

SQUID Operating Instructions

Login:

Rotate clock-wise the brightness and contrast knobs on the monitor; the screen should be blue. Press the ENTER key of the keyboard to access the login screen. Type in your login name, and then your password. Type the number 3 above the letter “e” (the number pad keys on the right side do not work here) to “Run MPMS Program”. The **M**agnetic **P**roperty **M**easurement **S**ystem (MPMS) software will startup.

MPMS Interface:

The screen is divided into 4 areas. There is a **Graph** area in the upper left corner. Under this is the **Status** window, showing the temperature, magnetic field, Helium level, and the current operating mode. On the lower left is the **Command list**, displaying the current and previous commands executed. A **Menu** is on the right side of the screen.

The “Menu” is selected with the **Function keys** at the top of the keyboard. A template around the function keys lists the menus:

F1	Help key
F2	Register Sample menu
F3	Measure Sample menu
F4	Set Parameters menu
F5	Graph menu
F6	Utilities menu
F7	Diagnostics menu
F8	Sequence Control menu
F9	Sequence Editor / File Control menu

On the Menu window is a “light bar” highlighting one of the menu selections. Move this up and down with the arrow keys (↑ or ↓). With a parameter or file name you just start typing in the value, and a box will pop up displaying the keys typed. No need to type Enter to display the box, and no need to erase the previous value. To abort any changes press Esc, or press Enter to accept the entry. A menu item with three periods at the end has a submenu. Press Enter to display the submenu.

With any parameter or command highlighted, pressing F1 will open a help window for that parameter or command. Consult the “Squid Manual” for more help.

Logbook:

Record your experiment time beginning and end (add date if overnight), and helium usage. The logbook is matched to the computer login data. As long as people use the instrument without unnecessarily long breaks and are honest about entering the time used in the logbook, billing can be limited to just experiment time.

Logout:

To exit the MPMS software press Esc. A box will ask, “Exit to DOS? [Y/N]”. Type Y and press Enter to exit.

Type the number 5 to “Log off system”. A blank blue screen will appear. Turn the brightness and contrast knobs counter clock-wise.

Sample Mounting:

There are a variety of ways to mount and position your sample. All methods use materials that have minimal magnetic susceptibility. This describes just a few of the ways to mount a sample. Another example is at the beginning of chapter 2 in the MPMS Users Manual. If you use their method skip step 5, and use our method with copper wire in place of step 6.

Samples are either powder or solid. Powders should be ground very fine. With solid samples be careful of fingerprints, i.e. finger oils. You need a very precise measure of the mass of the sample. This mass is used in the analysis and interpretation of the results. "Typical" sample sizes are 20 to 50 mg. But, samples often range between 5 to 60 mg.

Warning: Keep the grease off the quartz shroud

Don't use straws for work at very high temperatures $> 325^{\circ}\text{K}$.

N.B.: The sample holder rod is very easy to bend and damage irreparably. Do not use force when placing the straw on the rod. Don't use any pliers on the rod.

#3 capsule method

Here are two ways to prepare the sample with a #3 capsule and a clear plastic straw. This example works with solid samples. Place the sample in the #3 capsule. A wad of cotton can be placed in the capsule to hold the sample orientation. Next poke a small hole in the top of the capsule to allow air to be pumped out.

Alternatively, place the sample in the short fat half of the capsule. Then, press the longer thin half of the capsule upside down onto the sample. This works well with power samples. Be careful to minimize the "crawling" of the power up the side between the two capsule halves.

With a razor slit an "S" (some use an "X") on the side of the straw for about 2.5 cm (1") centered about 6cm ($2\frac{3}{8}$ ") from the bottom of the straw. Insert the capsule inside the straw at the slit. Place a piece of Kapton tape over the bottom of the straw, to keep the sample from sliding out the bottom of the straw.

The plastic bag method:

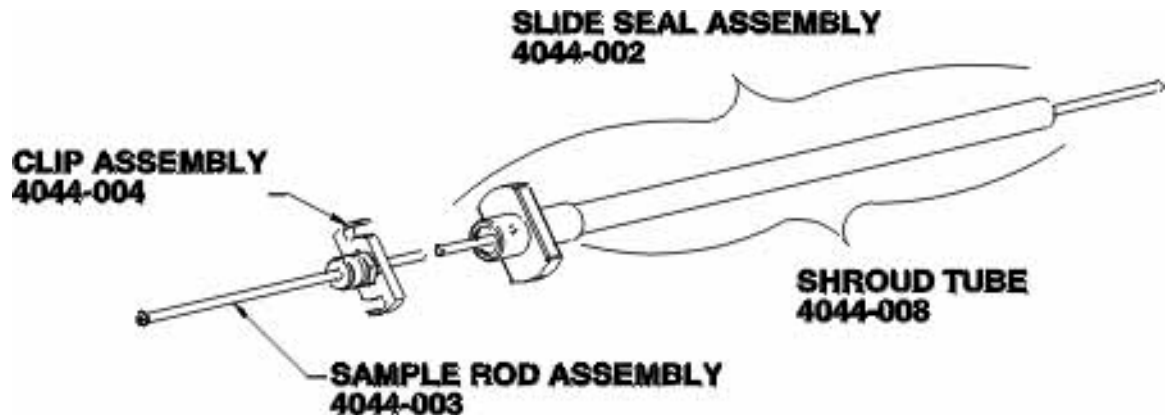
Instead of a #3 capsule a small plastic bag can be made, and the sample sealed inside, for placement of the sample in a straw. This uses the plastic bag sealer and the roll of 2mil plastic. It is reported that there is less noise with the plastic bags in measurements at low temperatures < 5 Kelvin. Prepare a bag about 2 cm x 2.5 cm (these dimensions are very approximate). Place the sample in the bag, and seal the open end. Roll up the bag, and insert into a straw about 5cm. Place a piece of Kapton tape over the bottom of the straw, to keep the sample from sliding out the bottom of the straw.

Mounting Straw on Rod:

Slide the top of the straw over the gold Kapton tape wound around the bottom of the sample holder rod. Pierce a small hole through the straw where it aligns with the hole at the bottom of the sample holder rod. Thread a short piece of the red copper wire

through the hole and loop around twice, then tie a double knot and trim the ends of the copper wire.

Slide the blue “slide seal assembly” with the quartz shroud over the straw. The shroud should pass over the copper wire knot without touching. The rod above the slide seal should not feel dry and should have a thin coat of grease. If necessary apply a small amount of the Apiezon N grease along the rod, running your fingers along the length of the rod to ensure a light even coating.



Check the position of the black “S-clip assembly” with the stainless knurled nut at the top of the sample rod. From the clip assembly (a.k.a. “sliding clamp”) to the center of the sample should be about 126.7 cm (49 ⁷/₈ inches). With the “S-clip assembly” held against the left edge of the bench, check the sample position against the marks on the tape on the bench top. The sample should not extend to the right of the “brick wall” marked on the tape.

Inserting the Sample in the Instrument:

There is a large (5.9cm, 2 1/3") black lever used to open and close the airlock valve between the airlock and the sample chamber. The airlock valve is closed when the lever is horizontal, and open when the lever is vertical. To open the valve, turn the lever clockwise from the horizontal to vertical position. To close the valve, turn the lever counter-clockwise from the vertical to horizontal position.

Remove airlock plug:

When no sample is in the instrument the blue airlock plug should be in place, and the airlock valve should be closed (lever in the horizontal position). If the green "READY" light is on then vent the vacuum by turning the lever slightly from horizontal then back to horizontal. With the green "READY" light off, turn the little black cam levers off the blue plug, and remove the blue plug. Make sure the three O-rings are in place, under where the blue plug sat. If the O-rings are not in place the instrument cannot be used.

Load sample into airlock:

Lift the rod above the instrument and into the space above the false ceiling, then lower the rod with the sample and quartz shroud into the airlock. The white dot should be facing forward on the blue slide seal. Place the blue slide seal where the blue airlock plug sat, and turn the little black cam levers to hold the slide seal.

Purge airlock:

Press the "PURGE AIR LOCK" button. The system will purge the airlock chamber four times, by alternately pumping a vacuum on the chamber followed by venting Helium gas into the chamber. The yellow "VENTING" led will light while venting. When the airlock is purged the green "READY" led will light. If the green led does not light, try to purge a second time.

Lower sample into sample chamber:

Open the airlock valve by turning the lever clockwise to the vertical "OPEN" position. Lower the rod slowly and carefully, without too much force. When the black clip assembly (sliding clamp) is level with the top of the sample transport, rotate the black clip assembly so the curved slots are under the two thumbscrews. **Gently** tighten these two thumbscrews.

Removing the Sample:

To remove the sample, first, loosen the two thumbscrews and rotate the black clip assembly from under the thumbscrews. Slowly and carefully, pull the rod up while rotating the rod. If there is resistance, or if you hear a squeaking noise, or the rod feels cold, STOP and pause 30 seconds while the O-ring and rod warm up. Pull the rod up until you see the straw and quartz shroud in the airlock window. Stop when the Kapton tape, where the top of the straw is mounted on, is at the top of the window.

Close the airlock valve, by turning the lever counter clock-wise until the lever is horizontal. The airlock will automatically vent. Turn the cam levers off the blue slide seal assembly. Pull the sample and rod completely above the sample transport, and then lower carefully so the rod is below the false ceiling.

Place the rod and sample on the counter. Slide the quartz shroud off the straw. Cut the copper wire holding the straw on the rod. Remove the straw and sample.

If finished running experiments, replace the blue airlock plug, and set the cam levers that hold the plug. Push the “PURGE AIR LOCK” button. Don’t forget to logout.

Register Sample:

F2 – Register Sample menu

Enter “**Sample Name**”, “**Sample Mass**”, and “**Comment**”.

F4 – Set Parameters menu

Enter new “**Data File Name**”, very important or you may have trouble finding this data set later. If you forget this, the data will be appended to the previous file name, which can lead to some confusion, particularly if the original file was created by someone else.

Center Sample:

F4 – Set Parameters menu

Set “**Magnetic Field (G)**” to 500 Gauss.

Set “**Scan Length (cm)**” to value for sample measurement (e.g. 4.00 cm).

Set “**Temperature (K)**” to your experiment starting temperature, or a good default value is 35°K.

F2 – Register Sample menu

“**Initialize Sample Transport**” this calibrates the sample transport with respect to its bottom limit switch.

“**Perform Full Length DC Scan**” a voltage response curve will be displayed in the Graph window. The “center” is ½ of the “Scan Length” set previously. Thus, if Scan Length = 4 cm, then you want your sample center at 2 cm (if Scan Length = 6 cm, center = 3 cm).

“**Adjust Sample Position**” If the center peak is off center by more than 0.05cm, use this option to center the sample.

When the sample is close to the center the option “**Adjust Position Automatically**” will appear. Select this option if available.

If the automatic positioning is not available, select “**Adjust Position Manually**”. Then you will be prompted to loosen the knurled nut at the top of the sample rod, and to “**Press <ENTER> When Nut is Loose**”.

Caution: When loosening, or tightening, the knurled nut hold onto the S-slide assembly so as not to put too much torque on the sample transport assembly. And, be cautious not to move the sample rod vertically, or the sample won’t be where the computer thinks it is.

The system will reposition the sample and then prompt you to tighten the knurled nut and “**Press <ENTER> When Nut is Tight**”.

Repeat until centered

“**Perform DC Centering Scan**” will measure the voltage response again. “**Adjust Sample Position**” Adjust the sample position as needed as above, using either the automatic or manual position adjustment.

CAUTION

Magnetic Field limits as a function of the liquid He Level. The measured liquid He Level limits the maximum absolute Field value.

Helium Level	Maximum Field +/- (Gauss)
40%	55,000
30%	20,000
25%	5,000
20%	1,000

Some Notes on temperature settings:

Setting the temperature is fairly straightforward. But, the time to equilibrate different temperatures varies. The change from 300°K to 35°K takes 20 minutes, whereas going from 35°K to 5°K takes 10 minutes. As it reaches the set temperature, the temperature will cycle above and below the final temperature with decreasing amplitude until it stabilizes at the set temperature.

To achieve a temperature below 4.2°K the system has to perform several manipulations, and the time required to stabilize is longer. It is not necessary, but it is recommended before beginning any work below 4.2°K, stabilize the temperature at a value between 5°K and 10°K. When the temperature is set at a value below 4.2°K the system will first transfer some liquid Helium into a special chamber. This step takes 12 minutes. Then, to lower the temperature below 4.2°K a partial vacuum is pumped on the special chamber. Overall, this process will take 15 to 20 minutes. Patience is a necessity here, since resetting the temperature during the process will just restart the time required.

On occasion the system will not stabilize at the set temperature. If the temperature just seems to be bouncing around the final temperature, and the amplitude of the cycling is not decreasing, then try typing in the temperature again. When trying to go below 4.2°K, if the temperature settles at a value above 4.2°K after 20 minutes, then try typing in the temperature again.

When the liquid Helium level drops below 40% working at lower temperatures will take longer; and, working below 4.2°K is not possible.

F3 – Measure menu:

There are a number of measurement modes listed with this menu. Alternatively, the Sequence Editor (F9) can be used to create a totally customized measurement scheme. Only the “Immediate” mode will be detailed here; although, the other modes listed in this menu are fairly self-explanatory. For completeness the listed modes are:

- Immediate . . .
- EMU vs Field . . .
- EMU vs 1/Field . . .
- EMU vs Temperature . . .
- EMU vs 1/Temp . . .

As an example select “**Immediate**” and a submenu is displayed. The “Set Field”, and “Set Temperature” are set as desired. “Pause (Sec)” suspends the start until the Field and Temperature have stabilized, and then pauses before starting the measurement. “Break Plot” allows different data point shapes and colors for EMU graphs. “Measure” begins the measurement.

F9 - Sequence Editor:

You can create or edit customized experiments in this menu. If you do edit a current sequence be sure to copy to a new file name and edit the new file, unless you own the sequence. There is no file ownership, so be careful not to overwrite somebody else’s work.

In the Editor mode a number of keys are used to perform functions. The Function keys (F2 to F9) bring up the function menus on the right side of the screen, which can be used to enter commands into the sequence. The left side of the screen contains the sequence file. “**Tab**” is used to move between the left and right side of the screen. “**G**” will let you go to specific record numbers in the sequence. “**P**” prints the sequence. “**L**” loads the current command list into the sequence editor. “**X**” inserts the editor contents into the command list and executes the sequence.

“**F**” loads a sequence file into the editor. Type in the file name, or hit “Enter” to display and select from a directory listing. An error message will often appear when displaying a directory listing. Ignore it, press and hold down the spacebar until the directory is displayed.

“**S**” saves the sequence editor contents to a file name. “**M**” marks a block of records to be copied or deleted. The first “**M**” marks the beginning, and the second “**M**” marks the end of the block. “**C**” copies the current record or the marked block into a temporary buffer. “**Del**” key copies and deletes the current record or the marked block. “**Ins**” key inserts above the cursor the record (or block) last deleted or copied.

This example is of a variable temperature, constant field experiment (EMU vs. temperature). Select “**Editor . . .**”, hit **F3**, then select “**EMU vs Temperature . . .**”. Enter the “**Start Temperature**” and “**End Temperature**”. Then enter either the “**Number of Temps**”, or “**Increment Spacing**”. Enter a value for “**Pause Before Measuring (Sec)**” to allow for maximum field and temperature stability with sensitive samples. Select “**Generate Sequence**” and the sequence of temperature measurements will appear in the left screen.

Return to the left screen and enter the beginning and end of the sequence. At the beginning hit **F4**, select “**Set Field**”, and enter the field value. Next, hit **F2**, and select “**Perform DC Centering Scan**”. Then, hit **F2**, and select “**Adjust Sample Position**”.

The sample should be re centered and the sample position adjusted every 5 to 10 degrees of temperature change. This compensates for changes in the sample rod length at different temperatures. Just add the steps in the sequence for a centering scan and adjustment of sample position.

At the end of every sequence remember to add the sequence steps to set the field to 0.0 Gauss, and set the temperature to 35°K. This protects the Magnetic field power module, and helps conserve liquid Helium. Remember to “Save” the sequence to a file.

e.g.

1. Set Field 5000G
2. DC Scan
3. Auto Adjust Sample Position
4. Set Temperature 4 K
5. Measure DC
6. Set Temperature 4.25 K
7. Measure DC
8. Set Temperature 8 K
9. DC Scan
10. Auto Adjust Sample Position
11. Measure DC
12.
- ...
45. Set Field 0.0
46. Set Temperature 35 K
47. End Sequence

F8 – Sequence Control:

Select for “**Sequence Type:**”, “**Programmed**”. Type in the “**Sequence File Name**”, or press Enter to select from a directory listing. If the directory listing gives an error message, ignore the error, press and hold the spacebar until the directory appears. Enter the value for “**Start at Record #**” (default is 1). Select “**Execute Sequence**”. The options at the bottom of “**Halt**”, “**Resume**”, and “**Abort**” are self-explanatory.

Experiments will run anywhere from 1 to 12 hours; although 4 to 8 hours are more typical. Check the instrument regularly to make sure it is “behaving”.

F6 – Utilities / Extract Data:

Under the Utilities menu select “**Extract Data**”. Under “**Data File**” type in the file name with the extension “**.dat**”, or find your file (if an error message appears, ignore, press and hold the spacebar until the directory listing is displayed.) Type in the “**Extract File**” name (e.g. a:\filename.dat). This example would write the extract file to the **a:** drive on a 3½” floppy. Finally, highlight “**Extract Data**” and hit “**Enter**”.

System Info:

This system is known as a MPMS (**M**agnetic **P**roperty **M**easurement **S**ystem), with a SQUID (**S**uperconducting **Q**Uantum **I**nterference **D**evice) detector. The manufacturer is Quantum Design, USA (www.qdusa.com).

The DC magnetic field can be run from: -55,000 to 55,000 Gauss (-5.5 to 5.5 Tesla).
The sample temperature can be run from: 1.7 to 400 Kelvin (°K) (-271.6 to 126.7 °C).

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Portions of guide based on manual by Curt Zaleski