DynaCool 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.

<u>MultiVu</u>

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:



- **Cryostat** Indicates the status of the cryostat and the magnet temperature. This set of controls is not used during normal system operation.
- Chamber Indicates the sample chamber pressure, the chamber 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), Vent Cont. (chamber is continuously flooded with helium), and HiVac (chamber is pumped down to ~10-5 torr; use Purge/Seal first).
- 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 ~220 Oe/sec), and Approach (Linear/No O'shoot/Oscillate see manual for definitions). (Note The magnet only operates in "Driven" mode, so there is no Driven/Persistent option.) When the field is changed, 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 field stabilizes and indicates "Holding".
- Temperature Indicates the system temperature and control state, the temperature setpoint, and the ramp rate and approach mode. Control fields include Setpoint (1.7 to 400 K), Rate (0 to 20 K/min), and Mode (Fast Settle/No O'shoot see manual for definitions). [Note that the system is technically capable of ramp rates up to 50 K/min, but ramping above 20 K/min is detrimental to the long-term health of the system hardware.] Actual maximum ramp rates depend on the option being used and the temperature range. When cooling from high temperatures to < 10 K, pauses for temperature uniformity in the chamber may be automatically built in by the software. A blue background on the temperature line indicates that an option/accessory (i.e. "User") temperature is active and controlling the system.
- Sequence See below.

Data and 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 comma-delimited 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, and the command line in progress is indicated in green. 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. [VSM has its own built-in M vs. H and M vs. T functions, so using the hardware commands for field and temperature scans is generally not necessary in this case.]
- At the end of all sequences, set the system temperature to 300 K and the magnetic field to zero Oe if the sample is to be removed (i.e. no additional measurements planned).

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. Standard and large-bore coil sets are available; each has their own separate kit (including sample holders0, guide tube and sample rod. The tube and rods with blue color coding go with the large-bore set. Because the system is currently only used for VSM measurements, it is not necessary to de-activate and remove the VSM option after use. It may be necessary to change between coil sets before starting measurements by de-activating one and then activating the other.

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 rack 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". 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.

[NOTE – The first two sections should be skipped if the desired coil set is already installed.]

De-activation and Removal

- 1. Verify that the system temperature is set to 300 K and the magnetic field to 0 Oe.
- 2. IMPORTANT: 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. [Note The head is quite heavy. Ask for help if necessary.]
- 6. Remove the sample guide tube and place it in the rack.
- 7. Using the puck tool, remove the coil set and place it in the appropriate VSM kit case.

Activation

- 1. Insert the appropriate VSM coil set using the puck insertion tool, making sure the key engages.
- 2. Insert the appropriate VSM sample guide tube and verify that the o-ring is properly seated in the flange.
- 3. Place the transfer head on top of the sample tube and secure the flange with the standard clamp.
- 4. Connect the VSM pre-amp/LEMO cable to the back of the sample chamber.
- 5. 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 temperature field in MultiVu will now have a blue background, indicating that the VSM coil thermometer is actively reading (incorrectly, due to the other coil set's calibration still being active) and being used to control the system temperature.
- 6. In the VSM control center, click Configure VSM System and enter the serial number for the coil set in use 501 for the standard bore or 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. If the chamber has been at or above room temperature for ~10 minutes or more, manually seal the chamber using the chamber controls module in order to conserve helium gas. If it has been cold, allow it to continue "flooding" (the word used by MultiVu to indicate the continuous venting state).
- 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 (usually multiple times to find the best range) and report the measured location. Make sure that the fit (green curve) matches the measured data (red crosses) and that the reported location matches the visually observed 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. In both cases, point spacing will be dictated by both the integration time and the field or temperature sweep rate. The right side of the window displays a list of fields or temperatures at which points will be generated, and the total amount of time it will take. Consult the VSM manual and/or the facility staff for advice on optimizing these measurements.

After measurements are completed, remove the sample using the Insert/Remove Sample Wizard. Continue with the wizard to begin with a new sample, or cancel the wizard, replace the cap, and purge and seal the chamber to quit.