Created by S. M. Loureiro.

Disclaimer

Please keep in mind that this tutorial was created just to simplify your life with the tasks you need to carry out in order to use the machine in an autonomous way. For technical specifications or advanced considerations, please consult the appropriate Quantum Design PPMS Manuals at your disposal in the measurement instruments room.

Overview

If you are interested in the primary characterization of the transport properties of your samples, you will use the Resistivity Option.

If you are screening for superconductivity, you will use the AC Susceptibility Option with a low magnetic field (<100 Oe).

If you want to analyse the magnetic moment of your sample you will use the DC Magnetization Option with the appropriate magnetic field. Use 1 T (10000 Oe) for routine scans.

Resistivity Measurements

Before placing any puck inside the chamber, check the contact resistance:

A - Plug the puck with the samples mounted into the Testing Station and insert the Resistivity plug into the Testing Station. It should look like this.

B - In the computer, using MultiVU software, option Instrument/Bridge Channels, check for the channels you will be using in the measurement. The current limit may be changed, but try to set as default values 100 ma, 1000 mV, 100mV, Drive mode AC and press Set. The value of the contact resistance will appear on the right side of the screen. It should be in the order of a couple of ohms, if you get something in the order of the mega-ohm, check which contacts are the origin of the problem with the multimeter and solve the problem under the microscope.

C - Remember to plug the Resistivity plug back into the system, otherwise you will wonder why you are not collecting data and have no clue about about it. It should look like this.

Before inserting the puck inside the chamber, first check the following:

Is the Temperature of the chamber above 295K? If not, there will be cryopumping of air into the chamber and we do not want that to happen do we? Set the Temperature for 300K.

Is the field below 1 T ? If you place the insertion tool with a high magnetic field inside there will be a strong force on the insertion tool that may actually overwhelm you and damage the pins while you are inserting the pucks. (Quantum dixit, and even if you doubt this, I would rather you wouldn't engage in any test strengths against the magnet at 9T). :-))) Set the magnetic field for 1T or less.

Now you are ready to insert the puck in the chamber. You will be careful in this operation not to bend the pins !!!!

Disengage the Sample Insertion Tool. The top part should be vertical. Insert the puck in the bottom part of the Sample Insertion Tool. Remember that the sample faces upwards and the holes of the puck should be on the outside. Rotate the puck slightly to check that it is correctly positioned (If it is stuck, try again before placing the sample installer inside the PPMS or you will damage the pins when you do it !). Now you can engage the Sample Insertion Tool by flipping the black switch down. Check if the puck is well levelled with the cylinder of the Sample Insertion Tool.

In the Computer, use the MULTIVU Software, option Sample/Install. This will initiate the venting of the chamber.

Open the clamp, remove the baffles and place the sample installer inside the chamber. Start inserting the Sample Insertion Tool carefully. Pay attention to the position of the key part of the puck (small metal part). If it was rightwards, you should rotate it left once inside, if it was left, you should rotate right. DO NOT FORCE IMMEDIATELY !!! When you have reached the correct position you will feel a slight depression. Press the Sample Installer Tool to plug the puck into the pins. The tab top part of the sample installer will slightly pop. Disengage the Sample Insertion Tool (Remember: black tab in the up position) and remove the sample installer. Do it slowly, specially at the beginning. If you feel resistance, you might not have inserted the puck well and it might have gotten caught in the tool, so remove the puck and insert again.

Now put the baffles back inside again and close the clamp. In the MULTIVU Software, you will notice that the Sample/Install option is still open so, click Finished. This will engage the purge/vacuum operation.

Okay, the puck is finally inside. What shall I do next?

Check for the contact resistance once more by using the MultiVU software, option Instrument/Bridge Channels, the values should be close to or slightly higher than those checked before. If you cannot read the values, some contact may have been broken while you were carrying out the insertion process. Remove and check your contacts again with the multimeter, repair them under the microscope.

How do I get the measurement running?

As much as I would like to shout at some machines to have them do their job, voice activated machines are still under development, so you will actually have to tell the PPMS6000 how to do it. This means writing a sequence (see Writing a Sequence).

Now, I have written several "Standard" Sequences that people can use and avoid all the trouble. Basically they will be explicit enough for you to select them immediately for the appropriate need. As an example, 300-5_5, would mean a zero field scan over the temperature range from 300K to 5K with several other parameters like rates and logging data for a three bridge set up measurement. 400-5_5, is thus obvious enough.

Okay, okay ... can I start measuring now ???

After this is all done, click in the MULITVU software, click Sequence/Run and then View to check the on-line data collection.

AC Susceptibility/DC Magnetization Measurements

Is the PPMS set up for this type of measurement? If not, you may wish to do this first.

Before inserting the sample inside the chamber, first check the following:

Is the Temperature of the chamber above 295K? If not, there will be cryopumping of air into the chamber and we do not want that to happen do we? Set the Temperature for 300K.

Is the field below 1 T? If you place the insertion tool with a high magnetic field inside there will be a strong force on the insertion tool that may actually overwhelm you and damage the pins while you are inserting the pucks. (Quantum dixit, and even if you doubt this, I would rather you wouldn't engage in any test strengths against the magnet at 9T). :-))) Set the magnetic field for 1T or less.

Insertion of the Sample:

Get your sample rod ready with the sample inside the straw or inside the appropriate sample holder. The sample should not be lose inside its container, so you may wish to put some cotton or some teflon tape to prevent this.

Remember to close any 32 bit software that is open (MULTIVU included). There is a conflict between the 32bit MULTIVU and the 16 bit ACDCMag software. Open the ACDCMag.exe software.

Choose the option Sample/Insert/Remove in the menu of the ACDCMag software. This will engage venting procedures and will allow you to place the sample inside the chamber. The top part will gently clip onto the chamber. Put the black cap on again and click Finish in the screen. This will engage the Purge/Seal procedures. (Do not close this menu, it will close on its own)

Centering of the Sample: Before running the measurement you will have to center the sample position. You should use the appropriate sample centering processing according to your measurement condition. This means that you will use an AC centering procedure if you will be measuring AC Magnetic Susceptibility and a DC centering procedure if you will be measuring the DC Magnetization.

AC centering:
In the ACDCMag Sotware, you will just have to select CENTER SAMPLE and check Run. Observe the shape of the curves in the right bottom of the ACDCMag Software window. For a good AC centering the curve should be smooth and anti-symmetric with respect to the axis. If this does not happen, please remove the sample, check if it is carefully tucked within the straw and does not move during the measurement and slighlty change the position of the sample within the straw. If the center position was negative, move it upwards.

DC Centering:
To run an DC centering, please remember to SET THE DESIRED FIELD before running the macro. Check condition #29 that I have set as a DC routine centering procedure. Observe the shape of the curves in the right bottom of the ACDCMag Software window. For a good DC centering the blue curve should either match the yellow one or be perfectly anti-symmetric.

Running the measurement:

In the menu of the ACDCMag software FILE/NEW DATA FILE, change the name of the sample to the one you wish to.

In File, Open the CONTROL PANELS. Size/Enlarge. Select CONTROL PANELS/File/Edit. This is where you will edit or modify your sequence. Set your sequence in a way similar to the

resistivity, with the sole exception that you will have to input the condition # of your desired measurement, if it is DC or AC. This is done with the option ADVICE on the right side of the PPMS Editor.

Go to File/Download and Overwrite and as for the resistivity option check if the sequence you are downloading is indeed the one you wanted too. Also wait for all the registers to be downloaded - the number should be the same.

Go to /RUN and start the measurement. You can go to the ACDC-mag menu and analyze the graphic if you wish.

Removal of the sample:

As stated before, choose the option Sample/Insert/Remove in the menu of the ACDCMag software. This will engage venting procedures and will allow you to remove the straw from the chamber. Put the black cap on again and click Finish in the screen. This will engage the Purge/Seal procedures. (Do not close this menu, it will close by its own).

Congratulations, you're done !!! :-))))

Writing a Sequence

For RESISTIVITY MEASUREMENTS:

In the MULTIVU software option File/New Sequence, set your parameters:

Scan Temperature at the desired rate
Uniform spacing or increments, 1/T or log T
Number of steps (as a function of the other variables)
Sweep (for routine measurements)

In the Measurement Commands/Log PPMS Data/General write the date file name of the sample you will be measuring.

Options Start, Create New File/Version, Repeat every 5s (for example).
Option Log PPMS Data/Standard Items, check for Temperature, Field, and the Bridges + Excitation you are using. (You can skip the diagnostic option but if you want you can check the System Status, Set point, Magnet and Flow Rate options.

After End Scan:

Go to Measurement Commands/Log PPMS Data/General again and check the Stop logging action (instead of start). This will allow you to stop collecting data points at your final temperature. If you donll either be collecting a huge amount of points at your final scanning temperature or at 300K (because you will be kind enough to set this to end your measurement J).

Set Temperature 300K, 10K/m (this is the maximum rate, so even if you put 100 it will still be 10 anyway), in fast settle mode.

If working overnight or on weekends and no on will follow immediately to do the next measurement, set the final Temperature to 100K to save some Helium. Or insert the Shutdown mode instead (it will keep the flow control valve at a rate of 100cc/m throught the cooling annulus

and the system heaters are turned off. The impedance heater is also kept at low power to avoid liquid helium to reach the cooling annulus).

NOTES:

There are obviously other options you may check in the sequence file editor like the Scan Field, etc., if you will be doing measurements under magnetic field. Or for very precise measurements (you will get good data with the previously mentioned options, but if you want to be really picky, picky, you may wish to use the wait option. This means that the program will wait until the variables you have selected are stable enough in order to proceed with the measurement. It will take you much more time, so donroutine data collection!). If you really need to do this, please write several sequences associated to several data files for your own protection. Like from 300 to 200, then scan closely from 200 to 100, then go for a normal rate from 100 to 5K, etc., as a mere example of course !). Also, if using fields higher than 2T (20000 Oe) please set the decreasing field rate for oscillating mode in order to restrain the remnant field to a maximum of 5 Oe).

FOR AC SUSCEPTIBILITY/DC MAGNETIZATION MEASUREMENTS:

The basic procedures are the same as for resistivity measurements. Remember that you will be using the ACDCMag Software and NOT the MULTIVU. In the writing process the only exception is that you will have to specify the type of condition you will be using in your measurement, if it is an AC or DC measurement. This is done using the ADVICE option of the sequence Editor and selecting the desired number.

As for the resistivity option, I have also set up a number of sequences that you can use for your routine measurements.

ACMS Setup

In order to set up the ACMS you will need to plug the ACMS Insert into the system. Please follow these procedures carefully due to the sensible nature of the coil set.

A - In the Control Panel of the PPMS 6000, select the Continuous Venting Mode.

B - Be sure that there are NO resistivity plugs inside the chamber.

C - In the top part of the ACMS Insert, tighten the screw until it blocks.

D - Place that assembly inside the chamber remembering the way you have put the resistivity plug inside. You should press when you fill you have reached the correct position (you will feel the same depression).

E - Remove the screw and be careful not to drop it inside the chamber. It should look like this inside.

F - Place the Sample Transport Assembly of the ACMS and plug the pre-amplifier at the back.

See, it was easy and you're ready to go !!!