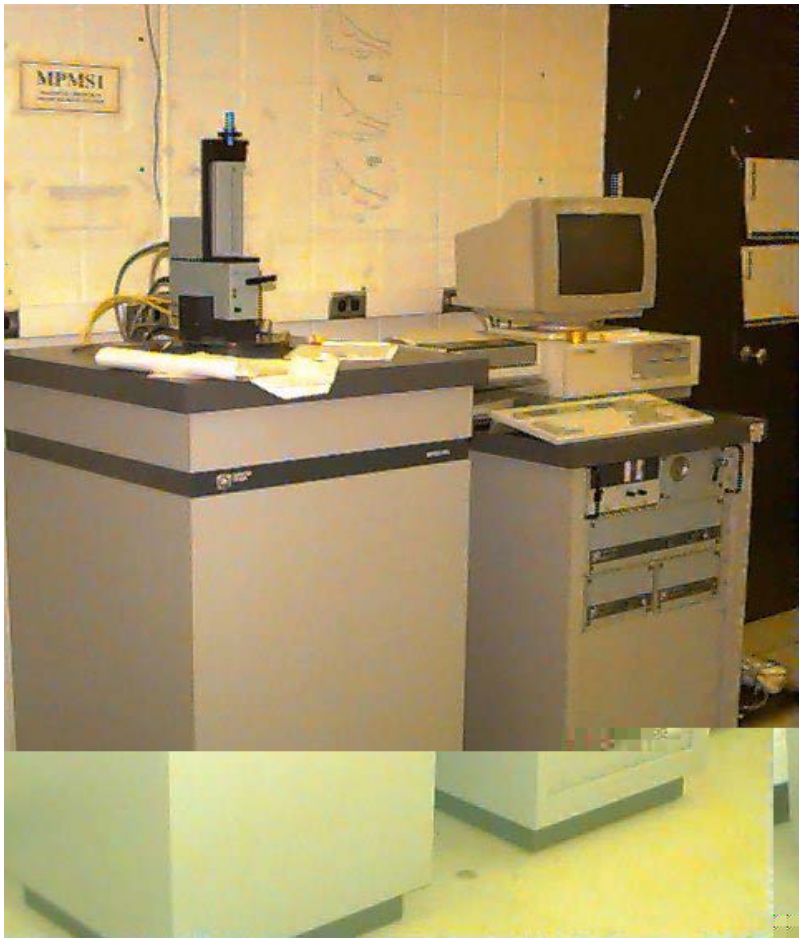
Superconducting Susceptometer (MPMS-5S)

Quantum Design
Room 296 (*MPMS*)

Sensitivity:	$1x10^{-11}$ A m^2
Applied DC fields:	0 T to 5 T
Applied AC fields:	0 G to 3 G (zero-to-peak), 0.01 Hz to 1000 Hz
Temperatures range:	2 K to 300 K

See *IRM Quarterly*, Spring 1992, Vol. 2, No. 1; Fall 1993, Vol. 3, No. 3; Spring 1994, Vol. 4, No. 1 (magnetic quantities and units); Winter 1998-1999, Vol. 8, No. 4; Winter 2002-2003, Vol. 11, No. 4



Contents:

I) Sample Preparation
II) Sample Loading/Unloading
III) Sample Centering
IV) Software Setup and Running Experiments
V) Helium Transfer

The MPMS consists of an instrument station, a control/electronics rack/station, a computer, a sample holder rod, various small tools, and supplies.
There are two possible **configurations** for the instrument:

1) Standard. This configuration incorporates an air lock, allowing samples to be loaded or removed at low temperature.

2) RSO (reciprocating-sample option). This configuration increases sensitivity for measurement of very weak samples, but lacks an air-lock mechanism, and thus requires that the system be at room temperature for sample loading or removal.

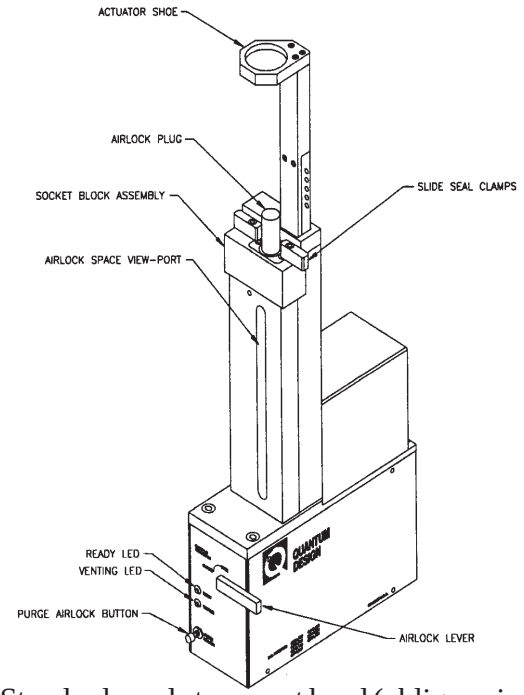
I. SAMPLE PREPARATION (For either RSO or standard mode)

1) Prepare the sample for the drinking straw holder:

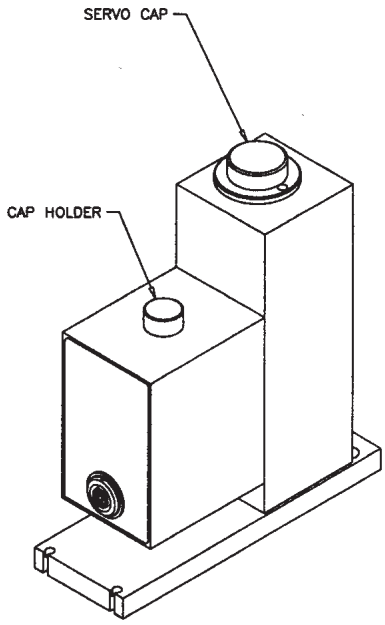
- **a) Gel-cap method**: Insert some fiberfrax or bits of kimwipe in the ends of a gel-cap (to keep the sample snug). After placing the sample in the gel-cap and closing it until it snaps shut, seal the side of the gel-cap with enough Capton tape to make a snug fit in the drinking straw.
- **b) Second straw method**: First, place the sample chip in a short piece of drinking straw. Then, insert this sideways into the main straw. Recommended only for single chips.

2) Determine instrument configuration before proceeding to load your sample into the MPMS (see Fig. 1below).

3) Cut the straw to the appropriate length and **position the sample** in the straw according to the tem-



Standard-mode transport head (oblique view)



RSO-mode transport head (posterior view)

Figure 1. Two possible head configurations for the MPMS. Standard (left), and reciprocating sample option (RSO; right).

plates.

4) Place a small piece of Capton **tape over the bottom of the straw** to prevent sample from falling out **(standard mode only)**.

5) Puncture straw near bottom and top to allow air to escape during airlock evacuation **(standard mode only)**.

II. SAMPLE REMOVAL/LOADING

1) Standard Configuration. Steps a-e are for removing a sample from the instrument. If there is no sample installed, proceed to **Step f** to load your sample.

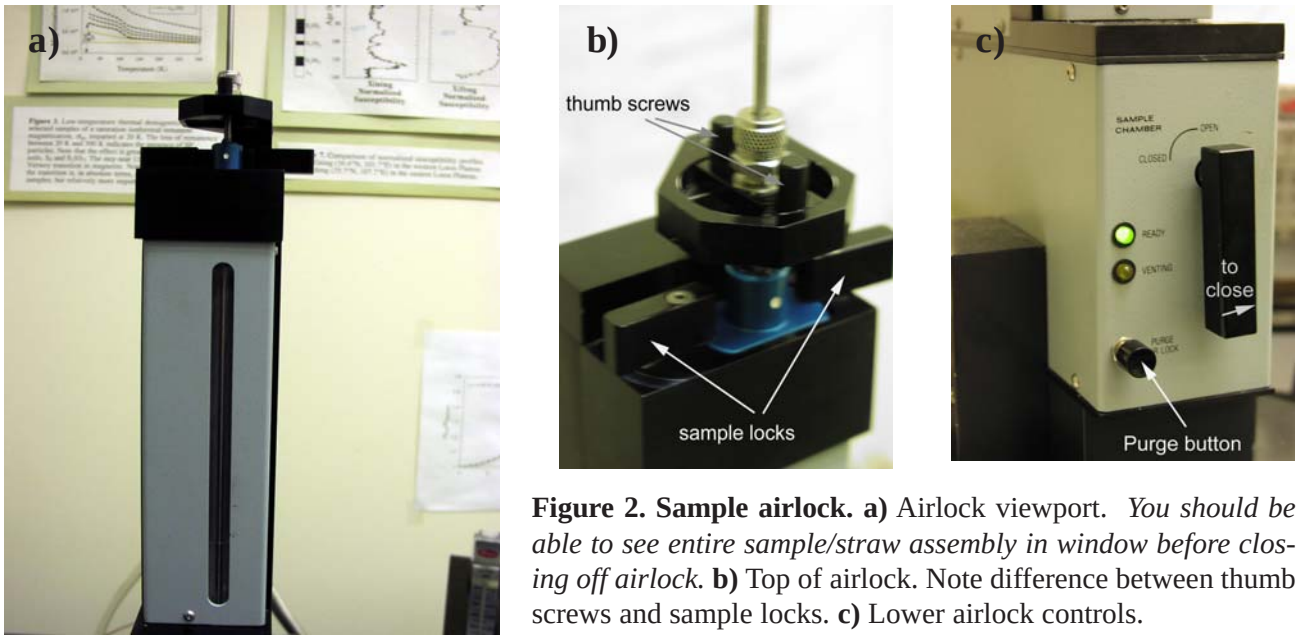


Figure 2. Sample airlock. a) Airlock viewport. *You should be able to see entire sample/straw assembly in window before closing off airlock.* **b)** Top of airlock. Note difference between thumb screws and sample locks. **c)** Lower airlock controls.

a) Loosen thumb screws (NOT the sample locks!! see Fig. 2b) and rotate the sliding-clamp bracket out from under the thumb screws.

b) Pull the sample rod assembly up until the sample is visible in the window (Fig. 2a). Make sure that you can see the bottom of the straw!

c) Close the sample chamber valve (Fig. 2c). The "venting" light will go on for a few seconds.

d) When the light goes out, **loosen sample locks** (Fig.2b) and **remove sample rod assembly**.

e) Replace blue plug and tighten sample locks. Press "Purge Airlock" button (Fig. 2c).

f) Exchange sample on rod:

i) Gently lay the standard rod on the long counter top with the bracket ('sliding clamp' in Fig. 3) hanging just over the edge.

ii) Slide the glass sleeve (Fig. 3) to the left, exposing the straw/sample assembly.

iii) Remove old sample.

iv) Attach new sample securely to the rod with Capton tape.

v) Position sample in straw according to template.

vi) Puncture straw near bottom and top several times to allow air to escape.

vii) Slide glass sleeve back as far to the right as it will go.

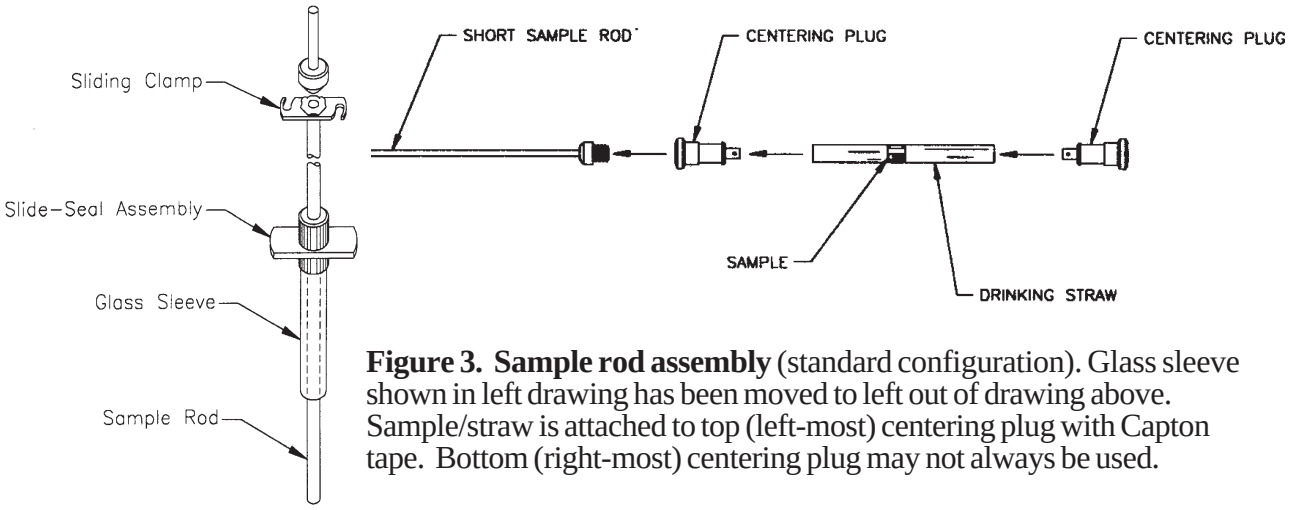


Figure 3. Sample rod assembly (standard configuration). Glass sleeve shown in left drawing has been moved to left out of drawing above. Sample/straw is attached to top (left-most) centering plug with Capton tape. Bottom (right-most) centering plug may not always be used.

g) Take the rod/sample back to the MPMS. The blue plug should be in place, the sample locks closed, and the green "Ready" light on.

h) Open sample chamber valve slightly and close it again. This should vent the airlock and the green light will go out.

i) Loosen sample locks and remove blue plug.

j) Insert prepared sample rod assembly with white dot facing forward, and tighten sample locks.

k) Push "purge air lock" button. The "venting" light will go on and off for about 30 seconds. Watch the sample through the window to make sure it does not move[i] or fall out of the straw[ii].

i) If the sample moves only a little (<few mm), it is still OK; proceed to next step. If it moves up or down too much, it will not be possible to center it. In this case, wait for the green "ready" light, open the sample chamber valve, then close it again, and return to step d to remove and reposition your sample.

ii) If the sample falls out of the straw, find an IRM staff member for assistance. DO NOT ATTEMPT TO RETRIEVE IT YOURSELF!

k) When the green "ready" light comes on, **open the sample chamber valve**. *If the ready light fails to show after a few minutes, there are two possible causes:*

i) The system is cooling to a set-point temperature. Check the current T and set-point T. If the system is cooling, you can interrupt it: click on the temperature display, select Set Temperature, and enter a value near (slightly above) the current temperature. Once the new set-point is registered, the green light should come on immediately. You can then re-enter the original set-point T and continue with sample installation as the system cools.

ii) If the temperature is stable and the green light still fails to illuminate, there is probably a vacuum leak. Find an IRM staff member ASAP!

l) Push the sample rod assembly down. At least once per day, apply a thin coat of vacuum grease to the rod as you insert it.

m) Fit the bracket under the thumb screws, and tighten the thumb screws.

2) Sample Loading for RSO Configuration.

a) Check system temperature. Look at the system status panel on the bottom of the computer screen and find the current temperature (both the set-point and actual temperature are shown). ***If the temperature is below 280K*** do ***not*** continue with sample loading until T is >= 280K. To change the temperature set-point, click on the temperature indicator and an edit box will open,

where you can enter a value of 300. Wait until T is at least 280K before continuing.

b) Click on the *Sample* menu on the top of the screen and select *Install/Remove Sample*. You will hear the sample space being flushed with He gas, and the top vacuum seal will break open.

c) Remove the black cap (which the software will refer to as the *servo*) and set it on the counter upside-down (so the rubber o-ring does not pick up dirt from the counter top).

d) Remove the clear plug from the top of the RSO transport head. Invert it and use the threaded tip to grab the matching threaded piece on the blue plug and **gently remove the RSO sample rod**. Replace the clear plug.

e) Exchange sample on rod:

i) Gently lay the RSO rod on the long counter top with the blue plug hanging just over the edge.

ii) Remove the previous straw/sample assemblage from the rod and remove the black end-plug from the straw.

iii) Press your straw/sample onto the RSO rod.

iv) Adjust the position of your sample in the straw, according to the *RSO 4-cm* position on the template.

v) Insert the end-plug.

f) Take the rod and sample back to the MPMS. Remove the clear plug, **lower the sample rod** (twisting gently if you encounter any resistance), press the blue plug into place (using a fingertip or the threaded plug tip), and replace the clear plug.

g) Click on the *Purge* button on the computer display. You will hear the sample space being evacuated and flushed with He gas. This will be repeated several times, and then the green *Ready* light on the RSO head will come on.

h) An editing box will open on the screen, allowing you to enter the new sample name, mass, etc. Then proceed to section III.

III. SAMPLE CENTERING (For either RSO or standard mode)

1) Make sure temperature is stable before centering for best results.

2) Click on the *Center* menu on the top of the screen.

3) Select *DC Centering*.

4) Click on Initialize Transport.

5) Run the *Center* procedure. A SQUID response curve will be displayed in about 30 seconds. The response curve should be symmetric with the large central bump (up for positive fields or moments) centered on the plot at ~2 cm, as in the example (Fig. 4). Check the magnetic moment; if > 0.5 emu, remove your sample, decrease the amount of material and re-start. *If there is not a recognizable central peak (positive or negative), there are two things to try:*

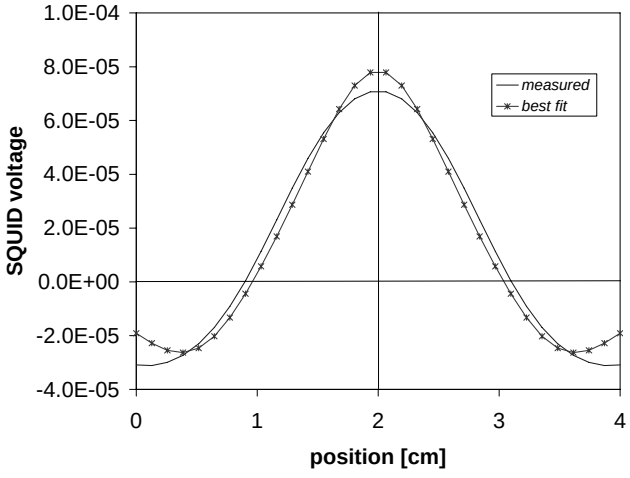


Figure 4. Output from Center procedure.

a) Apply a small field (e.g., 10 G or 100 G; click on the field display panel on the bottom of the

screen and enter the desired value). For weak samples that have not been previously magnetized, this may help to generate a measurable signal. Repeat Centering proceedure with field on.

b) Try a *Full DC Scan*. If the sample is far from the correct location, this full-length scan is a more effective way to detect its position.

5) Adjust sample position.

a) Adjust automatically. If sample location is only slightly (a few 10s of mm) offset from the 2 cm mark, click on *Adjust Position* and run the *Adjust Automatically* procedure. Check the box indicating you want to re-run the Center procedure when finished.

b) Adjust manually. If the sample is too far off-center, you will not be able to adjust the position automatically.

i) Standard configuration: you can run the *Adjust Manually* procedure, which involves loosening and tightening the knurled nut on the sample rod, as instructed by the software.

ii) RSO configuration: you will have to remove the sample, re-position manually, and start over.

IV. SOFTWARE SET-UP AND RUNNING EXPERIMENTS

The panel on the left side of the screen contains controls for selecting and running sequences (experiments) and for specifying data files for output.

1) Set sample name. The top box indicates the sample name. You can click on the *Change* button below to change the sample name, mass, etc. for the header of the data file. (Note that mass is not used.)

2) Select experiment sequence. The second box shows the currently selected *Sequence File*. Experiments are run by executing Sequence Files, which contain all the necessary instructions for temperature and field control, data acquisition, etc. To view the contents of the current sequence file, click on the *Edit* button. An edit window will open, and will display the contents of the selected file. To close the edit window, click on *File* and then *Close*. To view a list of sequence files, click on the *Change* button below the sequence file name. You can scroll through the list displayed, and select a file by double-clicking on its name. If you wish to modify any of the existing files, consult an IRM staff member.

3) Set data file name. The third box on the left panel is labeled *Sequence Base Data File Name*. All data collected by the sequence as it executes will be stored in the file designated in this box. To specify a new file, click on the *Change* button below the sequence file name. You can use the control buttons to change directories, or to create a new directory for your files.

4) Start sequence. The fourth and final box on the left-side panel is the *Sequence Control* box. Click on the *Run* button to begin the experiment.

5) Display plot. Once the sequence begins collecting data, you can display a graph of measurements as they are acquired. Click on the *View* button below the *Sequence Base Data File Name* box. A list of relevant files will appear. The *dat* file is most probably what you want to display: moment vs. temp, field, etc. The *lastscan* file allows you to look at the SQUID response curve for the previous measurement.

VI. HELIUM TRANSFER (IRM Personnel ONLY)

1) This is a two-person job.
2) Collect the following near the MPMS:
    - liquid helium dewar

- helium gas tank (in the corner behind the door)
- helium transfer tube (on the back wall)
- dewar exit valve adapter (on the transfer tube)
- two pair of gloves (in the cabinets)
- heat gun (in the cabinets).

3) Purge both the hose from the helium gas tank and the connector to the dewar with helium gas, then connect the hose to the dewar. Set the regulator for about 1 psi, but leave the inlet valve on the dewar closed.
4) Bleed the 0.5 psi relief valve on the dewar, then open the valve at the top of the dewar. Insert the long end of the transfer tube, making sure to seat the outer sleeve of the tube in the dewar adapter. Push in slowly.
5) Close the 0.5 psi relief valve and open the inlet valve to start gas flow into dewar.
6) Bleed off the pressure in the MPMS relief valve, then remove the cap when ready to insert the short end of the transfer tube [See next step].
7) When the sound of the gas plume coming out of the transfer tube starts to change (indicating liquid is nearing), insert the short end of the transfer tube into the MPMS. Do this quickly to avoid freeze-up, but with finesse to negotiate around all the internal baffles. Again make sure that the outer sleeve of the transfer tube seats in the connector to the MPMS.
8) Adjust the regulator to get about 1 psi for transfer pressure.
9) In the *Utilities* menu, select *Helium Fill*, and start the count-down timer by clicking on the up arrow once or twice. The whole transfer should take about 10 minutes.
10) Close the regulator when the MPMS is about 95% full. Pull the transfer tube out of the MPMS when the helium-level jumps up to 98% full. CAUTION! The tube is dangerously cold! USE GLOVES! Pull the other end of the transfer tube out of the dewar. USE GLOVES!
11) Replace the cap on the MPMS. Close the inlet valve on the dewar and open the 0.5 psi relief valve. Close the dewar exit valve, and remove the adapter
12) Stop and close the graph. The gas flow on the MPMS meters should drop back to below 100 cc/min in a few minutes.
13) Put everything away.