, remove the baffle/flange rod.
- 3. Visually verify that there is not a puck in the system, and if there is, remove it.

- 4. To place the puck in the puck insertion tool:
 - a. Flip the switch on the handle up to open the collar.
 - b. Put the puck into the bottom of the tool, sample side first.
 - c. Hold the puck in place while flipping the switch down to lock the puck in place.
- 5. Using the tool, slowly lower the puck into the probe. When it reaches the bottom, rotate it until the key aligns with the key slot (this happens with the key facing forward), then press down to engage the pins.
- 6. Flip the switch on the tool up and withdraw the tool.
- 7. Replace the baffle/flange rod and finish the install procedure in the software to purge and seal the system.
- 8. Place the puck insertion tool back on the wire shelving unit.
- 9. On the Data File tab, click Browse and set up a file location and name. Then, you will be prompted to enter sample info on the Sample 1 and Sample 2 tabs; click Next >> and Finish after each one, respectively.

All measurements made using AC Transport fall under one of four modes corresponding to the four buttons which are always visible at the bottom of the AC Transport Control Center (regardless of which tab is open): Hall Coefficient, Resistivity, IV Curve, or Critical Current. Clicking one of these buttons will open a new window which serves as the interface for that measurement. Since each interface and measurement procedure is unique, users should refer to the relevant sub-section of section 5.3 of the AC Transport manual for specific information on a measurement type. In general:

- Immediate measurements can be made by clicking the Measure button at the bottom of the measurement window. Results are displayed and can be saved to the specified file (see above) by clicking Save.
- Within the AC Transport Control Center, the Waveforms tab displays the expected and actual data waveforms for every measurement. This can be useful for seeing noise levels and determining if the input parameters and measurement range are appropriate.
- For information on setting up and running sequences, refer to the PPMS general instructions. ACT measurement commands are listed under "AC Transport" (click the + to expand the list of choices). When a measurement command is selected, the measurement interface includes tabs for Sample 1 and Sample 2; the measurement can be made on either or both samples (each with their own separate parameters) by clicking the respective Measure check-boxes, whereas one or the other is selected in an immediate measurement. The PPMS Data tab allows the user to select what parameters and results (from both the measurement and the system hardware) will be saved to the file.

To change samples, set the system temperature to 300 K and the magnetic field to 0 Oe (persistent); this should be done at the end of each sequence, but can be done manually if necessary. Use the sample change sequence as described above (with slight differences for puck removal). When finished, the AC Transport option can be left active in the system.

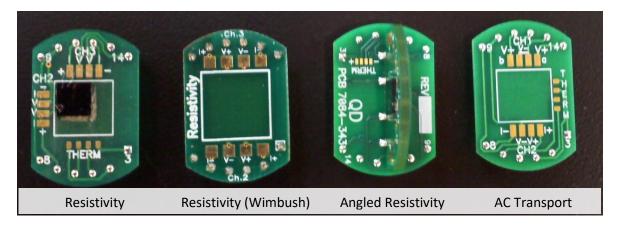
- 1. Click Utilities Activate Option... and select and de-activate the AC Transport option.
- 2. Remove the AC Transport LEMO connector from the back of the probe and, if necessary, the connectors from the back of the Model 6000 controller. Hang the LEMO connector from one of the hooks on the wall.

Physical Property Measurement System (PPMS) - Horizontal Rotator (HR)

The Horizontal Rotator is not technically an option; it is an accessory which can be used in conjunction with the Resistivity and AC Transport options. Any measurement that can be made with those options can be made with the rotator, with two exceptions: the current limit is reduced from 2 A to 0.5 A in ACT, and only two channels are available in Resistivity. The Horizontal Rotator consists of a motor and the rotator probe body. The motor control is always active, so it simply needs to be connected via the control cable, mounted on the system, and coupled to the rotator body (which is installed in the probe space) in order to be used. The probe can also be rotated manually outside the system using the motor coupling. The positions are defined such that the sample faces up at 0° and forward at 90°. Slightly more than one full rotation is possible so that the end positions are defined as -10° (going past 0 to 350 on the dial) and 370° (going past 0 to 10). An on-board thermometer is used rather than the main PPMS system thermometers.

Sample Preparation

The Horizontal Rotator uses special sample holders rather than the pucks which are used with the Resistivity or AC Transport options. Samples can be connected to them in the same manner as the pucks, and connections can be tested in the user bridge via the appropriate adapter (all parts referred to in this section are kept in the Horizontal Rotator accessory kit). The sample holders are shown below.

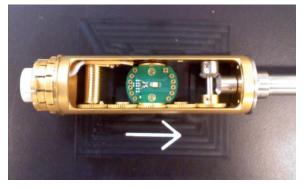


Resistivity – Channel 1 is designated for the thermometer – DO NOT USE THESE PADS. Channels 2 and 3 are for 4-point resistance measurements.

Resistivity (Wimbush) – Third-party version of the Resistivity holder with several improvements: larger sample area, clear "Resistivity" label, larger contact pads, and no exposed thermometer pads.

Angled Resistivity – Identical connections to the Resistivity holder, but with a second perpendicular board coming out of the main board so that the sample can be rotated within its own plane.

AC Transport –For 5-point Hall measurements, Channel 1 must be used; for other measurements, either Channel 1 or 2 may be used. Only the voltage pads are associated with the specific channels, and there are only two current leads which are shared by the channels and used for the active measurement at any given time. See page 7-4 of the AC Transport manual for details.



Once the sample is mounted to the holder, the holder must be attached to the rotator. Manually set the rotator position to 90° and use the rotator support tool to securely rest it on the table as shown to the left. Then, press the sample holder into the rotator to engage the pins; note that the pin configuration is asymmetric, so it will only attach in one orientation. The sample removal tool is designed for this purpose and can grip the sample holders at the curved edges.

Rotator Insertion and Motor Set-up

- Click Utilities Activate Option... and open the connection diagram for the appropriate rotator/option combination and connect all cables accordingly.
- 2. If necessary, select and activate the Resistivity or AC Transport option.
- 3. If necessary, vent the system and remove the flange/baffle rod assembly.
- 4. Visually verify that there is not a puck in the system, and if there is, remove it.
- 5. Place the rotator motor assembly on top of the system. Do not secure the flange yet. Move the motor off to one side as shown to the right.
- 6. Turn the rotator to -10°.
- 7. Carefully insert the rotator into the probe space. After the o-ring engages in the motor support, lift the support slightly so that it sits freely between the o-ring and the indicator dial. Turn the entire rotator until it the key engages and it drops into place. Then, firmly push it down to engage the pins.



- 8. Push the motor assembly down to re-engage the o-ring, then push the wings in to secure the flange.
- 9. Next, the rotator's on-board thermometer must be activated. Open the program PPMS Rom Config 32 (taskbar icon with magnifying glass and chip) and click the "Diag. all" check box. Then, click Send to PPMS Send Config and open the appropriate configuration file (HRE-442 for Resistivity or HREACT-442 for ACT). The User temperature on the Model 6000 will now be active, and the temperature in MultiVu will match it.
- 10. Open the motor control window by clicking Instrument Motion... The full range of -10° to 370° should be indicated on the scale, as well as the current motor position. Click Go to Index to move the motor to the end position of -10°.
- 11. The motor and the rotator should now be in the same position and can be coupled by moving the motor to the center and lowering it onto the coupler.

Rotator Control

The control window described in step 8 above is used for immediate control of the rotator. To move to an angle, simply type the desired angle in the "Move to:" box and click Set. At this point, an immediate measurement can be made using the active option as described in the appropriate option procedures. Note that there is a slight backlash, so a given position may differ by a few degrees depending on from which direction it is approached. **Do not use "Redefine Current Position:".** If you think something is wrong with the position definitions, consult the facility staff. When using sequences, "Set Position" and "Scan Position" are available in the command menu; these are used similarly to the equivalent commands for temperature and magnetic field.

- 1. First, the on-board thermometer must be de-activated. Open the program Mon6000 (taskbar icon with a blue 32) and enter the command line "usertemp 0". The User temperature on the Model 6000 will now be blank.
- 2. Vent the system.
- 3. Lift the motor to de-couple it from the rotator and move it to the side.
- 4. Carefully remove the rotator from the probe space, turn it to 90°, and set it on the rotator support tool on the table.
- 5. Remove the motor assembly from the flange and hang it from the designated hook on the wall.
- 6. Insert the flange/baffle rod assembly and purge and seal the system.
- 7. If necessary, de-activate and disconnect the active measurement option as described in the option procedures.
- 8. Remove your sample from the rotator and leave the rotator in the storage cabinet.

Physical Property Measurement System (PPMS) - Vibrating Sample Magnetometer (VSM)

All users must receive specific training for the VSM option regardless of training on other options. Reserve all necessary time in Coral and enable and disable when starting and finishing your experiment. Standard and large-bore coil sets are available; each has their own separate kit, guide tube and sample rods. The tube and rods with blue color coding go with the large-bore set.

Sample Preparation

Refer to Chapter 3 of the VSM manual for information on sample mounting. This includes detailed information about acceptable mounting materials/adhesives and effects of sample shape. Low-temperature varnish, double-sided Kapton tape, and other materials are available in the facility. Samples must be attached to the end of a sample rod, which should be kept in the cabinet when not in use. There are two main devices for mounting samples – the paddle (quartz or brass) and the brass trough. In both cases, samples should be mounted 30-35 mm from the end of the mounting device; this distance can be easily measured using the "sample mounting station", which should be stored with the transport head. The paddle is best for film samples mounted parallel to the magnetic field. The brass trough should be used for other samples, including perpendicularly mounted films, bulk pieces, or powders, using the quartz/Delrin rod sample braces or polypropylene powder holders as necessary. Sample holders should be cleaned after each use to remove any residue from the sample and/or mounting materials. Please keep solvents away from the plastic threaded connector at the top ends of the holders, and be careful with the quartz paddles, which are very delicate.

Activation

- 1. In the MultiVu software, de-activate any other active option, and physically remove the cables/hardware as necessary according to the respective SOP.
- 2. Vent and seal the chamber if necessary and remove the baffle/flange rod.
- 3. Visually verify that there is not a puck in the system, and if there is, remove it (see the "Measurement Procedure" section of the Resistivity or AC Transport procedures).
- 4. Insert the appropriate VSM coil set using the puck insertion tool, making sure the key engages.
- 5. Insert the appropriate VSM sample guide tube and verify that the o-ring is properly seated in the flange.
- 6. Remove the dust cap from the bottom flange of the VSM transfer head. Place the head on top of the sample tube and secure the flange with the standard clamp. [Note The head is quite heavy. Ask for help if necessary.]
- 7. Connect the VSM pre-amp/LEMO cable to the back of the sample chamber.
- 8. Click Utilities Activate Option... to open the Option Manager. Select and activate the VSM option. The VSM log should appear and eventually indicate that the option has been successfully activated. Note that the User temperature field on the Model 6000 will now be active and the temperature reading in MultiVu should indicate this temperature.
- 9. In the VSM control center, click Configure VSM System and enter/verify the serial number for the coil set in use 501 for the standard bore and LB690 for the large bore.

Measurement Procedure

Refer to Chapter 4 of the VSM manual for information on performing measurements. Once the option is activated, the VSM control center window contains most commands needed to set up and perform a measurement. To insert a sample:

1. Use the Insert tab and click the Install/Remove Sample button, which opens the VSM Insert/Remove Sample Wizard.

- 2. Click the Open Chamber button. This will move the motor to the load position and continuously vent the chamber, and if necessary, bring the chamber to room temperature.
- 3. Manually seal the chamber using the chamber controls module in order to conserve helium gas.
- 4. Click the Next>> button. The Wizard will not explicitly tell you to insert the sample, but it should be done now. Remove the cap and insert the sample rod; you should feel the magnets at the top of the rod firmly attach to the top of the motor. [Note Due to this magnet, the sample may experience a field of up to 200 Oe during insertion. If this is unacceptable, contact QD Support for advice.] Replace the cap.
- 5. Click Browse to specify a data file name and location, and enter sample properties if desired, then click Next>>. All measurement data will be saved to the selected file automatically.
- 6. The sample centering screen lets the user specify the exact sample location, which is necessary for accurate measurements. The Enter Offset manually button lets you simply enter the location as measured in the mounting station (use the location of the center of the sample). For best results, have the system measure to accurately determine the location. Apply a field necessary to induce a moment in the sample, then click the <== Scan for Sample Offset button. The system will scan the sample (potentially multiple times to find the best range) and report the measured location. Make sure that this matches the expected location.
- 7. Click Next>>, then Close Chamber. Wait for the purge and seal operation to complete.
- 8. Click Finish to exit the wizard.

When the sample is inserted, measurements can be made manually or as part of a sequence.

- Click the Measure... button to open the measurements window. The Settings tab allows you to specify a single or continuous measurement (sample is constantly vibrated, and measurement results are logged at specified interval) and the averaging time (1 second averaging yields ~10⁻⁶ emu precision; noise level is inversely proportional to square root of averaging time). The Centering tab includes controls for automatically centering the sample at defined intervals; note that it will center every 10 minutes by default, which may not be desirable. The Advanced tab can be used to edit measurement parameters.
- For information on setting up and running sequences, refer to the PPMS general instructions. VSM measurement commands are listed under "VSM" (click the + to expand the list of choices) and include the functions above plus "Moment vs. Field" and "Moment vs. Temperature" functions, which are essentially automated versions of Scan Field or Scan Temperature loops with continuous VSM measurements. Use caution when operating Moment vs. Field in sweep field mode to avoid excess liquid helium usage.

<u>De-activation and Removal</u> [Note – All VSM users must complete this process when finished UNLESS it is known that the next scheduled user will be using the VSM.]

- 1. Set the system temperature to 300 K and the magnetic field to 0 Oe (persistent).
- 2. If there is a sample rod installed, it must be removed before de-activating the VSM sample option. Use the VSM Insert/Remove Sample Wizard to remove the sample.
- 3. Once the sample has been removed, simply click Cancel to exit the wizard. Replace the transport head cap.
- 4. Click Utilities Activate Option... and select and de-activate the VSM option.
- 5. Remove the flange clamp and lift the transport head off the top of the system. Leaving the cable attached, place the head securely in the holder on the wooden case. Cover the bottom flange with the dust cap.
- 6. Remove the sample guide tube and place it in the cabinet.
- 7. Using the puck tool, remove the coil set and place it in the appropriate VSM kit case.
- 8. Remove the VSM amplifier/LEMO cable from the back of the probe and hang it on the wall.
- 9. Place the standard flange/baffle rod in the sample chamber, clamp it shut, and purge and seal the system.